

**Food inspection and analysis : for the use of public analysts, health officers, sanitary chemists, and food economists / by Albert E. Leach ; revised and enlarged by Andrew L. Winton.**

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# FOOD INSPECTION *and* ANALYSIS

FOR THE USE OF PUBLIC ANALYSTS, HEALTH OFFICERS,  
SANITARY CHEMISTS, AND FOOD ECONOMISTS

BY

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PREFACE TO FOURTH EDITION

AFFECTIONATELY DEDICATED TO THE MEMORY OF  
**Charles Pomeroy Worcester,**  
FORMER ANALYST OF THE MASSACHUSETTS STATE BOARD OF HEALTH,  
WHOSE LOVABLE PERSONALITY AND STERLING INTEGRITY WERE  
A CONSTANT INSPIRATION DURING MANY YEARS  
OF CLOSE COMPANIONSHIP TO  
THE AUTHOR.



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of active service to the  
State.



## PREFACE TO FOURTH EDITION.

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THE present revision has been carried out after a thorough search of the literature. A large amount of new material has been added or substituted for that in previous editions and the size of the book increased by 90 pages. While a somewhat different arrangement of the chapters may seem more logical, it was decided to retain the old order to which those who have hitherto used the book have become accustomed. The lists of references at the end of the chapters, which never aspired to be complete bibliographies, have been dropped and more attention has been given to footnote references.

The reviser is indebted for criticisms and notes to many friends, especially the following: Prof. E. H. Farrington, Prof. E. S. Guthrie, and Dr. L. L. Van Slyke (dairy products), Prof. H. S. Grindley (meat), Mr. F. C. Atkinson, Mr. Carl S. Miner, and Prof. Harry Snyder (cereal products), Mr. M. C. Albrech, Mr. F. M. Boyles, and Mr. A. E. Paul (spices), Mr. H. S. Bailey, Prof. E. F. Ladd, and Dr. David Wesson (oils), Dr. C. S. Browne, Mr. A. Hugh Bryan, Dr. W. D. Horne, and Mr. W. E. Rice (sugar), Mr. H. M. Loomis and Mr. W. E. Mathewson (colors), and Dr. A. R. Albright (flavoring extracts).

A special feature is a final chapter by Prof. Gerald L. Wendt, on the determination of acidity by the hydrogen electrode, a method which seems destined to play an important part in food analysis.

So far as possible original papers have been consulted, but whenever this was not possible owing to the war or other conditions the abstracts in the Experiment Station Record and Chemical Abstracts have proved invaluable.

A. L. W.

WILTON CONN., January, 1920.

# PRELIMINARY TO FOURTH EDITION

The present revision has been carried out after a thorough study of the literature. A large amount of new material has been added or substituted in the text in various instances and the size of the book increased by 50 pages. With a somewhat different arrangement of the chapters and a new frontispiece, it was desired to retain the old order to which those who have followed the book have become accustomed. The list of references at the end of the chapters which were added to be complete bibliographies have been dropped and more attention has been given to the text.

The review is indebted for criticism and advice to many friends, especially the following: Prof. H. H. Lamb, Prof. E. S. Gifford, and Dr. J. J. Van Slyke (University of California, Berkeley), Prof. E. C. Ashman, Mr. C. G. Allen, and Prof. H. H. Lamb (University of California, Berkeley), Mr. E. C. Ashman, Mr. E. S. Gifford, and Mr. E. H. Lamb (University of California, Berkeley), Mr. H. S. Gifford, and Mr. E. H. Lamb (University of California, Berkeley), Mr. C. G. Allen, Mr. A. H. Lamb, and Mr. E. H. Lamb (University of California, Berkeley), Mr. W. E. Lamb, Mr. H. S. Gifford, and Mr. E. H. Lamb (University of California, Berkeley), and Dr. A. H. Lamb (University of California, Berkeley).

A special feature is a final chapter on the subject of "The determination of the absolute intensity of light" which is intended to give an up-to-date account of the subject. The determination of the absolute intensity of light is a subject which has been of increasing importance in the study of the properties of light. The present edition is intended to give an up-to-date account of the subject. The present edition is intended to give an up-to-date account of the subject.

A. L. W.

UNIVERSITY OF CALIFORNIA, BERKELEY



## PREFACE TO FIRST EDITION.

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IN the preparation of the present work, the requirements of the public analyst are mainly kept in view, as well as of such officials as naturally cooperate with him in carrying out the provisions of the laws dealing with the suppression of food adulteration in states and municipalities. To this end special prominence is given to the nature and extent of adulteration in the various foods, to methods of analysis for the detection of adulterants, and to some extent also to the machinery of inspection.

While the analyst may not in all cases have directly to deal with the *minutiæ* of food inspection, his work is so closely allied therewith that this branch of the subject is of vital interest and importance to him. Indeed, in many smaller cities one official often has charge of the entire work, combining the duties of both inspector and analyst.

Endeavor has been made, furthermore, to deal with the general composition of foods, and to give such analytical processes as are likely to be needed by the sanitary chemist, or by the student who wishes to determine the proximate components of food materials.

It has been thought best to include brief synopses of processes of manufacture or preparation of certain foods and food materials, in cases where impurities might be suggested incidental to their preparation.

In view of the fact that Massachusetts was the pioneer state to adopt, over twenty years ago, a practical system of food and drug inspection, and for many years was the only state to enjoy such a system, no apology is perhaps needed for more frequent mention of Massachusetts methods and customs than those of many other states, in which the food laws are now being enforced with equal zeal and efficiency.

Considerable attention has been paid in the following pages to the use of the microscope in food analysis. Of the figures in the text illus-



trating the microscopical structure of powdered tea, coffee, cocoa, and the spices, fifteen have been reproduced from the admirable drawings of Dr. Josef Moeller, of the University of Graz, Austria. Acknowledgment is gratefully given Dr. Moeller for his kind consent to their use.

The photomicrographs in half-tone, forming the set of plates at the end of the volume, were all made in the author's laboratory, and may be divided into three classes: 1st, illustrations of powdered pure foods and food products, as well as of powdered adulterants; 2d, types of *adulterated* foods, chosen from samples collected from time to time in the routine course of inspection; and 3d, photographs of permanently mounted *sections* of foods and adulterants.

While recent works covering the whole field of general food analysis are comparatively few, the number of treatises, monographs, government bulletins, and articles scattered through the journals, dealing with special subjects relative to food and its inspection, is surprisingly large, and from a painstaking review of these much information has been culled, for which it has been the author's intention at all times to give credit.

Special mention should here be made of the valuable publications of the U. S. Department of Agriculture, both the bulletins issued from Washington, and those from the various experiment stations, an ever-increasing number of which are becoming engaged in human food work. The author has freely drawn from these sources, and especially from the data and material furnished by his coworkers in the recent and still pending labor of preparing food methods for the Association of Official Agricultural Chemists, and he wishes to extend his thanks to all of them for their assistance. Appreciation is also expressed for the care and discrimination shown by Mr. L. L. Poates in the preparation of the cuts. Thanks are especially due to Mr. Hermann C. Lythgoe, Assistant Analyst of the Massachusetts State Board of Health, for his invaluable cooperation, and to Dr. Thomas M. Drown for helpful hints and suggestions.

BOSTON, MASS., July 1, 1904.



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# FOOD INSPECTION AND ANALYSIS.

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## CHAPTER I.

### FOOD ANALYSIS AND OFFICIAL CONTROL.

#### INTRODUCTORY.

THE general subject of food analysis, in so far as the public health is concerned, is to be considered from two somewhat different standpoints: first, from the outlook of the government, state, or municipal analyst, whose mission it is to ascertain whether or not the food may properly be considered pure or free from adulteration; and second, from the point of view of the food economist, whose aim is to determine its actual composition and nutritive value. The one protects against fraud and injury, the other furnishes data for the arrangement of dietaries and for an intelligent conception of the rôle which the various nutrients play in the metabolism of matter and energy in the body. The two fields are as a rule distinct each from the other, often involving, in the examination of the food, different methods of procedure.

**Official Control of Food.**—In view of the importance of the consideration of food with reference to its purity, an ever-increasing number of states have realized the necessity of protecting their citizens from the unscrupulous manufacturers who in various lines are seeking to produce cheaper or inferior articles of food in close imitation of pure goods. Many of the states have laws in accordance with which the sale of such impure or adulterated foods is made a criminal offense, and some, but not all of these, are provided with public analysts and other officers to enforce these laws and punish the offenders. Numerous communities are awake to the importance of municipal control of such commonly used articles of food as milk, butter, and vinegar, and in many cases have machinery of their own for regulating the sale of these foods.



Since January 1, 1907, the federal government has been actively engaged in the enforcement of the national food law of June 30, 1906, through the Bureau of Chemistry of the U. S. Department of Agriculture. In addition to the central laboratories of this Bureau at Washington, a number of branch laboratories have been established in the principal cities of the United States to enforce the provisions of the national law which regulates interstate commerce in foods, as well as their manufacture and sale in the territories and the District of Columbia, and their importation from foreign countries.

**Food Analysis from the Dietetic Standpoint.**—The study of the principles of dietetics has been given increased attention during the last decade in the curricula of many of the technical schools and colleges. Much has been accomplished by certain of the state experiment stations working as a rule in connection with the United States Department of Agriculture along this line. Investigations of this character are especially valuable, and are indeed rendered necessary by the general tendency of the modern physician to regard the hygienic treatment of disease, especially with reference to the matter of diet, as often of far greater importance than the mere administering of drugs.

The food economist studies the varying conditions of age, sex, occupation, environment, and health among his fellow men, with a view to showing what foods are best adapted to supply the special requirements of various classes. The quantity and proportion of protein, fat and carbohydrates, or of fuel value best suited for the daily consumption of a given class or individual having been determined, dietaries are made up from various food materials to supply the need with reference as far as possible to the taste and means of the consumer.

Experiments are made on families, clubs, or individuals, representing various typical conditions of life, and extending over a given period, during which records are kept of the available food materials on hand and received during the term of the experiment, as well as of those remaining at the end. In the case of individuals, additional records may be kept of the amount and composition of the urine and feces. From such data the physiological chemist calculates the amount of nutrients utilized, and studies the metabolism of material in the human body.

Up to this point no very extensive apparatus is required, but if in addition the income and outgo of heat and energy are to be studied, which are important to a complete investigation of the economy of food in the body, the student will require a respiration calorimeter and its appurte-



nances. The calorimeter is so constructed that an individual may be confined therein for a term of days under close observation and with carefully regulated conditions. Such an equipment involves a large expenditure and is to be found in but few laboratories.

It is not the purpose of the present work to go beyond the strictly chemical or physical processes involved in making the analyses by which the proximate components of the foods are determined. For more complete information in the field of dietary studies and the metabolism of matter and energy in the body, the student is referred especially to the investigations of Atwater and his co-workers, as published in the annual reports of the Storrs Experiment Station at Middletown and in the bulletins of the U. S. Department of Agriculture, Office of Experiment Stations, also to studies conducted by Benedict of the Carnegie Institution.

**Commercial Food Analysis.**—The proper preparation of food products has long ceased to be carried on by the hap-hazard rule-of-thumb methods that formerly prevailed. Now in the manufacture of many prepared foods and condiments, especially on a large scale, it has become a necessity to use scientific processes, rendered possible only by the employment of skilled chemists. In fact it is coming to be more and more common for food manufacturers to establish chemical laboratories in connection with their works, in the interests both of economy and of improved production.

Frequently disputed points arise in the enforcement of the food laws that render the services of the private food analyst of great importance both to manufacturer and dealer. Thus a wide field is open to the analyst of foods outside the domain of the government or state laboratory, either in connection with the large food manufacturing plants directly, or in private laboratories for experimental research, or for analytical control work.

#### SYSTEMATIC FOOD INSPECTION.

**Functions of the Official Analyst.**—The public analyst is employed by city, state, or government to pass judgment on various articles of food taken from the open market by purchase or seizure, either by himself or by duly authorized collectors employed for the purpose. The sole object of his examination is to ascertain whether or not such articles of food conform to certain standards of purity fixed in some cases by special law, and in others by common usage or acceptance. Such a public analyst need not concern himself with the dietetic value of the food or whether it is of high or low grade. It is for him to determine simply whether it is genuine or



adulterated within the meaning of the law, and, if adulterated, how and to what extent. Aside from his skill as a chemist, it is often necessary for him to possess other no less important qualifications, chief among which are his ability to testify clearly and concisely in the courts, and to meet at any time the most rigid kind of cross-examination, it being of the utmost importance that he understand thoroughly the nature of evidence.

**Standards of Purity for Food Products.\***—Under an act of Congress approved March 3, 1903, standards of purity for certain articles of food have been established as official standards for the United States by the Secretary of Agriculture. The earlier of these standards were formulated under the Secretary's direction by a committee of the Association of Official Agricultural Chemists. Later, however, a joint committee of that association and of the association composed of state and national food officials has had charge of this work and still later a joint committee of these organizations and the Bureau of Chemistry. Standards have been adopted, covering the entire range of food products.

**Nature of the Analytical Methods Employed.**—Since usually only a small number of the samples submitted are adulterated, the analyst should, as quickly as possible, separate the pure from the impure, so as to concentrate his attention on the latter. The nature of the processes by which this is done varies with the foods. Experience soon enables one to judge much by even the characteristics of taste, appearance, and odor, though such superficial indications should be used with discretion. One or two simple chemical or physical tests may often suffice to establish beyond a doubt the purity of the sample, after which no further attention need be paid to it.

A sample failing to conform to the tests of a genuine food must be carefully examined in detail for impurities or adulterants. While in most cases usage or experience suggests the forms of adulteration peculiar to various foods, the analyst should be on the alert to meet new conditions constantly arising. His methods are largely qualitative, since technically he need only show in most cases the mere presence of a forbidden ingredient, though for the analyst's own satisfaction he had best determine the amount, at least approximately.

In reporting approximate quantitative results in court, especially when they are calculated from assumed or variable factors, or when they are the result of judgment based on the appearance of the food under

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\* U. S. Dept. of Agric., Off. of Sec., Circ. 19.



the microscope, the analyst should always be conservative in his figures by expressing the lowest or minimum amount of the adulterant, so as to give the defendant the benefit of any doubt. When exact standards are fixed by law, as in the case of total solids or fat in milk, for example, there is of course great necessity for preciseness in quantitative work.

A full analysis of an adulterated food beyond establishing the nature and amount of the adulteration is entirely unnecessary, and in most instances adds nothing to the strength of a contested case, as twenty years' experience in the enforcement of the food laws in Massachusetts has shown.

The responsibility resting upon the analyst is not to be lightly considered, when it is realized that his judgment and findings constitute the basis on which court complaints are made, and the payment of a fine or even the imprisonment of the defendant may be the result of his report. Therefore he should be sure of his ground, knowing that his results are open to question by the defendant. Where court procedure is apt to be involved, a safe rule is for the analyst to consider himself the hardest person to convince that his tests are unquestionable, making every possible confirmatory test to strengthen his position and consulting all available authorities before expressing his opinion; and finally, after being fully convinced that a sample is adulterated, and having so alleged, let him adhere to his statements and not waver in spite of the most rigid cross-examination to which he may be subjected.

While each state or municipality has its own peculiar code of regulations and restrictions concerning the duties of the analyst and other officials, these rules are in the main very similar. For instance, it is usually necessary, excepting in the case of such a perishable food as milk, for the analyst to reserve a portion of a sample before beginning the analysis, which sample, in the event of proving to be adulterated, shall be sealed, so that in case a complaint is made against the vendor, the sealed sample may, on application, be delivered to the defendant or his attorney.

**Adulteration of Food.**—Except in special cases a food in general is deemed to be adulterated if anything has been mixed with it to reduce or lower its quality or strength; or if anything inferior or cheaper has been substituted wholly or in part therefor; or if any valuable constituent has been abstracted wholly or in part from it; or if it consists wholly or in part of a diseased, decomposed, or putrid animal or vegetable substance; or if by coloring, coating, or otherwise it is made to appear of greater value than it really is; or if it contains any added poisonous



ingredient. These provisions briefly expressed are typical of the general food laws adopted by most states and by the government, though the verbiage may differ. Laws covering compound foods and special foods vary widely with the locality. As to the character of adulteration, nine out of ten adulterated foods are so classed by reason of the addition of cheaper though harmless ingredients added for commercial profit, rather than by the addition of actually poisonous or injurious substances, though occasional instances of the latter are found.

Authentic instances of actual danger to health from the presence of injurious ingredients are extremely rare, so that the question of food adulteration should logically be met largely on the ground of its fraudulent character. Indeed the commoner forms of adulteration are restricted to a comparatively small number of food products, the most staple articles of our food supply, such as sugar and the cereals, eggs, fresh meat, fresh vegetables and fruit being less often subject to adulteration.

**Misbranding.**—Under the federal food law and the laws of many of the states misbranding constitutes an offense as well as adulteration. By misbranding is meant any untrue or deceptive statement or design on the label of a food package, either regarding the nature of the contents, or of the place of manufacture or name of manufacturer. One of the commonest forms of misbranding consists in the incorrect statement of weight or measure. Extravagant and untrue claims as to nutritive value have hitherto constituted a frequent form of misbranding.

**A Typical System of Food Inspection.**—The efficiency of a system of public food inspection is greatly enhanced if the business part of the work, including the bookkeeping and attending to the outside public, be done wholly through some person other than the analyst, as, for example, a health officer, to whom the collectors of samples and the analyst may report independently as to the results of their work, and whose duty it is to determine what shall be done in cases of adulteration. In this way the analyst knows nothing of the data of collection nor the name of the person from whom the sample was purchased, so that he can truthfully state in court that his analysis was unbiased.

Suppose, for example, that three collectors are employed to purchase samples of food for analysis, their duties being to visit at irregular intervals different portions of a state or municipality. Each collector keeps a book in which he enters all data as to the collection of the sample, includ-



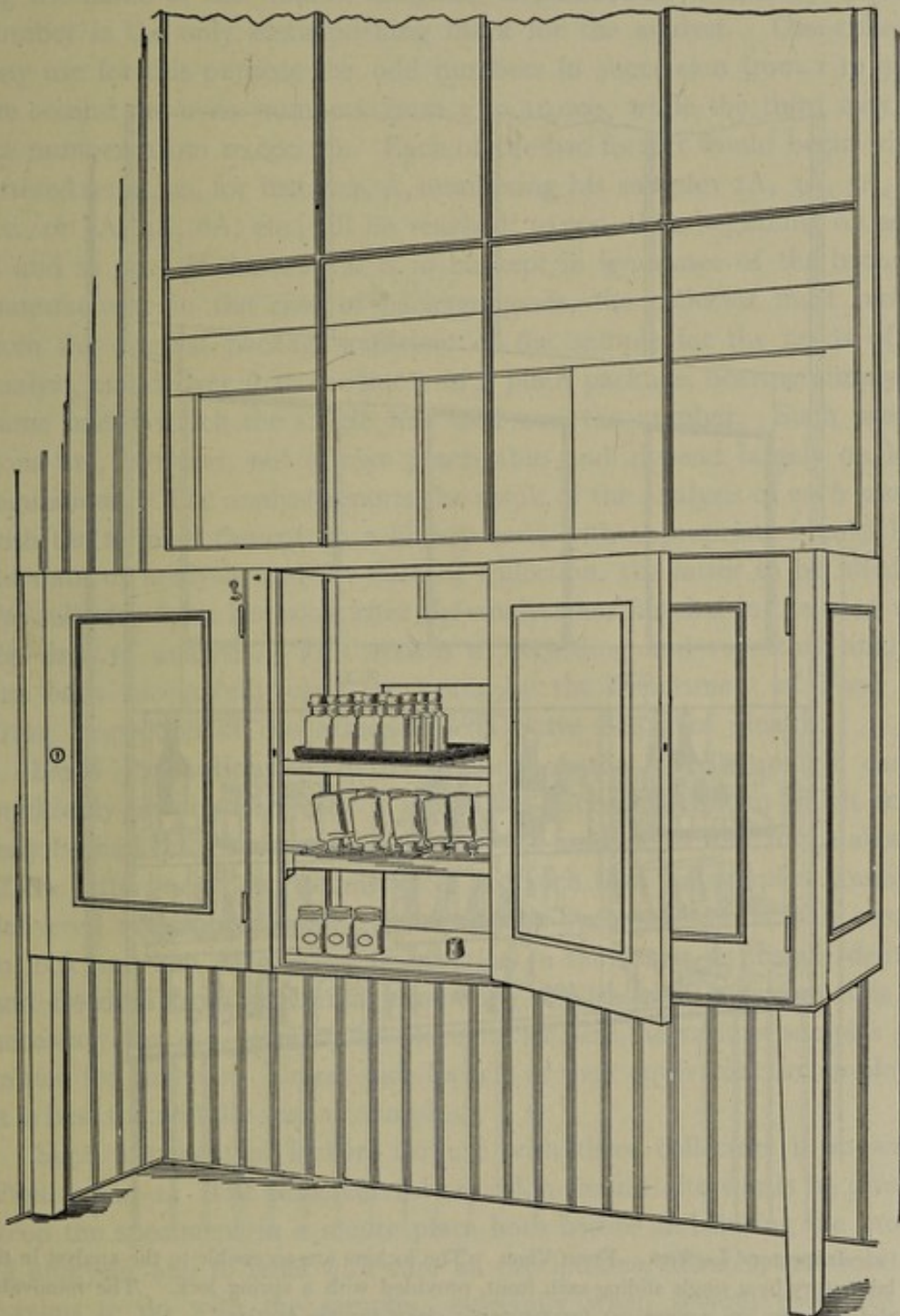


FIG. 1.—Inspectors' Lockers. Insuring safe legal delivery of samples collected by three inspectors. Each locker has a door in the rear accessible, from an anteroom, to the inspector holding key to that locker only.

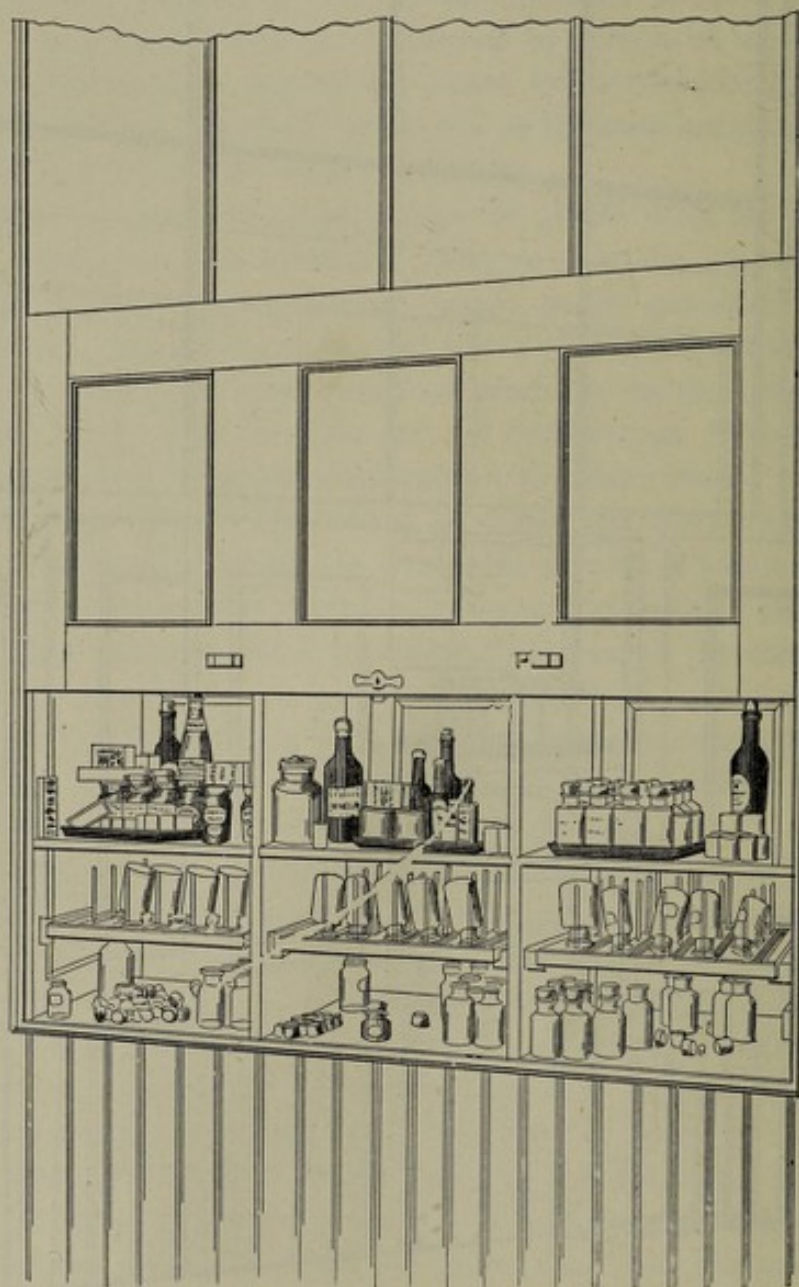


FIG. 2.—Inspectors' Lockers. Front View. The lockers are accessible to the analyst in the laboratory by a single sliding-sash front, provided with a spring lock. The removable sliding-racks are convenient for returning clean sample bottles.



ing the name of the vendor, assigning a number to each sample, which number is the only distinguishing mark for the analyst. One collector may use for this purpose the odd numbers in succession from 1 to 9999, the second the even numbers from 2 to 10,000, while the third may use the numbers from 10,000 up. Each of the two former would begin with a lettered series, as, for instance, A, numbering his samples 1A, 3A, 5A, 7A, etc., or 2A, 4A, 6A, etc., till he reached 10,000, then beginning on series B and so on. If the analyst is to be kept in ignorance of the brand or manufacturer in the case of package goods, the collector must remove from the original package sufficient of the sample for the needs of the analyst, and deliver it to the latter in a plain package, bearing simply the name under which the article was sold and the number. Such precautions are, however, not always practicable and depend largely on local regulations. The analyst reports the result of the analysis of each sample with the number thereof on a library card, with appropriate blanks both for data of analysis and for data of collection, the latter to be filled by the collector from his book after the analyst has handed in the card with the data of analysis. This system of recording and reporting analyses has been successfully used for years by the Department of Food and Drug Inspection of the Massachusetts State Board of Health.

**Legal Precautions.**—The laboratory of the public analyst should preferably be provided with a locker for each collector, to which access may be had only by that collector and the analyst, so that in the absence of the latter, or when circumstances are such that the samples cannot be delivered to him personally, there may be such safeguards with respect to lock and key as to leave no question in the courts as to safe delivery and freedom from accidental tampering. With such a system it is unnecessary for the collector to place under seal the various samples submitted for analysis. Unless such lockers or their equivalent are employed, it is best to carefully seal all samples.

Such a system of lockers for use with three collectors is shown in Figs. 1 and 2. The same careful attention should afterwards be given to keep the specimens in a secure place both before and during the process of analysis, and to label with care all precipitates, filtrates, and solutions having to do with the samples, especially when several processes are being simultaneously conducted, in order that there may be no doubt whatever as to their identity. The importance of precautions of this kind in connection with court work can hardly be too strongly emphasized.



**Practical Enforcement of the Food Law.**—In the case of foods actually found adulterated, there are three practical methods of suppressing their further sale, viz., by publication, by notification, and by prosecution. These may be separately employed or used in connection with each other, according to the powers conferred by law on the commission, board, or official having in charge the enforcement of the law, and according to the discretion of such official.

**Publication.**—Under the laws of some states, the only means of protecting the people lies in publishing lists of adulterated foods with their brands and manufacturers' names and addresses in periodical bulletins or reports. Sometimes it is considered best to publish for the information of the public lists of unadulterated brands as well, and, again, it is held that only the offenders should thus be advertised.

Such publication, by keeping the trade informed of the blacklisted brands and manufacturers, certainly has a decidedly beneficial effect in reducing adulteration, and involves less trouble and expense than any other method. It is obviously an advantage, however, in addition to this to be able in certain extreme cases to use more stringent methods when necessary.

**Notification and Prosecution.**—The adulteration of food is best held in check in localities where under the law cases may be brought in court and are occasionally so brought. The mere power to prosecute is in itself a safeguard, even though that power is not frequently exercised. Under a conservative enforcement of the law, actual prosecution should be made as a last resort. Neither the number of court cases brought by a food commission nor the large ratio of court cases to samples found adulterated are criteria of its good work. Except in extreme cases, it is frequently found far more effective to notify a violator of the law, especially if it is a first offense, giving warning that subsequent infraction will be followed by prosecution. Such a notification frequently serves to stop all further trouble at once and with the minimum of expense. Instances are frequent in Massachusetts where, by such simple notification, widely distributed brands of adulterated foods have been immediately withdrawn from sale.

Massachusetts was the first of all the states to enact pure-food legislation, and since the year 1883 has had a well-established system of inspection, prosecuting cases under its laws through the Food and Drug Department of the State Board of Health. Cases are brought in court with practically no expense for legal services. Complaints are entered by



the collector, or, as he is termed, inspector, who makes complaint not in his official capacity, but as a citizen who under the law has been sold a food found to be adulterated, and who is entitled to conduct his own case, which he does with the aid of the analyst and such other witnesses as he may see fit to employ. Experience is readily acquired by the inspector in conducting such cases in the lower police or municipal courts, where they are first tried, and years ago the services of legal counsel in Massachusetts were dispensed with as superfluous. Where such a practice is in vogue an intelligent inspector must of course be chosen with reference to his ability to do this court work. The food laws are few and simple, as are also the court decisions rendered under them, so that it is no great task for the inspector to become much more familiar with them than the average general lawyer whom he meets in court and who not infrequently consults the inspector for information regarding these laws.

Statistics in the annual reports of the Massachusetts Board show with what uniform success these trials have been conducted. While more often settled in the lower courts, occasional appeal cases are carried to the superior courts, where the services of the regular district attorney are of course availed of in prosecuting the case.

Such a system as the above, while admirable for a state or city after long experience in the enforcement of food laws in the courts, is obviously impracticable with newly established systems of state food inspection.



## CHAPTER II.

### THE LABORATORY AND ITS EQUIPMENT.

**Location.**—The selection of a location for a food laboratory cannot always be made solely with reference to its needs and its convenience, but it is more often subject to economic conditions beyond the analyst's control. Under very best conditions, such a laboratory should be situated in a building designed from the start exclusively for chemical or biological and chemical work. Almost any well-lighted rooms in such a building can be readily adapted for the purpose. When, however, as is frequently the case, rooms for such a laboratory are provided in municipal, government, or office buildings, in which for the most part clerical work is done, the problem of adequately utilizing such rooms so that they may not at the same time prove offensive to or interfere with the comfort of other occupants of the building is sometimes difficult. It is obvious that basement rooms in such a building, as far as ventilation is concerned, are less readily adapted for the requirements in hand than are those of the top floor, though, if the light is good and there are abundant and well-arranged ventilating-shafts, such rooms may be made to serve every purpose. In the basement one may most easily obtain water, gas, and steam, and dispose of wastes without annoyance to one's neighbors. When, however, it is possible to do so, rooms on the top floor of an office building should be utilized for a food laboratory, for in such rooms the problems of lighting, heating and ventilating are comparatively simple and may usually be solved without regard to other occupants. In such a case ample provision must be made, preferably through shafts which are readily accessible for water-, gas-, steam-, and soil-pipes passing down below.

The actual equipment of the food laboratory depends of course largely on its particular purpose; and while it is manifestly impossible to do otherwise than leave the details to the individual taste and needs of the analyst,



modified by the means at his disposal, a few general suggestions regarding important essentials may prove helpful. These imply a fairly liberal though not extravagant outlay, with a view to saving both time and energy by convenient surroundings well adapted to the work in hand.

**Floor.**—The best material for the floor of the working laboratory is asphalt. Such a floor is firm but elastic, is readily washed by direct application of running water, if necessary, and resists well the action of ordinary reagents. An occasional thin coating of shellac with lampblack applied with a brush gives the asphalt floor a smooth, hard surface and may be applied locally to cover spots and blemishes.

**Lighting.**—The ideal arrangement is with benches for analytical work running north and south, the principal light being from south windows, and with benches for microscopes, balances, colorimeters, and standard solutions along the north wall where the north windows admit a soft light and never direct sunshine.

#### FIXTURES.

**Ventilation** by forced draft is a great convenience. For this purpose an exhaust fan driven by an electric motor and controlled in speed by a fractional rheostat is admirable. Such a fan would best be located in a small closed compartment or closet near the centre of the series of rooms designed to be ventilated by it, and this closet should have directly over the fan an outlet-shaft passing through the roof of the building. With such a system, a series of branching air-ducts should radiate from the fan closet, conveniently arranged either above or along the ceiling and communicating with the various hoods, closets, and rooms near the top.

**Benches.**—The working benches should have wooden or glazed tile tops. White glazed tile, if properly laid, furnish a very clean, sanitary, and resistant surface, besides being often convenient for color tests. If laid on a plank surface, cement should not be applied directly, as it swells the wood before drying out and results in a loose and often uneven surface. Cement may be avoided altogether and the tiles after first soaking in oil may be laid in putty directly on the wood. Tiles may be laid in cement by first covering the plank surface with cheap tin plate, overlapping the edges and securing by tacks. This prevents swelling of the wood. The tin may be covered to advantage with cheap paint. The tiles may then be embedded in a layer of cement spread over the tin surface.

Soft encaustic glazed tiles commonly used for wall finish are not as



effective as hard floor tiles specially glazed, since the former crackle and lose color when subjected to heat. A suitable material for the top of the titration bench is opal plate glass with a polished surface. Jet-black plate glass with a honed surface is admirably suited for the microscope table.

When wooden bench tops are used they may be treated to advantage by staining with the following solutions:

*Solution 1.* 100 grams of anilin hydrochloride, 40 grams of ammonium chloride, 650 grams of water.

*Solution 2.* 100 grams of copper sulphate, 50 grams of potassium chlorate, 615 grams of water.

Apply solution 1 thoroughly to the bare wood and allow it to dry; then apply 2 and dry. Repeat these applications several times. Wash with plenty of hot soap solution, let dry and rub well with vaseline. It is claimed that wood so treated is rendered fire-proof and is not acted on by acids and alkalies. When the finish begins to wear, an application of hot soap solution or vaseline will bring back the deep black color.

Gas and water outlets, sinks and waste pipes should be conveniently arranged, while the space beneath the benches should be utilized for drawers and cupboards. A clear bench width of 24 inches is ample for most work; if wider there is a temptation to allow apparatus to accumulate at the back. At the back of the bench and within easy reach, a raised narrow shelf should be provided to be used *exclusively* for common desk reagents. This again should not be so wide as to allow the accumulation of useless bottles. A narrow raised guard or beading at the edge of the reagent shelf prevents the bottles from accidentally slipping off.

**Hoods.**—Closed hoods with sliding sash fronts are almost indispensable. These hoods should be directly connected with the ventilating shafts or pipes, or with the air-ducts that radiate from the exhaust-fan closet, when such a system is provided. Gas outlets inside the hoods are necessary.

When there is a good draft, either natural or forced, a hooded top over the working bench, such as that shown in Fig. 3, is quite as efficient as a closed hood for most purposes. This is best made of galvanized iron, painted on the outside and treated on the inside with a preparation of graphite ground in oil. Here are best carried out all the processes involving the giving off of fumes and gases, which, if the ventilation is efficient, should pass directly up the flues and not come out in the room.



**Sinks and Drains.**—The sinks should preferably be of iron or porcelain. If iron, they should at frequent intervals be treated with a coat of



FIG. 3.—Hooded Top of Galvanized Iron over Working-bench, Connected with Ventilating Air-ducts.

asphalt varnish. A great convenience is a hooded sink (Fig. 4) in which foul-smelling bottles, or vessels giving off noxious or offensive fumes



or gases, may be rinsed under the tap while completely closed in. Open-work rubber mats at the bottom of the sinks help to insure against breakage. Open plumbing of simplest design should be used, and a multiplicity of traps should be avoided. Sinks may be variously located for

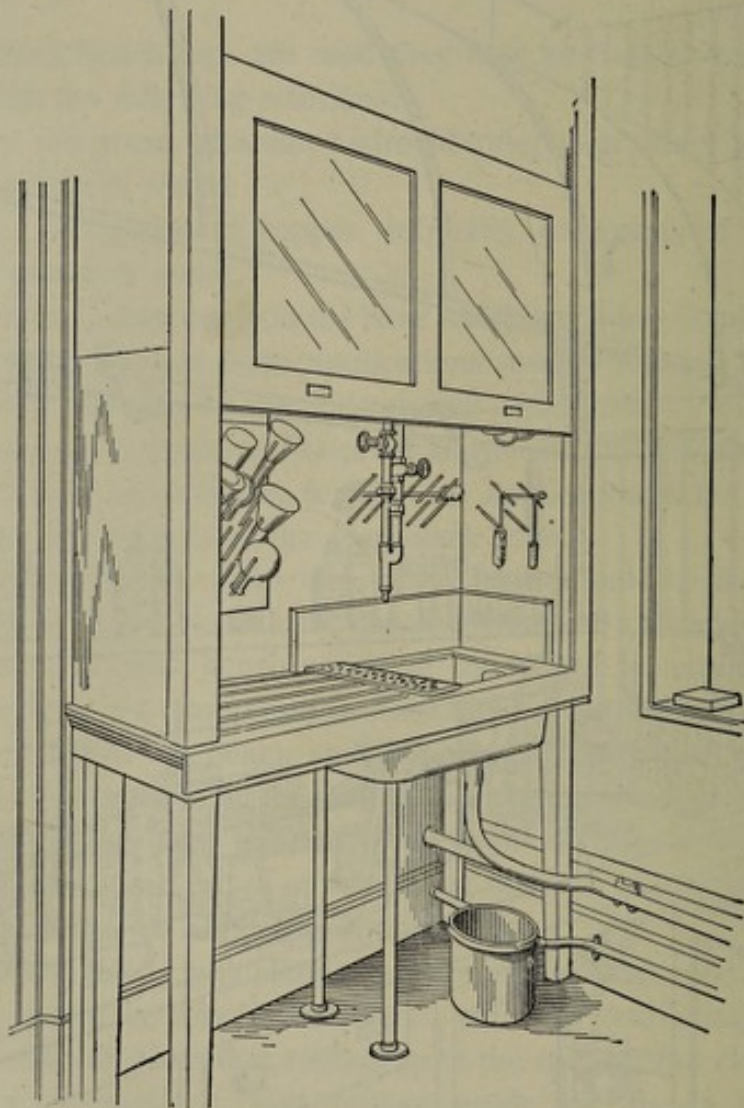


FIG. 4.—A Hooded Sink. An injector-like arrangement of steam and cold-water pipes furnishes water of any desired temperature.

convenience without regard to situation of soil-pipes, if the floor is thick enough to allow an open drain with sufficient pitch to flow readily. Such open drains are much more readily cleaned than closed pipes, and are best constructed by splitting a lead pipe and laying it in an iron box which is sunk into the floor. The edges of the lead pipe are rounded over those of the box as in Fig. 5, filling the joints with hydraulic cement, and the top of the drain is covered by a series of readily removable iron plates



flush with the top of the floor. Waste-pipes from sinks, still-condensers, refrigerators, and various forms of apparatus involving flowing water may be led into this drain, holes being drilled in the iron cover for their insertion.

**Gas, Electricity, and Steam.**—While formerly gas, made either in public or private plants, was the sole dependence for laboratory work, to-day gas, electricity, and steam are often on tap in the same laboratory, for some processes one and for others another giving the best results. If only one can be had, gas is usually the cheapest and most satisfactory, but in many office buildings only electricity is available as it may be impracticable to pipe in gas from the city mains and against insurance regulations to make it on the premises from gasoline. Laboratories and

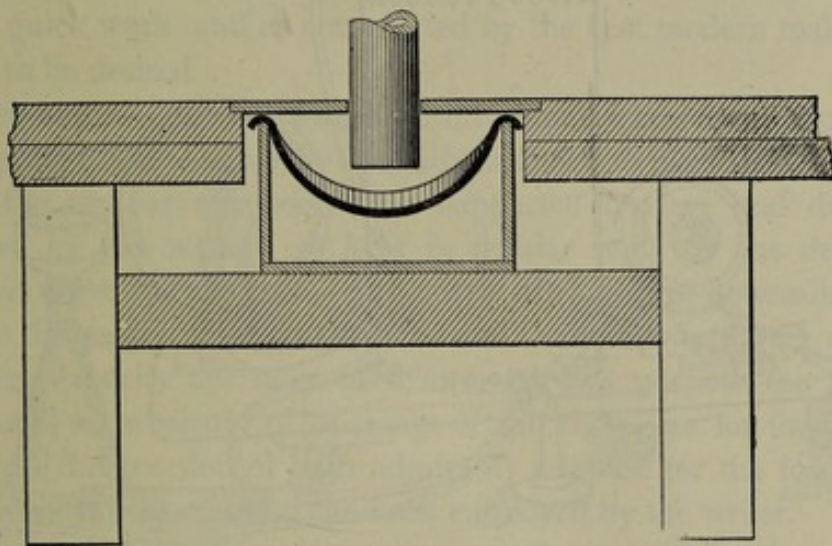


FIG. 5.—Section of Open Drain-pipe in Floor.

works remote from centers often have an abundance of home-generated electricity and steam, but no gas.

Fortunately electrical heaters for almost every kind of laboratory apparatus, such as furnaces, drying ovens, evaporators, thermostats, Kjeldahl digestors, and stills, are obtainable, although somewhat expensive. An electric current is also of great value in carrying out electrolytic methods and in running motors for driving centrifuges, shaking apparatus, ventilating fans, air pumps, etc. Whenever in an electrically equipped laboratory a free flame is indispensable, which is rarely the case, alcohol or blue flame kerosene oil burners are fairly satisfactory. Steam, when available, may be used to advantage for boiling ether or benzine in connection with continuous fat-extraction apparatus, for furnishing the motive power for driving the Babcock centrifuge, for heating water-baths and hot closets, and, in connection with cold water, to furnish a supply



of hot water when wanted at the sink. The latter application is illustrated in Fig. 4.

**Suction and Blast.**—If the water-pressure is ample, both air-pressure and exhaust for blast-lamps, vacuum filtration, and other purposes are readily available through the agency of the various devices used in connection with the flow of water, as, for instance, the Richards pump. When however, the water pressure is insufficient, other means must be employed for furnishing these much-needed requisites. Fig. 6 illustrates a simple

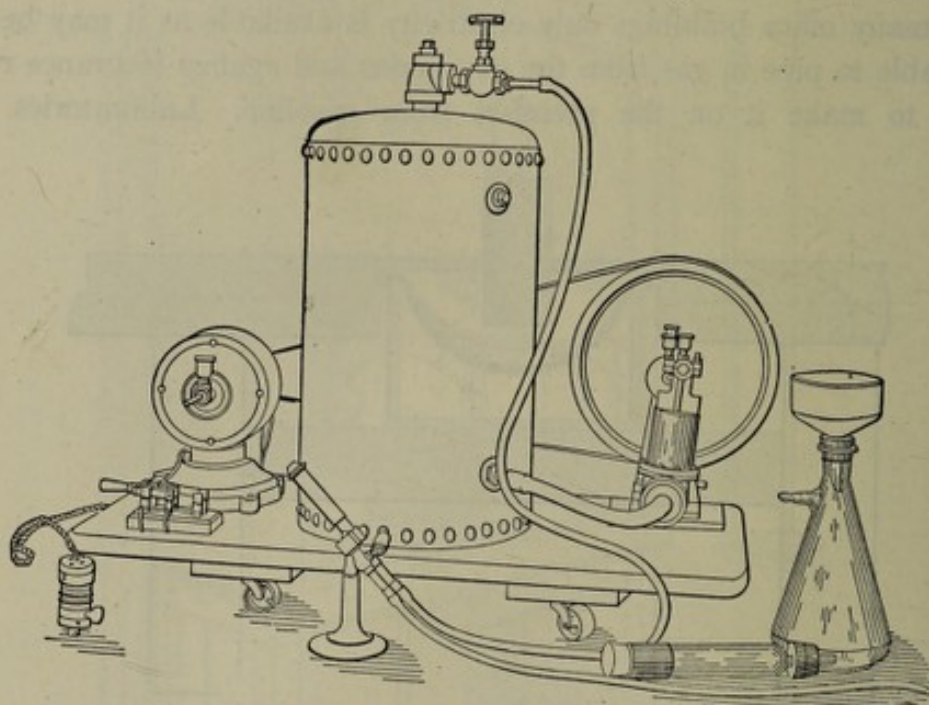


FIG. 6.—Portable Pressure- and Exhaust-pump Run by Electric Motor. Useful for blast-lamps, vacuum filtration, etc.

and almost noiseless pressure and exhaust pump run by a  $\frac{1}{8}$ -H.P. electric motor, which with the pressure-equalizing tank and the appropriate connections are mounted on a light wheel truck, and readily movable to any part of the laboratory. By simply screwing the plug into an electric-light outlet, either suction or blast may be had at will, depending on the position of a knife-edge switch which determines the direction of the current. By means of a fractional rheostat the speed may be varied and the pressure thus controlled.

#### APPARATUS.

The laboratory is of course to be supplied with the usual assortment of test-tubes, flasks, beakers, evaporating and other dishes of porcelain, platinum and glass, funnels, casseroles, crucibles, mortars, burettes,



pipettes, graduates, rubber and glass tubing, lamps, ring-stands and various supports, clamps and holders, the nature, number, and sizes of which are determined by individual requirements. Special forms of apparatus peculiar to certain processes of analysis or to the examination of special foods will be described in their appropriate connection. The following apparatus of a general nature may be regarded as indispensable for the proper fitting out of the food laboratory:

**Balances.**—These should include (1) an open pan balance for coarse weighing, having a capacity up to 1 kilogram and sensitive to 0.1 gram, with a set of weights; and (2) an analytical balance, enclosed in a case, sensitive to .0001 gram under a load of 100 grams, with an accurate set of non-corrosive weights. The short-beam analytical balance is preferable for quick work, and as constructed by the best modern makers leaves nothing to be desired.

**Water-baths.**—These are such an important accessory to food analysis that they should, if possible, be specially designed to meet the requirements, though the ordinary copper baths, supported on legs and designed to be heated by gas-burners, as kept in regular stock by the dealers, will sometimes serve the purpose. For nearly all moisture determinations the platinum dishes described on page 119 and the somewhat larger wine-shells of 100 cc. capacity are most used, and for this purpose the top of the bath should have plenty of openings of the right size for these. A very economical construction of bath admirably adapted for the food analyst's use is shown in Fig. 7, being the form employed by the writer.

The size and number of openings are determined by the number of samples to be simultaneously analyzed. A steam coil within the body of the bath serves to boil the water. Fig. 7 also shows the hood for carrying off the steam and fumes, the sliding front of which is furnished with a hasp and a padlock, so that it may always be kept locked by the analyst whenever he is temporarily absent from the laboratory. This is a useful precaution, when the residues left thereon are from samples which are to form subjects for possible prosecution in court later.

Steam, if available at all seasons of the year, or electric immersion coils furnish a ready means of heating the bath. In the absence of both steam and electricity, the bath must be boiled by gas burners.

**Drying-oven.**—Water ovens heated by gas and steam ovens are commonly used, although the drying cell seldom reaches a temperature above 98° C. The electric oven shown in Fig. 8 obviates this difficulty, the regulator permitting of adjustment so that full 100°, as well as any de-



sired temperature can be attained. Fig. 9 shows an asbestos-covered, jacketed air-oven, heated by a gas burner, with an efficient form of gas-pressure regulator.

**Water-still.**—An efficient still should be provided, capable of supply-

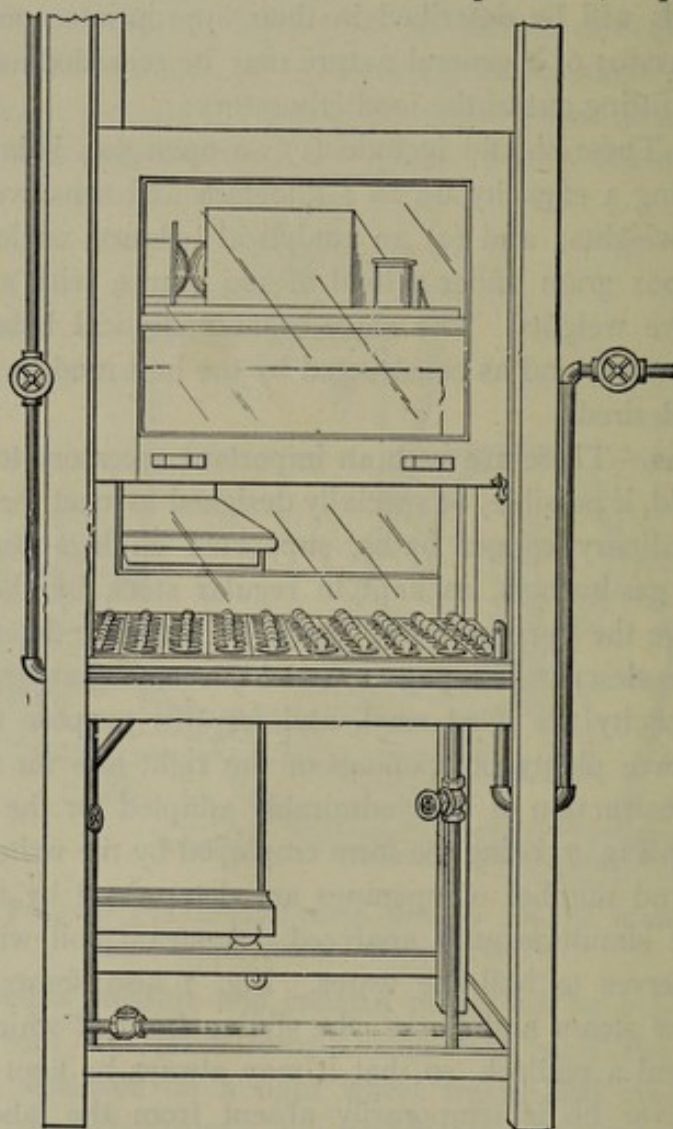


FIG. 7.—Water-bath, Enclosed in Hood, with Sliding-sash Front.

ing the laboratory with an ample quantity of pure water for analytical purposes. Fig. 10 illustrates a compact form of still, which is particularly economical in view of the fact that a single stream of inflowing cold water first serves to cool the condenser, and, rising, becomes vaporized in the boiler directly connected with the condenser at the top. This apparatus is capable of distilling six gallons of water in twelve hours.

**Universal Centrifuge.**—This convenient apparatus merits a separate brief description, being useful for a wide variety of purposes, such as



breaking up ether and other emulsions, quickly settling out precipitates, and roughly estimating chlorides, sulphates, phosphates, etc., by the volume of the precipitate in graduated tubes.

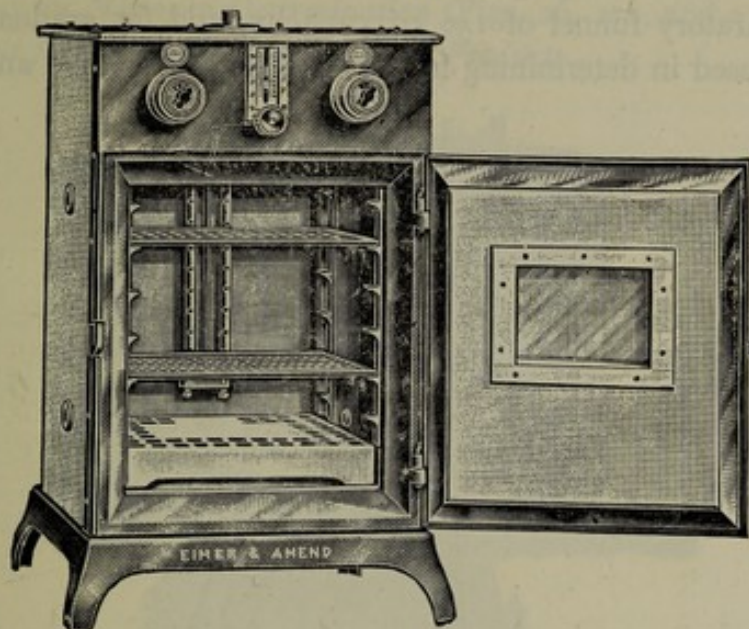


FIG. 8.—Freas Electrically Heated Drying Oven with Accurate Temperature Control.

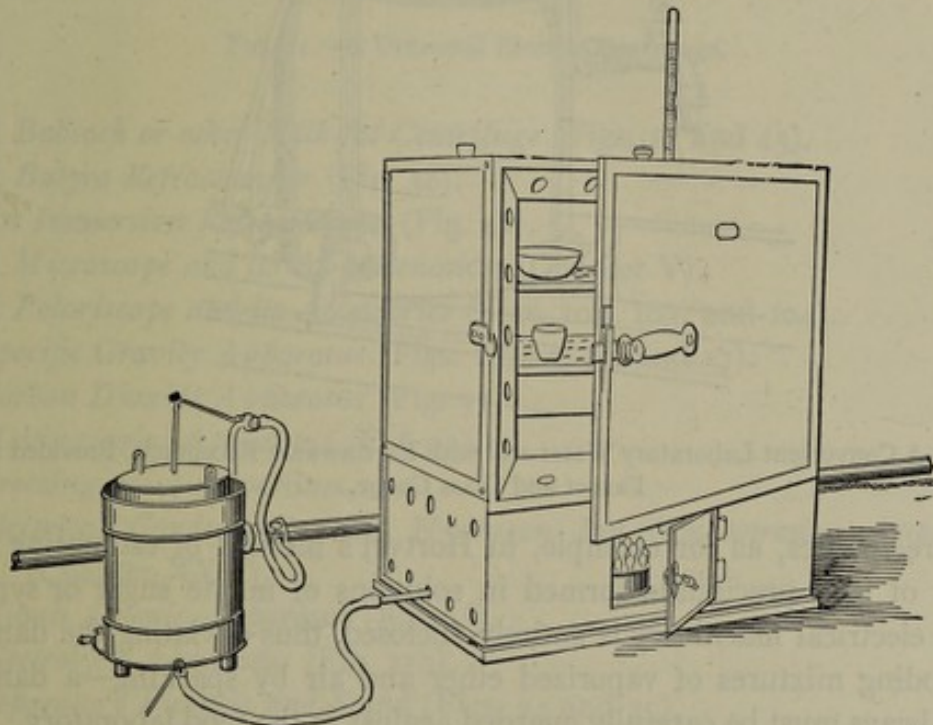


FIG. 9.—Asbestos-covered Air-oven, with Gas-pressure Regulator.

The centrifuge (Fig. 11) is inclosed within a cast-iron case and is driven by an electric motor concealed at the base. The vertical spindle



is provided with interchangeable heads carrying various forms of swinging holders for tubes, bottles, beakers, and separatory funnels. Holders are obtainable for tubes ranging from 2 cc. to 200 cc. capacity, for Squibb's form of separatory funnel of 150 cc. capacity, and for graduated bottles such as are used in determining fat by the Babcock method and in meas-

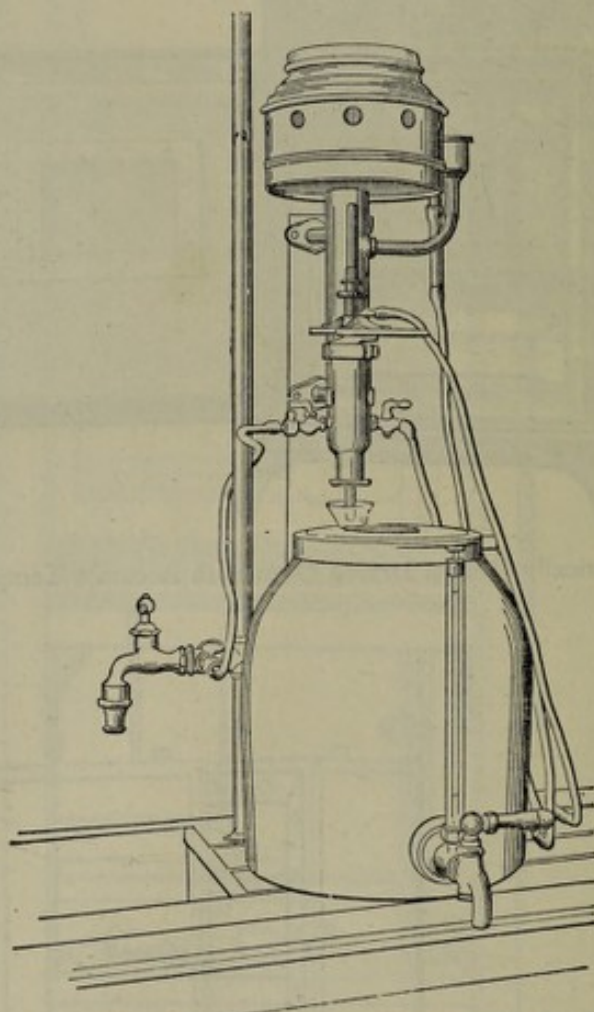


FIG. 10.—A Convenient Laboratory Water-still with Earthenware Receptacle, Provided with Faucet and Glass Gauge.

uring precipitates, as for example, in Hortvet's method of estimating the amount of lead precipitate formed in solutions of maple sugar or syrup.

The electrical machinery is entirely enclosed, thus obviating the danger of exploding mixtures of vaporized ether and air by sparking—a danger which always must be carefully guarded against in the food laboratory.

The various types of centrifuges designed for the Babcock test (page 124) may also be used for general work especially if fitted with interchangeable heads carrying different forms of holders.



**Other Special Apparatus.**—The following list includes pieces which are more or less indispensable:

*Continuous Extraction Apparatus* (Figs. 20, 21, and 22).

*Apparatus for Nitrogen Determination* (Figs. 26, 27a, and 27b).

*Apparatus for Distilling Various Food Products*.

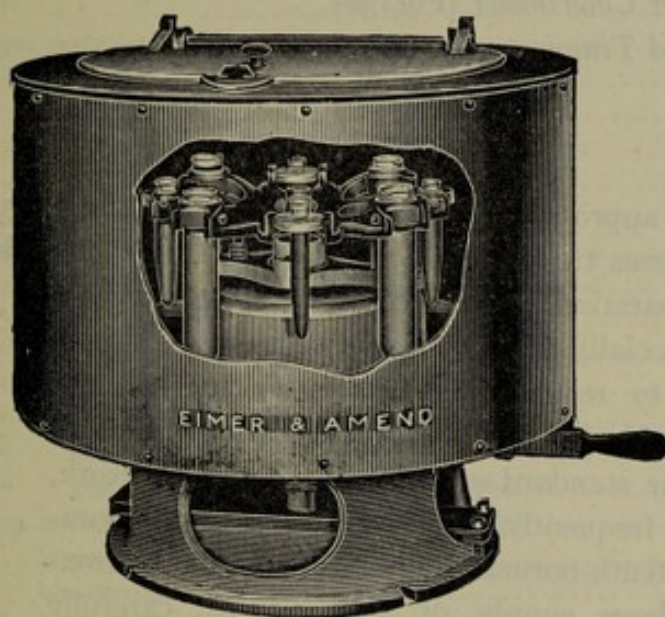


FIG. 11.—A Universal Electric Centrifuge.

*A Babcock or other Milk-fat Centrifuge* (Figs. 11 and 45).

*A Butyro Refractometer* (Fig. 38).

*An Immersion Refractometer* (Fig. 42).

*A Microscope and its Appurtenances* (Chapter V).

*A Polariscope and its Accessories* (Figs. 102, 103, and 104).

*Specific Gravity Apparatus* (Figs. 14, 15, 16, and 17).

*Carbon Dioxide Apparatus* (Fig. 71).

*Melting-point Apparatus* (Fig. 93).

*Freezing-point Apparatus*.

*Electrical Conductivity and Hydrogen Ion Concentration Apparatus*  
(Chapter XXII).

*Marsh Arsenic Apparatus* (Fig. 28).

*Electrolytic Apparatus* (Fig. 110).

*Separatory Funnels and Stand* (Figs. 24 and 25).

*A Spectroscope*, either of the direct-vision variety for the pocket, or the Kirschhoff & Bunsen style on a stand.

*Spectroscope Cells*, parallel-sided, for observation of absorption spectra.



*A Photomicrographic Camera and Appurtenances* \* (pp. 80. to 85).

*A Muffle Furnace*, gas (Fig. 3), or, preferably, electric (Fig. 19).

*An Ebullioscope* (Fig. 113).

*An Assay Balance*, for weighing arsenic mirrors to 0.01 mg.

*An Abbé Refractometer* (Fig. 39).

*A Schreiner Colorimeter* (Fig. 30).

*A Lovibond Tintometer* (p. 67).

#### REAGENTS.

Under the appropriate methods are described the reagents for carrying out the processes treated of in this volume, together with their strength, mode of preparation when necessary, and other data. Reagents, especially those constantly employed, should be assigned to regular places on the shelves, and should invariably be kept in place when not in use.

Among the standard solutions for volumetric work, none is more frequently of service in the food laboratory than a tenth-normal solution of sodium hydroxide, and a large supply of this reagent, carefully standardized, should be at all times conveniently at hand. Besides being useful for standardizing tenth-normal solutions, it is constantly needed for determining various acids in food products, such as milk, vinegar, butter, lime juice, cream of tartar, liquors, and many others. Time is well spent in carefully adjusting this solution to its exact tenth-normal value, thus simplifying the calculation of results. A large stock bottle (say of two gallons capacity) containing the standard tenth-normal sodium hydroxide, is conveniently mounted with a side-tube burette in connection, in some such manner as shown in Fig. 12. A small connecting side bottle contains a strong solution of sodium hydroxide through which the air that enters the large bottle is passed, thus depriving it of  $\text{CO}_2$ . In this manner the standard solution may readily be kept of unvarying strength for a year or more.

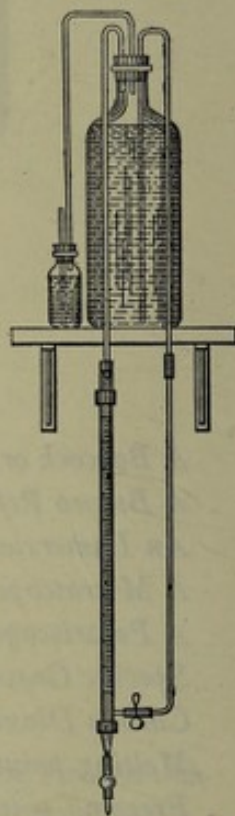


FIG. 12.—Stock Bottle of Tenth-normal Alkali.

\* A photographic dark room is also necessary if photomicrographic work is to be done.



## EQUIVALENTS OF STANDARD SOLUTIONS.

DECINORMAL SULPHURIC ACID. One cc. is equivalent to

Ammonia gas.....	$\text{NH}_3$ .....	0.0017	gram
Ammonia.....	$\text{NH}_4\text{OH}$ .....	0.0035	"
Ammonium carbonate.....	$(\text{NH}_4)_2\text{CO}_3$ .....	0.0046	"
" ".....	$(\text{NH}_4)_2\text{CO}_3 \cdot \text{H}_2\text{O}$ .....	0.0057	"
Calcium carbonate.....	$\text{CaCO}_3$ .....	0.0050	"
Calcium hydroxide.....	$\text{Ca}(\text{OH})_2$ .....	0.0037	"
" oxide.....	$\text{CaO}$ .....	0.0028	"
Lead acetate.....	$\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$ .....	0.0189	"
Magnesia.....	$\text{MgO}$ .....	0.0020	"
Magnesium carbonate.....	$\text{MgCO}_3$ .....	0.0042	"
Nitrogen.....	$\text{N}$ .....	0.0014	"
Potassium acetate *.....	$\text{KC}_2\text{H}_3\text{O}_2$ .....	0.0098	"
" bicarbonate.....	$\text{KHCO}_3$ .....	0.0100	"
" bitartrate *.....	$\text{KHC}_4\text{H}_4\text{O}_6$ .....	0.0188	"
" carbonate.....	$\text{K}_2\text{CO}_3$ .....	0.0069	"
" citrate *.....	$\text{K}_3\text{C}_6\text{H}_5\text{O}_7 \cdot \text{H}_2\text{O}$ .....	0.0106	"
" hydroxide.....	$\text{KOH}$ .....	0.0056	"
" and sodium tartrate.....	$\text{KNaC}_4\text{H}_4\text{O}_6 \cdot 4\text{H}_2\text{O}$ .....	0.0141	"
Sodium acetate.....	$\text{NaC}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$ .....	0.0136	"
" benzoate *.....	$\text{NaC}_7\text{H}_5\text{O}_2$ .....	0.0144	"
" bicarbonate.....	$\text{NaHCO}_3$ .....	0.0084	"
" borate.....	$\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ .....	0.0191	"
" carbonate.....	$\text{Na}_2\text{CO}_3$ .....	0.0053	"
" ".....	$\text{Na}_2\text{CO}_3 \cdot 10\text{H}_2\text{O}$ .....	0.0143	"
" hydroxide.....	$\text{NaOH}$ .....	0.0040	"
" salicylate *.....	$\text{NaC}_7\text{H}_5\text{O}_3$ .....	0.0160	"

DECINORMAL SODIUM HYDROXIDE SOLUTION. One cc. is equivalent to

Acid, acetic.....	$\text{H}_3\text{C}_2\text{H}_3\text{O}_2$ .....	0.0060	gram
" boric.....	$\text{H}_3\text{BO}_3$ .....	0.0062	"
" citric.....	$\text{H}_3\text{C}_6\text{H}_5\text{O}_7 \cdot \text{H}_2\text{O}$ .....	0.0070	"
" hydrobromic.....	$\text{HBr}$ .....	0.0081	"
" hydrochloric.....	$\text{HCl}$ .....	0.00365	"
" hydriodic.....	$\text{HI}$ .....	0.0128	"
" lactic.....	$\text{HC}_3\text{H}_5\text{O}_3$ .....	0.0090	"
" malic.....	$\text{C}_4\text{H}_6\text{O}_5$ .....	0.0067	"
" nitric.....	$\text{HNO}_3$ .....	0.0063	"
" oxalic.....	$\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ .....	0.0063	"
" phosphoric.....	$\text{H}_3\text{PO}_4$ { to form $\text{K}_2\text{HPO}_4$ with } phenolphthalein }	0.0049	"
" phosphoric.....	$\text{H}_3\text{PO}_4$ { to form $\text{KH}_2\text{PO}_4$ with } methyl orange }	0.0098	"
" sulphuric.....	$\text{H}_2\text{SO}_4$ .....	0.0049	"
" tartaric.....	$\text{H}_2\text{C}_4\text{H}_4\text{O}_6$ .....	0.0075	"
Potassium bitartrate.....	$\text{KHC}_4\text{H}_4\text{O}_6$ .....	0.0188	"
Sodium borate.....	$\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ .....	0.00955	"

\* To be ignited.



## DECINORMAL IODINE SOLUTION. One cc. is equivalent to

Arsenious oxide. ....	$\text{As}_2\text{O}_3$ .....	0.00495	gram
Potassium sulphite. ....	$\text{K}_2\text{SO}_3 \cdot 2\text{H}_2\text{O}$ .....	0.0097	"
Sodium bisulphite. ....	$\text{NaHSO}_3$ .....	0.0052	"
" sulphite. ....	$\text{Na}_2\text{SO}_3 \cdot 7\text{H}_2\text{O}$ .....	0.0126	"
" thiosulphate. ....	$\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ .....	0.0248	"
Sulphur dioxide. ....	$\text{SO}_2$ .....	0.0032	"
Sulphurous acid.....	$\text{H}_2\text{SO}_3$ .....	0.0041	"

## DECINORMAL SODIUM THIOSULPHATE SOLUTION. One cc. is equivalent to

Bromine.....	Br. ....	0.0080	gram.
Chlorine.....	Cl. ....	0.00355	"
Iodine.....	I. ....	0.01266	"
Iron (in ferric salts).....	Fe. ....	0.0056	"

## DECINORMAL SILVER NITRATE SOLUTION.\* One cc. is equivalent to

Ammonium bromide.....	$\text{NH}_4\text{Br}$ .....	0.0098	gram.
" chloride. ....	$\text{NH}_4\text{Cl}$ .....	0.00535	"
Chlorine.....	Cl. ....	0.00355	"
Cyanogen.....	$(\text{CN})_2$ .....	0.0052	"
Hydrocyanic acid.....	HCN with indicator.....	0.0027	"
" ".....	HCN { to formation of precip- itate.....	0.0054	"
Hydrobromic acid. ....	HBr. ....	0.0080	"
Potassium bromide. ....	KBr. ....	0.0119	"
" chloride.....	KCl. ....	0.00745	"
" cyanide. ....	KCN with indicator.....	0.0065	"
" ".....	KCN { to formation of precip- itate.....	0.0130	"
Sodium bromide.....	NaBr. ....	0.0103	"
" chloride.....	NaCl. ....	0.00585	"

## DECINORMAL POTASSIUM BICHROMATE SOLUTION.† One cc. is equivalent to

Ferrous carbonate. ....	$\text{FeCO}_3$ .....	0.0116	gram.
Ferric oxide.....	$\text{Fe}_2\text{O}_3$ .....	0.0080	"
Ferrous oxide. ....	$\text{FeO}$ .....	0.0072	"
" sulphate.....	$\text{FeSO}_4$ .....	0.0152	"
" ".....	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ .....	0.0278	"
Iron (ferrous).....	Fe. ....	0.0056	"

## DECINORMAL POTASSIUM PERMANGANATE SOLUTION. One cc. is equivalent to

Oxalic acid. ....	$\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ .....	0.0063	gram,
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and to same weights for iron salts as given under  $\text{N}/10 \text{ K}_2\text{Cr}_2\text{O}_7$ .

\* Use potassium chromate solution as an indicator, or add till precipitate appears.

† Use a freshly prepared solution of potassium ferricyanide as an indicator, applying a drop of titrated solution to a drop of indicator on a white surface.



The following table from Talbot\* shows the reactions of the common indicators used in acidimetry:

Indicator.	Reaction with Acids.	Reaction with Alkalies.	Use with Carbonic Acid in Cold Solution.	Use with Carbonic Acid in Hot Solution.	Use with Ammonium Salts.	Use with Organic Acid.
Litmus. ....	Red	Blue	Unreliable	Reliable	Reliable	Reliable
Methyl orange. ...	Pink	Yellow	Reliable	Unreliable	Reliable	Unreliable
Phenolphthalein. .	Colorless	Pink	Unreliable	Reliable	Unreliable	Reliable
Lacmoid. ....	Purple-red	Blue	Unreliable	Reliable	Reliable	Unreliable (?)
Cochineal. ....	Purple-red	Blue	Reliable	Reliable	Reliable	Unreliable
Rosolic acid. ....	Yellow	Pink	Unreliable	Reliable	Unreliable	Unreliable†
Alizarine. ....	Yellow	Red	Unreliable	Reliable	Reliable	Reliable

\* Talbot, Quantitative Analysis, page 75.

† Reliable with oxalic acid.



### CHAPTER III.

#### FOOD, ITS FUNCTIONS, PROXIMATE COMPONENTS, AND NUTRITIVE VALUE.

**Nature and General Composition.**—Food is that which, when eaten, serves by digestion and absorption to support the functions and powers of the body, by building up the material necessary for its growth and by supplying its wastes. The raw materials that constitute our food-supply are not all available for nourishment, but often contain a proportion of inedible or refuse matter, which it is customary to remove before eating, such as the bones of fish and meat, the shells of clams and oysters, eggshells, the bran of cereals, and the skins, stones, and seeds of fruits and vegetables. The proximate components which make up the edible portion of food include in general water, fat, various nitrogenous bodies consisting chiefly of proteins, carbohydrates, organic acids, and mineral matter. Of these water is hardly to be considered as a nutrient, though it plays an important part in nearly all foods as a diluent and solvent. The fats, proteins, and carbohydrates all contribute in varying degree to the supply of fuel for the production of heat and energy. Besides this universal function, the fats and the carbohydrates serve especially to furnish fatty tissue in the body, while the proteins are the chief source of muscular tissue.

Liebig's classification of foods into *nitrogenous*, or flesh formers, and *non-nitrogenous*, or heat generators, is now no longer accepted as strictly logical, in view of the well-known fact that the nitrogenous materials, besides building up the body, aid in supplying the wastes and yielding energy, and may even be converted into fats or carbohydrates, while the non-nitrogenous aid in furnishing tissue growth in addition to serving as fuel.

**THE FAT OF FOOD.**—Fats and oils consist essentially of the glycerides of the fatty acids, the characteristics of the various edible fats and



oils being treated under their appropriate headings elsewhere. Fat in human food is supplied by milk and its products, by the adipose tissue of meat, and in slight extent by the oil of cereals and by the edible table oils. The term "ether extract" is sometimes used in stating the results of the analysis of foods and this includes other substances than fat which when present are extracted by ether, such as chlorophyll and other coloring matters, lecithin, alkaloids, etc.

The glycerides occurring in foods are of acids belonging in four series as follows, the value for  $n$  being in parentheses:

**A. ACETIC SERIES** ( $C_nH_{2n}O_2$ ).—Butyric (4), caproic (6), caprylic (8), capric (10), lauric (12), myristic (14), palmitic (16), stearic (18), arachidic (20), behenic (22), and lignoceric (24).

**B. OLEIC SERIES** ( $C_nH_{2n-2}O_2$ ).—Hypogæic (16), oleic (18), isooleic (18), rapic (18), and erucic (22).

**C. LINOLIC SERIES** ( $C_nH_{2n-4}O_2$ ).—Linolic (18).

**D. LINOLENIC SERIES** ( $C_nH_{2n-6}O_2$ ).—Linolenic (18).

**E. CLUPANODONIC SERIES** ( $C_nH_{2n-8}O_2$ ).—Clupanodonic (18).

Fats contain not only simple glycerides, consisting of glycerol combined with three equivalents of the same fatty acid, but mixed glycerides with two or three acids in the same molecule. Other substances present are free fatty acids, lecithin, cholesterol, phytosterol, sitosterol, coloring matter, and other matters in minute amount.

**NITROGENOUS COMPOUNDS AND THEIR CLASSIFICATION.**—These substances may for convenience be grouped as follows:

A. Proteins, B. Amino-acids, Amides, Amines, etc., C. Alkaloids, D. Nitrates, E. Ammonia, F. Lecithin, and G. Cyan Compounds.

**A. PROTEINS.—Occurrence.**—Under the term proteins are included numerous bodies consisting, according to our present knowledge, essentially of combinations of  $\alpha$ -amino-acids and their derivatives. Proteins in one form or another exist in nearly all natural foods both animal and vegetable, but are supplied chiefly by the flesh of meat and fish, by milk, cheese, and eggs, and in the vegetable kingdom by grain, seeds, nuts, and vegetables, especially the legumes. The proportion of crude protein, often designated merely as "protein," is commonly estimated by multiplying by 6.25 the percentage of nitrogen found in the material analyzed. This is done on the assumption that all of the nitrogen present in the substance belongs to protein and that the protein contained 16 per cent of nitrogen, neither of which assumptions is usually true, although for most purposes the results are sufficiently accurate. In certain



cases, as for example, wheat flour and milk, special factors (5.70 and 6.38 in the cases cited) are used. Methods depending on the separation of the proteins as such are used in special investigations, but these, with few exceptions, are not adapted for practical purposes.

There is no marked distinction in chemical constitution between animal and vegetable proteins, although some of the types have as yet been found only in one or the other kingdom. The terms "proteids" or "albuminoids" were formerly used generically as synonymous with "protein" to include all nitrogenous bodies of this group, but in 1908 a joint committee on protein nomenclature of the American Physiological Society and the American Society of Biological Chemists recommended that the word "proteid" be abandoned; that "protein" be used to designate the entire group; and that the word "albuminoids" be restricted to a sub-group of proteins. A committee of the Physiological Society of England also made the same recommendation as to the use of the term protein. The classification and most of the definitions here given are those adopted by the American committee.\* The examples in most cases were kindly furnished by Dr. T. B. Osborne. For further details the reader is referred to the works of Mathews,† Osborne,‡ Plimmer,§ and Jones,|| also journal articles by Emil Fischer, Kossell, and their students.

**I. THE SIMPLE PROTEINS.**—Protein substances which yield only  $\alpha$ -amino acids or their derivatives on hydrolysis.

Although no means are at present available whereby the chemical individuality of any protein can be established, a number of simple proteins have been isolated from animal and vegetable tissues which have been so well characterized by constancy of ultimate composition and uniformity of physical properties that they may be treated as chemical individuals until further knowledge makes it possible to characterize them more definitely.

(a) **Albumins.**—Simple proteins soluble in pure water and coagulable by heat.

*Examples.*—Seralbumin of blood and other animal fluids; lactalbumin of milk; leucosin of the seeds of wheat, rye, and barley; legumelin of leguminous seeds.

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\* Amer. Jour. Phys., 21, 1908, xxvii.

† Physiological Chemistry, New York, 1916.

‡ The Vegetable Proteins, London, 1912.

§ The Chemical Constitution of the Proteins, London, 1917.

|| Nucleic Acids, London, 1914.



**Coagulation.**—Animal albumins usually coagulate at about 75°; vegetable albumins at about 65°.

**Miscellaneous Reactions.**—Very dilute acids precipitate albumins with the aid of heat. Nitrate of mercury (in dilute nitric acid) precipitates albumins from their solutions; also Mayer's solution acidified with acetic acid. They are precipitated by saturation with ammonium sulphate.

These reactions are not, however, characteristic of the group.

(b) **Globulins.**—Simple proteins insoluble in pure water, but soluble in neutral solutions of salts of strong bases with strong acids.

**Examples.**—Myosin of muscle substance; legumin of leguminous seeds; amandin of almonds.

**Qualitative Tests.**—Globulins are precipitated from their solution by dialysis or dilution. Albumins are not thus precipitated.

(c) **Glutelins.**—Simple proteins insoluble in all neutral solvents, but readily soluble in very dilute acids and alkalies.

**Examples.**—Glutenin of wheat is the only well defined protein of this group.

(d) **Prolamins.**—Simple proteins soluble in relatively strong alcohol (70-80 per cent), but insoluble in water, absolute alcohol, and other neutral solvents.

**Examples.**—Gliadin of wheat; zein of maize; hordein of barley. Found as yet only in the seeds of cereals.

The use of appropriate prefixes will suffice to indicate the origin of compounds of sub-classes a, b, c, and d, as for example: ovoglobulin, myalbumin, etc.

(e) **Albuminoids.**—Simple proteins which possess essentially the same chemical structure as the other proteins, but are characterized by great insolubility in all neutral solvents.

**Examples.**—Keratins of hair, nails, hoofs, horn, feathers, etc.; elastin of connective tissues; collagen of connective tissues and cartilage; fibroin and sericin of raw silk. No albuminoids have yet been discovered in plants.

**Gelatin** is usually regarded as an albuminoid but does not come strictly within the requirements of the above definition. It is an artificial derivative of collagen and is formed from it by boiling with water or subjecting to steam under pressure. It is prepared from bones and other animal parts, and is insoluble in cold, but soluble in hot water. When the hot water solution containing one per cent or more of gelatin cools, it forms a jelly. By prolonged boiling the gelatinizing power is lost. Aqueous solutions are strongly lævo-rotary.



Gelatin in common with most proteins is precipitated from its solution by mercuric chloride, picric acid, and tannic acid. It is readily distinguished from soluble proteins, in that it is not precipitated from its solution by lead acetate, nor by most of the metallic salts that throw down proteins.

(f) **Histones.**—Soluble in water and insoluble in very dilute ammonia, and, in the absence of ammonium salts, insoluble even in an excess of ammonia; yield precipitates with solutions of other proteins, and a coagulum on heating, which is easily soluble in very dilute acids. On hydrolysis they yield a large number of amino-acids, among which the basic ones predominate.

*Examples.*—Thymus histone. Not found in plants.

(g) **Protamins.**—Simpler polypeptides than the proteins included in the preceding groups. They are soluble in water, uncoagulable by heat, have the property of precipitating aqueous solutions of other proteins, possess strong basic properties, and form stable salts with strong mineral acids. They yield comparatively few amino-acids, among which the basic amino-acids greatly predominate.

*Examples.*—Salmin, clupein, and other protamins of fish spermatozoa. Not found in plants.

**II. CONJUGATED PROTEINS.**—Substances which contain the protein molecule united to some other molecule or molecules otherwise than as a salt.

(a) **Nucleoproteins.**—Compounds of one or more protein molecules with nucleic acid.

*Examples.*—The nucleins salmin nucleate and clupein nucleate.

(b) **Glycoproteins.**—Compounds of the protein molecule with a substance or substances containing a carbohydrate group other than a nucleic acid.

*Examples.*—Mucins; ovomucoid; ovalbumin; ichthulin.

(c) **Phosphoproteins.**—Compounds of the protein molecule with some yet undefined phosphorus-containing substance other than a nucleic acid or lecithins.

*Examples.*—Casein of milk; vitellin of egg yolk.

(d) **Haemoglobins.**—Compounds of the protein molecule with haematin or some similar substance.

*Example.*—Oxyhaemoglobin of red blood corpuscles.

(e) **Lecithoproteins.**—Compounds of the protein molecule with lecithins, (lecithans, phosphatides).

*Examples.*—Lecithalbumin; lecithin-nucleovitellin.



### III. DERIVED PROTEINS.

1. PRIMARY PROTEIN DERIVATIVES.—Derivatives of the protein molecule, apparently formed through hydrolytic changes which involve only slight alterations of the molecule.

(a) **Proteans**.—Insoluble products which apparently result from the incipient action of water, very dilute acids or enzymes.

*Examples*.—Edestan; blood fibrin; insoluble myosin.

(b) **Metaproteins**.—Products of the further action of acids or alkalies, whereby the molecule is so far altered as to form products soluble in very weak acids and alkalies, but insoluble in neutral fluids.

*Examples*.—Acid albumin; alkali albumin.

This group will thus include the familiar "acid proteins" and "alkali proteins," not the salts of proteins with acids.

(c) **Coagulated Proteins**.—Insoluble products which result from (1) the action of heat on their solutions, or (2) the action of alcohol on the protein.

*Examples*.—Albumin coagulated by heat or alcohol.

2. SECONDARY PROTEIN DERIVATIVES. Products of the further hydrolytic cleavage of the protein molecule.

(a) **Proteoses**.—Soluble in water, uncoagulated by heat, and precipitated by saturating their solutions with ammonium or zinc sulphate.

As thus defined this term does not strictly cover all the protein derivatives commonly called proteoses, e.g. heteroproteose and dysproteose.

*Subdivision of the Proteoses*.—According to the proteins from which they are derived the proteoses may be designated *albumose*, from albumin, *globulose*, from globulin, *vitellose*, from vitellin, *caseose*, from casein, etc.

Proteoses are subdivided into *proto-proteose*, soluble in water (both cold and hot) or in dilute salt solutions, but precipitated by saturation with salt; *hetero-proteose*, insoluble in water, and *deutero-proteose*, soluble in water, but not precipitated by saturation with salt.

Vegetable proteoses are sometimes called phyt-albumoses.

*Qualitative Tests*.—Besides responding to the biuret reaction (p. 34) proteoses are precipitated by nitric acid, the precipitate being soluble on heating, but reappearing on cooling.

Proto-proteose is precipitated from its solution by mercuric chloride and copper sulphate; hetero-proteose is precipitated by mercuric chloride, but not by copper sulphate.

(b) **Peptones**.—Soluble in water, uncoagulated by heat, and not precipitated by saturating their solutions with ammonium sulphate.



*Qualitative Tests.*—Besides giving the biuret reaction, peptones are precipitated from their solution by tannic acid, picric acid, phosphomolybdic acid, and by sodium phosphotungstate acidified by acetic, phosphoric, or sulphuric acid.

Peptones are the only soluble proteins not precipitated by saturation with ammonium sulphate.

(c) **Peptides.**—Definitely characterized combinations of two or more amino-acids, the carboxyl group of one being united with the amino group of the other, with the elimination of a molecule of water.

The peptones are undoubtedly peptides or mixtures of peptides, the latter term being at present used to designate those of definite structure.

**Qualitative Tests for Proteins.**—*Xanthoproteic Reaction.*—Concentrated nitric acid containing nitrous acid formed during standing added to a solution of a protein may or may not form a precipitate. It, however, produces a yellow coloration on boiling. Addition of ammonia in excess turns the precipitate or liquid yellow or orange; proteins in suspension also react.

*Millon's Reaction.*—Millon's reagent is prepared by dissolving metallic mercury in twice its weight of concentrated nitric acid, diluting with an equal volume of water, and allowing to settle. When added to a protein solution it produces a white precipitate, which becomes brick-red on heating. Solid proteins give the red color by direct treatment. Sodium chloride prevents the reaction. Various organic substances are precipitated by Millon's reagent, but these precipitates do not turn red on heating.

*Biuret Reaction.*—If a solution of a protein in dilute sulphuric acid be made alkaline with potassium or sodium hydroxide and *very dilute* copper sulphate be added, a reddish to violet coloration is produced, similar to that formed if biuret be treated in the same way, hence the name. An excess of copper sulphate should be avoided lest its color obscure that of the reaction.

In solutions which are strongly colored this reaction is of little use unless modified as follows: A considerable quantity of the dilute copper sulphate solution is added to the solution made alkaline with a large excess of potassium hydroxide, and then solid potassium hydroxide is dissolved to almost complete saturation in the solution. The mixture is then shaken with about one half its volume of strong alcohol. On standing the alcohol separates as a clear layer or a violet or crimson color if proteins are present.

**B. AMINO-ACIDS, AMIDES, AMINES, AND ALLIED PRODUCTS.**—Under this head are included products derived from acids or bases, the radicles



of which replace one or more hydrogen atoms in ammonia. The most common bodies of this class occurring in foods follow:

**I. AMINO-ACIDS**—The following are obtained by the hydrolysis of the different proteins: 1. glycocoll; 2. alanine; 3. valine; 4. leucine; 5. glyco-leucine, 6. iso-leucine, 7. serine, 8. cysteine, 9. aspartic acid, 10. glutamic acid, 11. arginine, 12. lycine, 13. cystine, 14. tyrosine, 15. phenyl-alanine, 16. proline, 17. oxy-proline, 18. histidine, 19, tryptophane. Of these, 1 to 13 inclusive belong to the aliphatic series, 14 and 15 to the carbocyclic series, and 16 to 19 inclusive to the heterocyclic series.

**II. AMIDES**.—Asparagin occurs in the young shoots of asparagus, lettuce and other green vegetables, and marshmallow root. Glutamine occurs in seeds during sprouting.

**III. AMINES**.—Choline is found in meat, egg yolk, and certain fungi. Betaine is a constituent of beets, hops, and certain mollusks. Carnitine occurs in meat extract.

**IV. CREATINE AND CREATININE**.—These are constituents of meat extracts.

**V. PURINE BASES**.—In the vegetable kingdom these are represented by the caffeine of tea, coffee, and cocoa, and the theobromine of cocoa, in the animal kingdom by xanthine, hypoxanthine, guanine, and adenine of meat and meat extracts. They are also classified with the alkaloids.

**C. ALKALOIDS**.—This group is characteristic of certain drugs; in foods they are of infrequent occurrence. Aside from the purine bases caffeine and theobromine, the piperine and piperidine of pepper are the only common examples.

**D. NITRATES**.—These occur mostly in growing parts of the plant and only in traces.

**E. AMMONIA**.—This occurs in ripened cheese of certain varieties and meat that is undergoing decomposition.

**F. LECITHIN**.—This is a phosphorized fat occurring in egg yolk and other animal and vegetable substances.

**G. CYAN COMPOUNDS**.—The bitter cassava contains hydrocyanic acid. Cyanides and sulphocyanides (thiocyanates) are found in small amounts, in various foods. Common examples of sulphocyanides are the pungent principles of mustard and horse radish. Amygdalin of bitter almonds is a glucoside containing the cyan group.

**CARBOHYDRATES AND THEIR CLASSIFICATION**.—Of the total number of carbohydrates which have been described only a limited number occur in food products and of these a considerable number do not exist in the



original vegetable or animal substance, but are formed during manufacture.

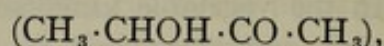
A classification of the common food carbohydrates is given below. Descriptions of the more important individuals appear in chapters X and XIV. Other details will be found in the works of Armstrong,\* and Browne,† as well as in special papers by Emil Fischer, Tollens, and their students.

**I. MONOSACCHARIDES.**—These, also known as simple carbohydrates, are either aldehyde alcohols (aldoses) or ketone alcohols (ketoses) with usually one carbonyl and one or more alcohol groups. One of the hydrogens of the end group  $\text{CH}_2\text{OH}$  may be replaced by an alkyl group, usually methyl. The formulæ of the *l*-forms are mirror images of the *d*-forms.

(a) **Dioses.**—No representative of this group occurs in foods, but an example is here given to illustrate the simplest form of monosaccharide.

*Example.*—Glycolose ( $\text{CH}_2\text{OH} \cdot \text{CHO}$ ), prepared synthetically.

(b) **Methyl Dioses.**—*Example.*—Dimethylglycolose

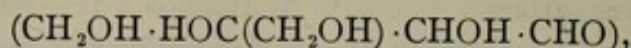


occurs in vinegar and other fermented products.

(c) **Trioses.**—*Example.*—Dioxyacetone ( $\text{CH}_2\text{OH} \cdot \text{CO} \cdot \text{CH}_2\text{OH}$ ), a ketose, is formed in various fermentation processes.

(d) **Tetroses.**—No example in food products.

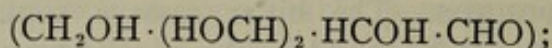
(e) **Methyl Tetroses.**—*Example.*—Apiose



a constituent of the glucoside apiin of parsley.

(f) **Pentoses** ( $\text{C}_5\text{H}_{10}\text{O}_5$ ).—These sugars occur seldom and in only small amounts in foods, but are prepared from the corresponding pentosans by hydrolysis.

**Aldoses.**—*Examples.*—*d*-Arabinose



*l*-arabinose ( $\text{CH}_2\text{OH} \cdot (\text{HCOH})_2 \cdot \text{HOCH} \cdot \text{CHO}$ ), a constituent of certain glucosides; *l*-xylose ( $\text{CH}_2\text{OH} \cdot \text{HOCH} \cdot \text{HCOH} \cdot \text{HOCH} \cdot \text{CHO}$ ); *d*-ribose ( $\text{CH}_2\text{OH} \cdot (\text{HOCH})_3 \cdot \text{CHO}$ ), a constituent of various nucleic acids.

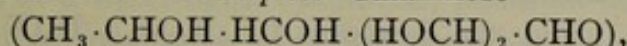
**Ketoses.**—Little studied.

\* Simple Carbohydrates, London, 1912.

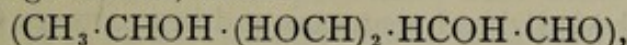
† Handbook of Sugar Analysis, New York, 1912.



(g) **Methyl Pentoses.**—*Examples.*—Rhamnose



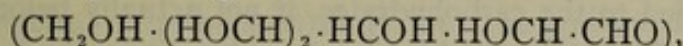
occurs in various glucosides; fucose



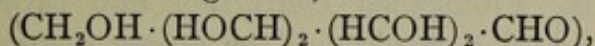
derived by hydrolysis of fucosan.

(h) **Hexoses** ( $\text{C}_6\text{H}_{12}\text{O}_6$ ).

**Aldoses.**—*Examples.*—*d*-glucose or dextrose

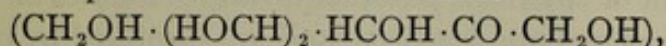


abundant in nature, forming with *d*-fructose invert sugar, occurs in numerous glucosides, formed by hydrolysis of starch, and is one of the chief constituents of commercial glucose; *d*-mannose

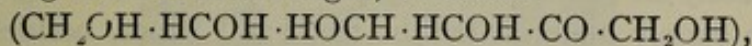


found in plant juices, germinating seeds, and molasses; *d*-galactose ( $\text{CH}_2\text{OH} \cdot \text{HOCH} \cdot (\text{HCOH})_2 \cdot \text{HOCH} \cdot \text{CHO}$ ), a constituent of certain glucosides, occurs free in whey and germinating seeds; *l*-galactose ( $\text{CH}_2\text{OH} \cdot \text{HCOH} \cdot (\text{HOCH})_2 \cdot \text{HCOH} \cdot \text{CHO}$ ); *d*, *l*-galactose or racemic galactose, identified in certain oriental food products.

**Ketoses.**—*Examples.*—*d*-Fructose or levulose



occurs with *d*-glucose in invert sugar; *d*-sorbose



formed by fermentation of the juice of the sorb apple; glucose, found in molasses.

**II. DISACCHARIDES.**—These yield on hydrolysis two monosaccharides. Their constitutional formulæ have not been fully decided on.

*Examples.*—Sucrose or common sugar ( $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ ); maltose ( $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ ) formed by the action of diastase on starch; lactose or milk sugar ( $\text{C}_{12}\text{H}_{22}\text{O}_{11} \cdot \text{H}_2\text{O}$ ); trehalose or mushroom sugar ( $\text{C}_{12}\text{H}_{22}\text{O}_{11} \cdot 2\text{H}_2\text{O}$ ) melibiose ( $\text{C}_{12}\text{H}_{22}\text{O}_{11} \cdot 2\text{H}_2\text{O}$ ), formed by action of yeast on raffinose. Of these maltose, lactose, and melibiose are copper reducing.

**III. TRISACCHARIDES.**—These yield on partial hydrolysis a monosaccharide and a disaccharide.

*Example.*—Raffinose ( $\text{C}_{18}\text{H}_{32}\text{O}_{16} \cdot 5\text{H}_2\text{O}$ ), occurs in sugar beets, cotton seed, etc.

**IV. TETRASACCHARIDES.**—These yield on partial hydrolysis a monosaccharide and a trisaccharide.

*Example.*—Stachyose ( $\text{C}_{24}\text{H}_{42}\text{O}_{21} \cdot 4\text{H}_2\text{O}$ ), found in various roots and in ash manna.



**V. POLYSACCHARIDES.**—This group includes the pentosans  $((C_5H_8O_4)_n \cdot H_2O)$  and the hexosans  $((C_6H_{10}O_5)_n \cdot H_2O)$ . The value of  $n$  is so large that the water may for practical purposes be ignored. For descriptions of the individual pentosans and hexosans see Chapter X.

(a) **Pentosans.**—*Examples.*—Araban; metaraban; xylan.

(b) **Hexosans.**—*Examples.*—Mannan; galactan; inulin; dextrin; starch; cellulose.

Closely allied to the carbohydrates, if not actually belonging to them, are *inosite*  $(C_6H_{12}O_6)$ , occurring in muscular tissue, and *pectose*, found in green fruits and vegetables.

**THE ORGANIC ACIDS.**—These acids are minor though important constituents of foods. From their conversion into carbonates within the body, they are useful in furnishing the proper degree of alkalinity to the blood and to the various other fluids, besides being of particular value as appetizers. They exist in foods both in the free state and as salts. Acetic acid is supplied by vinegar; lactic acid by milk, fresh meat, and beer; citric, malic, and tartaric acids by the fruits.

**MINERAL OR INORGANIC MATERIALS.**—These substances occur in food in the form of chlorides, phosphates, and sulphates of sodium, potassium, calcium, magnesium, and iron, and are furnished by common salt, as well as by nearly all animal and vegetable foods. The inorganic salts are necessary to supply material for the teeth and bones, besides having an important place in the blood and in the cellular structure of the entire body.

**FUEL VALUE OF FOOD.**—In order to express the capacity of foods for yielding heat or energy to the body, the term fuel value is commonly used. By the fuel value of a food material is meant the amount of heat expressed in calories equivalent to the energy which we assume the body could obtain from a given weight of that food material, if all of its nutrients were thoroughly digested, a calorie being the amount of heat required to raise a kilogram of water  $1^\circ C$ . This definition applies to what is known as the large calorie, which is one thousand times as large as the small calorie. Large calories are meant wherever the term occurs in this volume. The fuel value, or, as it is sometimes called, "heat of combustion," may be determined experimentally with a calorimeter, or may be calculated by means of factors based on the result of many experiments showing the mean values for protein, fats, and carbohydrates.

*The Bomb Calorimeter.\**—This apparatus in its most approved form,

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\* U. S. Dept. of Agric., Off. of Exp. Sta., Bul. 21, pp. 120-126.



Fig. 13, consists of a water-tight, cylindrical, platinum lined, steel bomb, adapted to hold in a capsule the substance whose heat is to be determined, and containing also oxygen under pressure. This bomb is immersed in water contained in a metal cylinder, which is in turn placed inside of concentric cylinders containing alternately air and water. The heat for igniting the substance is supplied by the electric current passing through wires to the interior of the bomb and acting upon a cleverly devised mechanism therein. The heat developed by the ignition is measured by

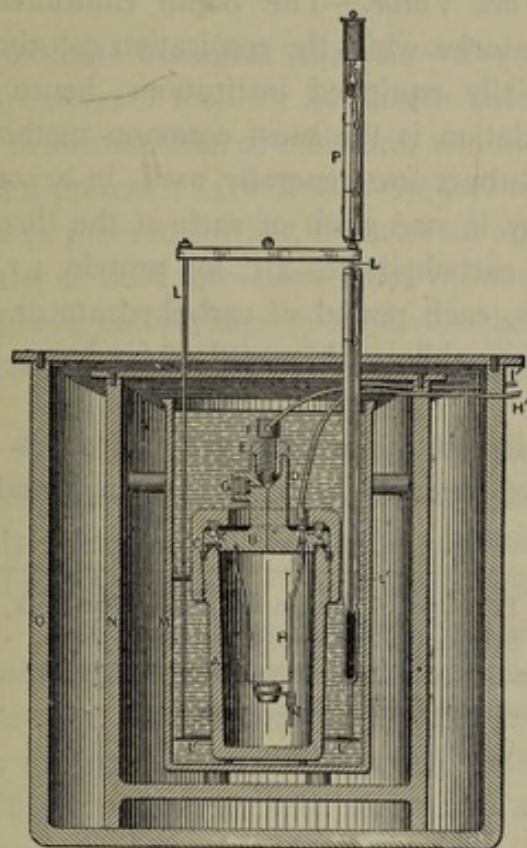


FIG. 13.—Bomb Calorimeter of Hempel and Atwater

the rise in temperature of the water surrounding the bomb, as indicated by a very delicate thermometer graduated to hundredths of a degree, certain corrections being made, as, for instance, for the heat absorbed by the metal of the apparatus. A mechanical stirrer serves to equalize the temperature of the water surrounding the bomb.

*The Respiration Calorimeter* is a combustion apparatus on a large scale, of which a living human being or animal confined in a tight chamber, is a part. The food is carefully weighed and analyzed and the oxygen is supplied in known amount from a cylinder to replace that consumed by oxidation in the lungs. The water and carbon dioxide exhaled are



absorbed in calcium chloride tubes and potash bulbs or their equivalents on a large scale while the excreta is collected, weighed, and analyzed. The heat produced is measured by delicate appliances. In the United States human calorimeters are maintained at the Carnegie Nutrition Laboratory, Boston, under the direction of F. G. Benedict and at The Department of Agriculture, Washington, under the direction of Langworthy. A calorimeter for farm animals is in operation at State College, Pennsylvania, by Armsby.

*Calculation of Fuel Value.*—The bomb calorimeter is beyond the reach of many laboratories while the respiration calorimeter can be maintained only in specially equipped institutions, hence the expression of fuel values by calculation is the most common method employed. For this the factors of Rubner are generally used, in accordance with which the amount of energy in one gram of each of the three principal classes of nutrients are, for carbohydrates 4.1, for protein 4.1, and for fats 9.3. Expressed in pounds, each pound of carbohydrate or protein has a fuel value of 1860 calories, while each pound of fat has a fuel value of 4220 calories.

For further details on the caloric value of foods and the science of nutrition the works of Jordan,\* Lusk,† Sherman,‡ and Snyder§ may be consulted.

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\* The Principles of Human Nutrition, New York, 1914.

† The Science of Nutrition, Philadelphia, 1917.

‡ Chemistry of Food and Nutrition, New York, 1918.

§ Human Food, New York, 1916.



## CHAPTER IV.

### GENERAL ANALYTICAL METHODS.

**Extent of a Proximate Chemical Analysis.**—For purposes of studying the proximate composition of food for its dietetic value, it is nearly always necessary to make determinations of moisture, ash, fat, total nitrogen, and carbohydrates (when present), as well as of the fuel value. In some cases it may be desirable to proceed further, to make an analysis of the ash, for instance, to separate, at least into classes, the various nitrogenous bodies, especially in flesh foods, and perhaps to subdivide the starch, sugar, gums, and cellulose or crude fiber that make up the carbohydrates in the case of cereals.

An analysis is considered complete whenever the purpose for which the examination has been made has been accomplished, and on that purpose depends solely the extent to which the various compounds present shall be subdivided and determined. Such a subdivision may be extended almost indefinitely. For example, a milk analysis may consist simply in the determination of the total solids and (by difference) the water. Again, it may be desirable to divide the milk solids into fat and solids not fat, and in some cases to carry the subdivision still farther and separate the solids not fat into casein, albumin, milk sugar, and ash.

Determinations of one or more of the proximate components natural to food are frequently of great service in proving their purity or freedom from adulteration. For the latter purpose, especially in such foods as milk, vinegar, oils, and fats, the determination of specific gravity is also an important factor. Special methods of a peculiar nature are often necessary in the examination of particular foods, and such methods will be treated subsequently under the appropriate headings. In the present chapter only such general methods as are applicable to a large variety of cases will be discussed.

**Expression of Results of a Proximate Analysis.**—However complete the division of the various proximate compounds or classes of compounds



which the analyst sees fit to make, the results of his various determinations in a proximate analysis are expected to aggregate about 100%. If every determination be directly made, the result will rarely be exactly 100, but the precision of the work is apt to be judged by its approach to 100.

It is often the custom to determine certain compounds or classes of compounds by difference. Thus in cereals moisture, proteins, fat, crude fiber and ash may be determined by the regular analytical methods, and by subtracting their sum from 100 the difference may be expressed as "nitrogen-free extract" or carbohydrates. It has long been customary in food analysis to calculate the protein by multiplying the total nitrogen by the factor 6.25, and on this basis analyses of thousands of animal and vegetable foods have been made. While the figure thus obtained is an arbitrary one, being at best but a rough approximation of the amount of protein present, yet for many reasons there is much to commend this practice of reporting results. In the first place, in most cases it actually does approach the truth. Again, the nitrogenous ingredients of many foods are so numerous and varied, that for the every-day study of dietaries and food values it would be well-nigh impossible with our present knowledge to subdivide these compounds with any degree of accuracy, and especially with uniformity between different chemists, to say nothing of the time involved.

From the fact that so many valuable analyses have already been expressed on the basis of  $N \times 6.25$  for protein, the advantage of comparison with the results thus recorded would seem to be in itself a good reason for continuing the practice, especially until a factor that gives better average results can be adopted. By recording the actual nitrogen found as well as the "protein," old results may at any time be recalculated under new conditions, if found desirable.

In flesh foods, when carbohydrates are known to be absent, the total protein may conveniently be determined by difference. Rather more progress has been made in the separation of the nitrogenous compounds of meats than of the vegetables and cereals, though the methods are by no means accurate or uniform.

Most of the recorded analyses of vegetable foods express the carbohydrates as a whole without attempting to subdivide them, at least further than possibly to express the crude fiber or cellulose separately. A much more intelligible idea of the dietetic value of these foods would be gained by a further separation into starch and sugars.



**Preparation of the Sample.**—It is at the outset of the utmost importance in all cases that a strictly representative portion of the food to be examined should be submitted to analysis. All refuse matter, such as bones, shells, bran, skins, etc., are removed as completely as possible from the edible portion and discarded.

If the composition of the entire mass cannot be made homogeneous throughout, it may be best to select from various portions in making up the sample for analysis, in order to represent as fair an average of the whole as possible.

Finally the sample, if solid or semi-solid, should be divided as finely as possible, by chopping, shredding, pulping, grinding, or pulverizing according to its nature and consistency.

For disintegrating such substances as vegetables and meats for analysis, the common household rotary chopping-machine is admirably adapted. For pulverizing cereals, tea, coffee, whole spices, and the like, the mortar and pestle may be used, or a rotary disk mill or spice-grinder.

**Specific Gravity or Density of Liquids.**—Where formerly it was customary to compare the density of liquids with that of water at 4° C. (its maximum density) it is now more common to refer to water at 15.5° C. or 20° C., making the determination at that temperature. A common form of expressing the temperature of the determination and the temperature of the standard volume of water with which that of the substance is to be compared, is the employment of a fraction, the numerator of which expresses the temperature of the determination and the denominator that of the standard volume of water, as  $\frac{15.5^{\circ}}{4^{\circ}}$ ,  $\frac{15.5^{\circ}}{15.5^{\circ}}$ ,  $\frac{100^{\circ}}{15.5^{\circ}}$ ,  $\frac{4^{\circ}}{4^{\circ}}$  C.\*

When extreme accuracy in the determination of density is required, the pycnometer or Sprengel tube should be employed.

**The Hydrometer.**—This instrument furnishes the most convenient and ready means of determining the density of liquids where extreme nicety is not required. If well made and carefully adjusted, the hydrometer may be depended on to three decimal places, but before relying on its accuracy, it should be tested by comparison with a standard instrument, or with the pycnometer.

The liquid whose density is to be determined is contained in a jar whose inner diameter should be at least  $\frac{3}{8}$ " larger than that of the spindle-

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\* Unless otherwise stated, all specific gravities in this volume are assumed to be expressed on the basis of  $\frac{15.5^{\circ}}{15.5^{\circ}}$



bulb, and the temperature of the liquid should be exactly  $15.5^{\circ}$  when the reading is taken.

For best results for use with liquids of varying densities, the laboratory should be furnished with a set of finely graduated hydrometers, each limited to a restricted part of the scale, together with a universal hydrometer coarsely graduated, covering the entire range, to show by preliminary test which of the special instruments should be used.

A convenient set of seven such hydrometers are graduated as follows: 0.700–0.850, 0.850–1.000, 1.000–1.200, 1.200–1.400, 1.400–1.600, 1.600–1.800, 1.800–2.000, while the universal hydrometer has a scale extending from 0.700 to 2.000. Another less delicate set of three only has one for liquids lighter than water and two for heavier liquids. Some instruments have thermometers in the stem. Others require a separate thermometer.

*The Westphal Balance* (Fig. 14).—This instrument consists of a scale-beam fulcrumed upon a bracket, which in turn is upheld by a supporting pillar. The scale-beam is graduated into ten equal divisions. From a hook on the outer end of the beam hangs a glass plummet provided with a delicate thermometer, the beam being so adjusted that when the dry plummet hangs in the air, the beam is in exact equilibrium, i.e., perfectly horizontal as shown by the indicator on its inner end. If the large rider be placed on the same hook as the plummet and the latter immersed in distilled water of the standard temperature at which the determinations are to be made (say  $15.5^{\circ}$  C.), the scale-beam should again be in equilibrium if the instrument is accurately adjusted. As commonly made, the weight of the plummet including the platinum wire to which it is attached amounts to 15 grams, and the displacement of its volume to 5 grams of distilled water at  $15.5^{\circ}$  C., the normal temperature on which the determinations are based. Thus the unit (or largest) rider should weigh 5 grams, while the others weigh 0.5, 0.05, and 0.005 gram respectively.

If, instead of distilled water, the plummet be immersed in the liquid whose density is to be determined, the position of the riders on the scale-beam, when so placed as to bring the same into equilibrium, and when read in the order of their relative size (beginning at the largest), indicates directly the specific gravity to the fourth decimal place.

If the liquid is lighter than water, the large rider is first placed in the notch where it comes closest to restoring the equilibrium of the beam, but with the plummet still underbalanced. The rider next in size is then applied in a similar manner, and, unless equilibrium is exactly re-



stored, the third and the fourth riders successively. If it happens that two riders should occupy the same position on the beam, the smaller is suspended from the larger.

If the liquid is heavier than water, the method employed is the same except that one of the largest or unit riders is in this case always hung from the hook which supports the plummet, while the others cross the

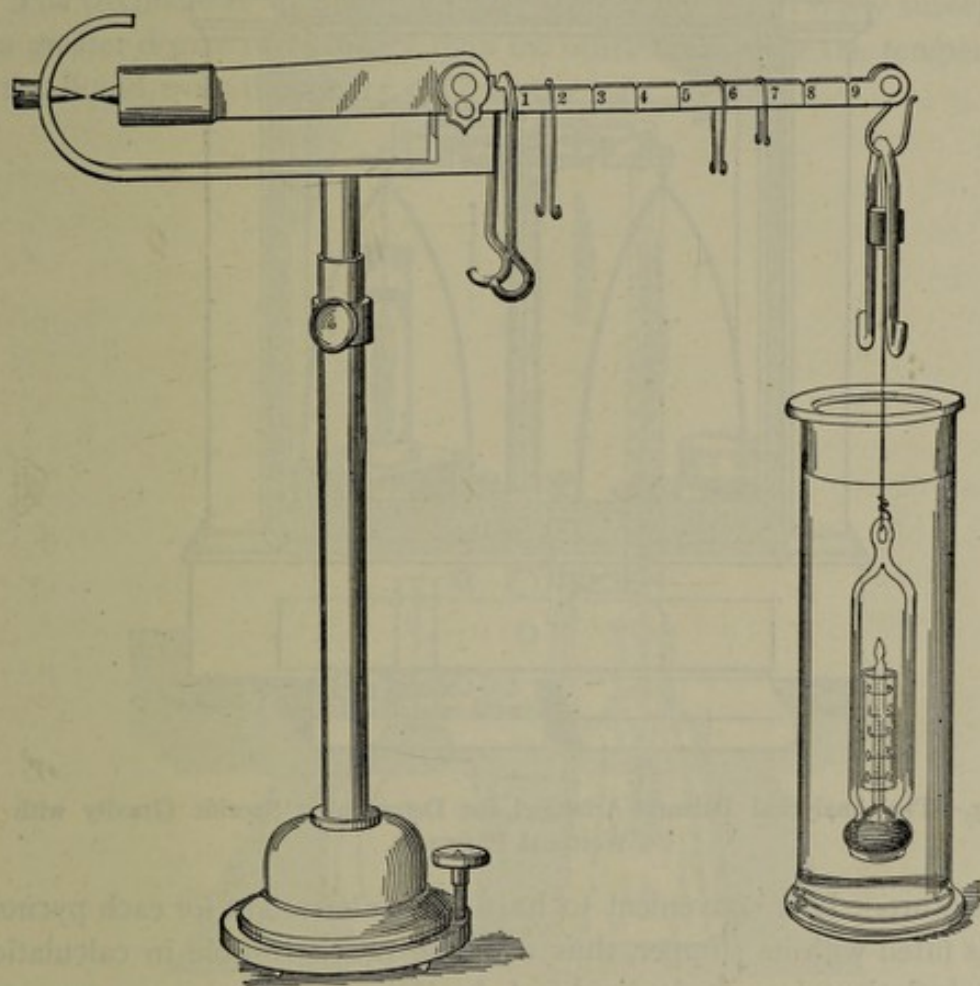


FIG. 14.—The Westphal Balance.

beam at the proper points. If carefully made and adjusted, the Westphal balance is capable of considerable accuracy.

A delicate analytical balance can be used in place of the less carefully adjusted Westphal instrument, by hanging the Westphal plummet from one of the scale-hooks of the same, and employing a fixed support for the glass jar that holds the liquid in which the plummet is to be immersed. The support is so arranged that the scale-pan below it can move freely without coming in contact with it. This arrangement is shown in Fig. 15.

*The Pycnometer, or Specific-gravity Bottle.* — Fig. 16 shows the two



forms of pycnometer commonly made. The plain form has a ground-glass stopper with a capillary passage through it, the other has a fine thermometer connected with the stopper and a capillary side tube provided with a ground hollow cap. Both are made in different sizes to hold respectively 10, 25, 50, and 100 grams of distilled water at the standard

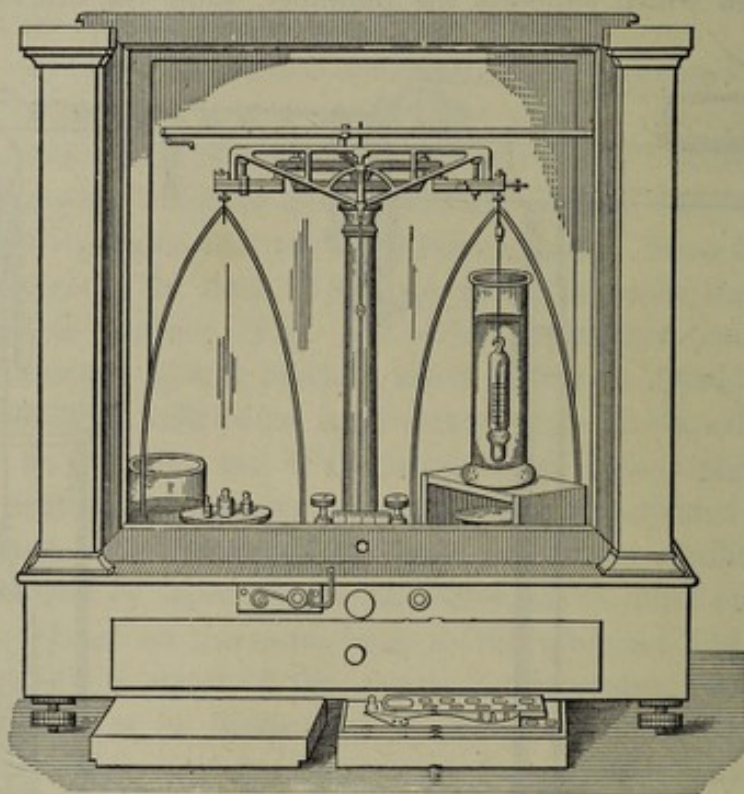


FIG. 15.—The Analytical Balance Arranged for Determining Specific Gravity with the Westphal Plummet.

temperature. It is convenient to have a counterweight for each pycnometer as fitted with its stopper, thus avoiding much trouble in calculation. The calculation of results is simplified also if the pycnometers are accurately constructed to contain *exactly* the weight of distilled water which they purport to contain at the standard temperature, but it is rather difficult to procure such instruments, especially of the form furnished with the thermometer. Most instruments hold *approximately* the amount specified, the exact net weight of distilled water which they hold at standard temperature being found by careful test and kept on record. In determining the density of a liquid, the pycnometer is carefully filled with it at a temperature below the standard, the stopper carefully inserted, and the bottle wiped dry. Care should be taken that the liquid completely fills the bottle and is free from air-bubbles. The net weight of the liquid is then taken



on the balance, when the temperature has reached the standard (say  $15.5^{\circ}$  C.), being careful to wipe off the excess of liquid that exudes from the capillary due to expansion. The net weight of the liquid is divided by that of the same volume of distilled water, previously ascertained under the same conditions at the same temperature, the result being the density of the liquid.

The pycnometer with thermometer attachment is obviously susceptible of a greater degree of accuracy than the other form, since the temperature of the liquid, even though  $15.5^{\circ}$  C. at the start, soon rises.

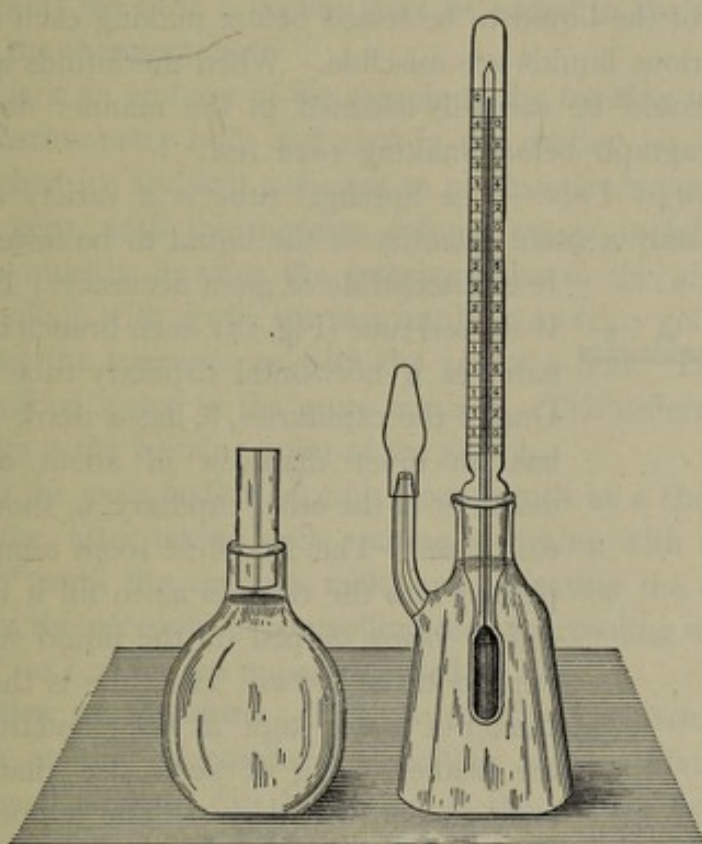


FIG. 16.—Types of Pycnometer.

The writer prefers to use the pycnometer provided with the thermometer, but without the hollow cap that covers the capillary side tube, unless liquids like strong acids are to be operated on, that might otherwise injure the balance. By keeping the liquid to be tested for some time in a refrigerator, it acquires a temperature of from  $10$  to  $12^{\circ}$  C. It is then transferred in the regular manner to the pycnometer and the thermometer-stopper inserted (but not the hollow cap) and the bottle wiped dry. There is ample time to adjust the balance-weights with extreme care while the temperature of the liquid is rising, leisurely wiping off

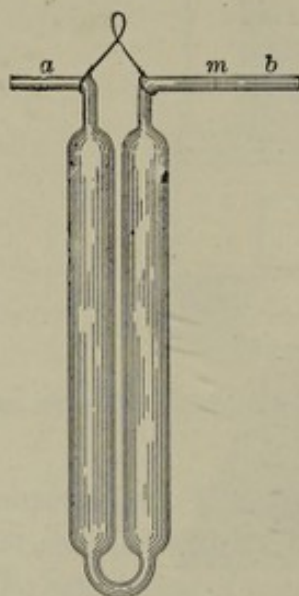


at intervals with a soft towel the excess that exudes from the capillary tube, the final weight of the dry bottle and contents being made at the exact temperature of  $15.5^{\circ}\text{C}$ .

In taking the tare or adjusting the counterweight of a specific-gravity bottle, the latter should be perfectly clean and dry. It had best be rinsed first with water, then with alcohol, and finally with ether, all traces of the latter being removed by a current of dry air, or otherwise, before weighing.

In making successive determinations of density of a number of different liquids with the same pycnometer, it is sufficient to rinse the bottle once with a little of the liquid to be tested before making each determination, when the various liquids are miscible. When the liquids are immiscible, the bottle should be carefully cleaned in the manner described in the previous paragraph before making each test.

*The Sprengel Tube.*—The Sprengel tube is a variety of pycnometer useful when only a small quantity of the liquid to be tested is available.



It is susceptible of great accuracy. It consists of a U-shaped tube (Fig. 17), each branch of which terminates in a horizontal capillary tube bent outward. One of the capillaries, *b*, has a mark *m* thereon and has an inner diameter of about 0.5 mm. The diameter of the other capillary, *a*, should not exceed 0.25 mm. The liquid at room temperature is aspirated into the tube so as to fill it completely, the end *b* being dipped in the liquid while suction is applied at the end *a*. The tube is then placed in a beaker of water kept at the standard temperature, the beaker being of such size that the capillary ends rest on the edge. The temperature of the liquid in the tube may be assumed to be constant

FIG. 17.—Sprengel Tube for Determining Specific Gravity.

when there is no further movement due to contraction in the larger capillary end, *b*. The meniscus of the liquid, when cooled, should not be inside the mark *m*, and is brought exactly to the mark by applying a piece of bibulous paper to the other end, *a*. If a drop or two of liquid has to be added, this may be done by applying to the end *a* a glass rod dipped in the liquid. When exactly adjusted, the whole is wiped dry and quickly weighed, hung from the arm of the analytical balance. To avoid evaporation by contact with the air, the ends of the capillaries are sometimes ground to receive hollow glass caps not shown in the figure.



**Determination of Freezing Point.**—*The Beckmann Apparatus*\* consists of a cooling jar provided with a stirrer, an ordinary thermometer registering temperatures below zero, and a siphon for emptying, an air jacket, a freezing tube with a stirrer, and a Beckmann thermometer graduated to  $0.01^{\circ}\text{C}$ .

The reservoir in the top of the Beckmann thermometer is for a reserve supply of mercury. If the capillary tube contains so much mercury that the top of the column when cooled to the freezing point is not within the scale, by gently tapping a portion may be made to drop into the reservoir; if it contains too little a portion may be added in the same manner after inverting the thermometer.

*Process.*—Place an amount of the sample in the freezing tube sufficient to cover the thermometer bulb and cool in the cooling jar, containing a mixture of crushed ice and salt sufficient to produce a temperature several degrees below zero, until the mercury column ceases to fall and begins to rise. Then quickly transfer the freezing tube to the air jacket and continue the cooling, with gentle stirring, until the mercury column remains constant. Read the temperature with the aid of a lens. Determine the reading for distilled water in the same manner. The difference between the two readings is the freezing point of the sample.

Keister † in the examination of milk recommends as a check removing the freezing tube, after taking each reading, warming with the hands or in water at  $40^{\circ}$  until the contents melt, and repeating the cooling. He also emphasizes the necessity of controlling the supercooling within narrow limits—from  $1^{\circ}$  to  $1.2^{\circ}$  for the apparatus used by him.

**Determination of Moisture.**—This is usually calculated from the loss in weight at the temperature of boiling water. Platinum dishes (Fig. 51) are well adapted for the drying as the residue can be ignited for the determination of ash. If only the moisture is desired, dishes of other metals or glass weighing bottles may be used. Caps for wide-mouthed bottles made of tinned lead are convenient and can be thrown away after using. Viscous substances are best spread over asbestos or sand to hasten the drying.

Some materials must be heated above  $100^{\circ}\text{C}$ ., while certain saccharine products are dried at  $70^{\circ}\text{C}$ . *in vacuo* to avoid decomposition. If alcohol, acetic acid, essential oils, or other volatile substances are present the loss includes these as well as moisture. As the water or steam oven seldom

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\* Zeits. physik. Chem., 2, p. 638.

† Jour. Ind. Eng. Chem., 9, 1917, p. 862.



attains a temperature above  $98^{\circ}$ , the loss sustained in these is, strictly speaking, at the "temperature of boiling water." Figs. 8 and 9 show electric and gas ovens for heating at full  $100^{\circ}$ . Benedict has shown that certain materials can best be dried at room-temperature over sulphuric acid *in vacuo*. Trowbridge\* has shortened this process in the case of meat, by gently agitating the desiccator during the drying.

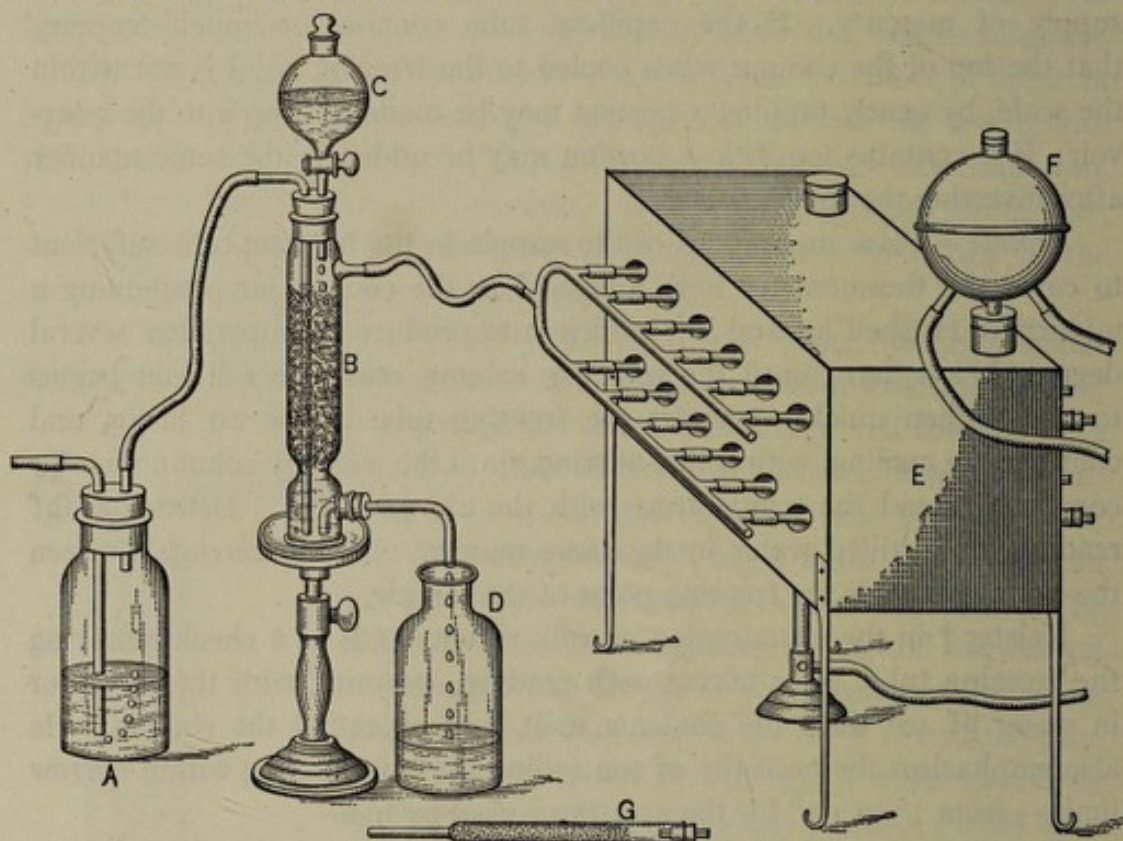


FIG. 18.—Apparatus for Drying in Hydrogen.

*Drying in Hydrogen.*—Fig. 18 shows the apparatus devised by Winton† for drying cereal products, legumes, cattle foods, etc.. The material is weighed out on a watch glass and transferred to the drying tube (G), wisps of cotton, too small to contain an appreciable amount of moisture, being used at both ends to prevent mechanical loss. The hydrogen is purified by passing through sodium hydroxide solution (A) and dried by sulphuric acid in the jar (B). The acid drops over the glass beads into the chamber at the bottom of the jar where the gas bubbles through it before passing out over the beads. A siphon automatically removes the

\* U. S. Dept. of Agric., Bur. of Chem., Bul. 122, p. 219.

† Conn. Agric. Exp. Sta., Rep. 1889, p. 187.



excess of acid. The drying tubes pass through the copper tubes of the water oven and are fitted at the posterior ends with capillary exit tubes of 0.5 mm. bore, thus creating a slight pressure and insuring even distribution of current. When the drying is begun the exit tubes should be within the copper tubes to avoid stoppage of the current by condensed moisture, but later they should be pushed out, as in the cut, and each tested by lighting.

**Determination of Ash.**—The residue from the determination of moisture or else a new portion, is burned at a very faint red heat until white or gray, cooled in a desiccator and weighed. A flat-bottomed platinum dish is most convenient for the purpose. Platinum, however, is attacked by free chlorine, bromine, and iodine, sulphur and phosphorus, sulphates and phosphates with reducing agents, all sulphides, sodium or potassium hydroxide, nitrate and cyanide, metals, and metallic compounds reduced in fusion, such as lead, tin, zinc, bismuth, mercury, arsenic, and antimony. In such cases porcelain must be used.

The degree of heat employed in ashing should be the lowest possible to insure complete oxidation of the carbon, so as to avoid driving off certain volatile salts that are sometimes present and that would be lost if the heat were too high. At a bright red heat potassium and sodium chloride are slowly volatilized, and calcium carbonate is converted into oxide; furthermore alkali phosphates fuse about particles of carbon, protecting them from oxidation. To avoid overheating it is recommended not to allow the flame to impinge directly against the dish, but to carry out the burning on a piece of asbestos paper supported on a triangle. The asbestos also serves to distribute the heat and to protect the dish from the injurious action of the direct flame on long heating. In order to burn off the last traces of carbon, a second piece of asbestos paper may be placed over the top of the dish, or the incineration may be completed in a gas or electric muffle furnace (Figs. 3 and 19). Heating should be continued till the carbon is all oxidized, which is in most cases indicated by a white

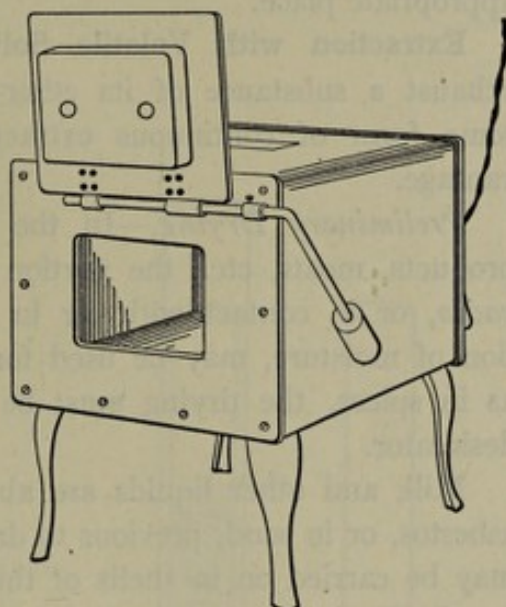


FIG. 19.—Hoskins Electric Furnace.



ash. It is, however, sometimes impossible to obtain a perfectly white ash, but the appearance of the ash usually indicates when all the carbon has been burnt off. It is sometimes necessary to stir the contents of the dish with a stiff platinum wire from time to time during the ignition.

Methods for the detection and determination of the various ash ingredients are described in detail in Chapter X. Such cases as are peculiar to certain foods, like the metallic impurities that occur in canned, bottled, and preserved foods under certain conditions, will be considered in their appropriate place.

**Extraction with Volatile Solvents.**—Whenever it is necessary to exhaust a substance of its ether-soluble or alcohol-soluble ingredients, some form of continuous extraction apparatus is employed with advantage.

*Preliminary Drying.*—In the case of cereal, legume, and oil-seed products, meats, etc., the portion of the material dried in hydrogen, *in vacuo*, or in contact with air in an ordinary oven, for the determination of moisture, may be used for extraction. If volatile oil is present, as in spices, the drying must be performed at room temperature in a desiccator.

Milk and other liquids are absorbed in a roll of bibulous paper, in asbestos, or in sand, previous to drying (Chapter VII). The evaporation may be carried on in shells of thin glass (Hoffmeister Schälchen) which are finally broken previous to extraction, or in tinned lead bottle caps which may be crumpled up and inserted in the extractor.

*The Soxhlet Extractor.*—This apparatus is shown in Fig. 20. The substance to be extracted is subjected to successive treatment with freshly distilled portions of the solvent in the tube *S*. Dry powders are contained in extraction thimbles of filter paper or in filters folded over the end of a flat-bottomed cylinder so as to form a cartridge; liquids, such as milk, previously dried in a paper coil or in a wad of asbestos, are extracted without a filter. The vapor from the solvent, boiling in the flask *F*, passes up through the side tube *a'* into the condenser *C*, where it is liquefied and falls drop by drop on the substance.

When the level of the solvent in the tube *S* reaches the top of the siphon the liquid drains off into the tared flask *F*, carrying with it whatever it dissolves. The operation is automatically repeated, the substance being successively extracted with freshly distilled portions of the solvent, which leaves behind in the flask *F* the material in solution.

The heater employed should be a hot plate heated by steam, or, as



shown in Fig. 20, an electric stove, which may be provided with a fractional rheostat for varying the amount of heat. If neither of these is available the extraction flask may be rested on a piece of asbestos paper

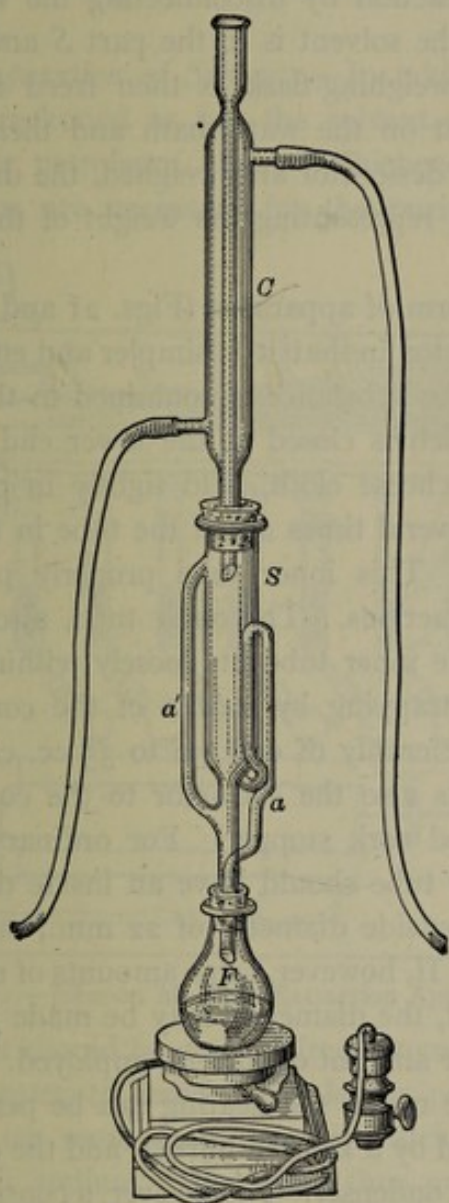


FIG. 20.—The Soxhlet Extractor with Electric Heater.

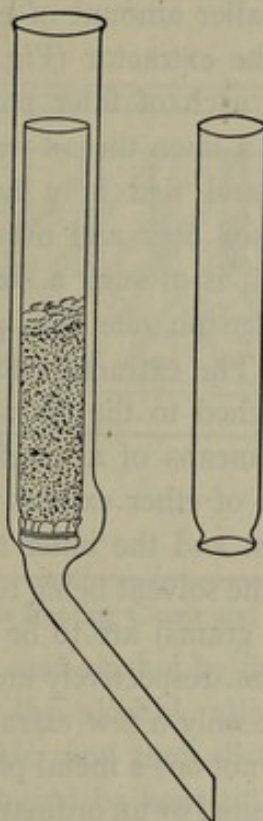


FIG. 21.—Johnson Extraction Tubes.

supported by a lamp stand, the heat being supplied by an ordinary Bunsen burner.

The degree of ebullition is so regulated as to allow the solvent to saturate the sample and siphon over into the flask *F* from six to twelve times an hour, the extraction being continued from two to six hours, or until all the ether-soluble material has been removed. Care should be taken also that



the rate of boiling and the rate of condensation are so regulated that no appreciable loss of reagent occurs during the extraction. A strong smell of ether perceptible at the top of the condenser indicates a loss. The solvent is recovered at the end of the extraction by disconnecting the weighing flask at a time when nearly all of the solvent is in the part *S* and before it is ready to siphon over. The weighing-flask is then freed from all traces of the solvent by drying first on the water-bath and then in the oven, after which it is cooled in the desiccator and weighed, the difference between this and the first weighing representing the weight of the fat or ether extract.

*The Johnson Extractor.*—This form of apparatus (Figs. 21 and 22) has the advantage of the Soxhlet extractor in that it is simpler and employs a much smaller amount of ether. The substance is contained in the inner tube of the extractor (Fig. 21), which is closed at the lower end by one thickness each of filter paper and cheese cloth, held tightly in place by means of a linen thread wrapped several times about the tube in the constriction and tied in a fast knot. This inner tube properly prepared can be used over and over for extractions. The outer tube, also shown in Fig. 21, is of such a size that the inner tube fits loosely within it. A slight bulge on one side prevents trapping by means of the condensed solvent. The extraction flask is preferably of only 30 to 35 cc. capacity. It is attached to the extractor, as is also the extractor to the condenser tube, by means of a carefully bored cork stopper. For ordinary determinations of ether extract the outer tube should have an inside diameter of 26 mm. and the inner tube an outside diameter of 22 mm., only 8 to 10 cc. of the solvent being required. If, however, large amounts of material (25 to 50 grams) are to be extracted, the diameters may be made 32 mm. and 28 mm. respectively and a larger amount of solvent employed.

Where only a few extractions are made, the heating can be performed over (but not on) a metal plate heated by a Bunsen burner, and the condensation effected by an ordinary Liebig condenser. If, however, a considerable number of extractions are carried out, the set apparatus shown in Fig. 22 will be found convenient and also economical of space. It may be attached to the wall or placed at the back of a working desk. The heating, as shown in the cut, is effected by means of two steam pipes, but some form of electric heater answers equally well. The case with glazed door prevents the radiation of heat. At the top is shown the multiple condenser consisting of a copper tank with block tin tubes. Water is introduced at the left and carried off at the right.



The solvent is best poured through the material, thus obviating in large degree the crawling of the extract. The door should be opened several times during the extraction and kept open for a few minutes for the purpose of rinsing down the sides of the tubes by means of the condensed vapors.

*Preparation of Solvents.*—In taking the so-called ether extract, sometimes reckoned as fat, the solvent employed is either ethyl ether or the cheaper petroleum ether. Whichever reagent is employed, certain precautions are necessary for the purity of the reagent. If ethyl ether is

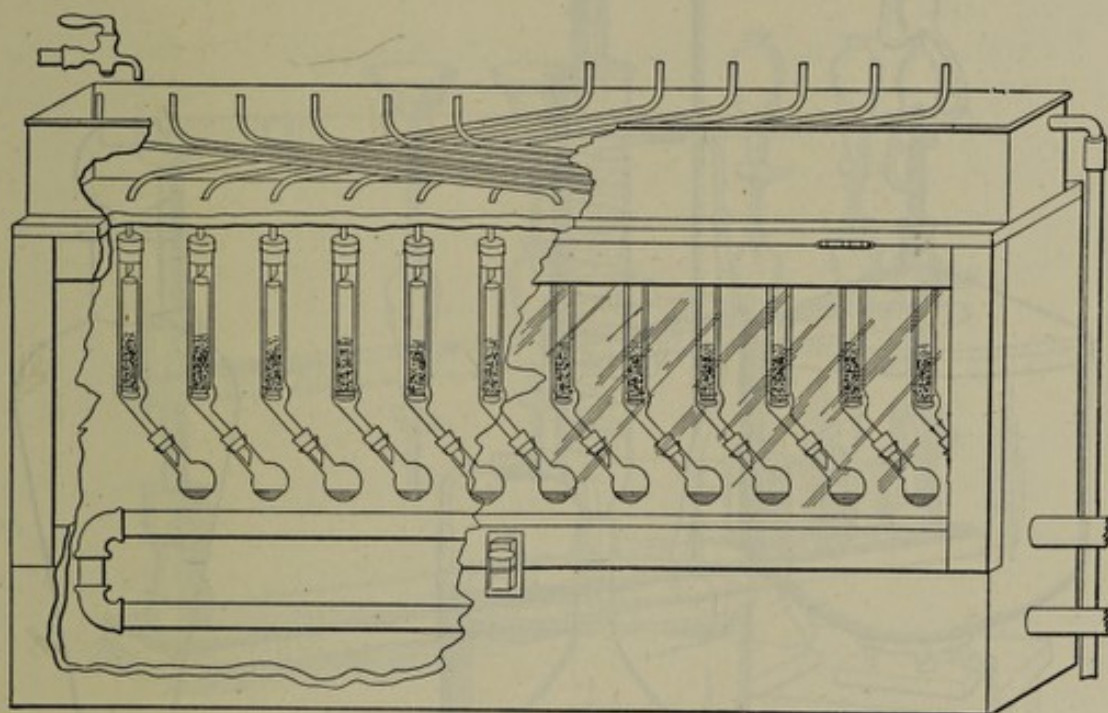


FIG. 22.—Johnson Multiple Extraction Apparatus with Heating Closet and Condenser.

used, it should be entirely freed from moisture and alcohol by first shaking with water to remove the larger portion of the alcohol, allowing it to stand for some time over dry calcium chloride, and then distilling over metallic sodium. The ether thus prepared should be kept till used with sodium in the container, the latter being somewhat loosely corked, to allow escape of the hydrogen formed.

Petroleum ether is variously termed benzine, naphtha, or gasoline. It should be low-boiling, preferably between  $35^{\circ}$  and  $50^{\circ}$ , and it is always best to redistil it before using, in order to be sure it is free from residue. As to the choice of the two reagents for use in fat extraction, it may be said that ethyl ether is the solvent most used, but if a large number of determinations are to be made, the lower cost of petroleum ether is to



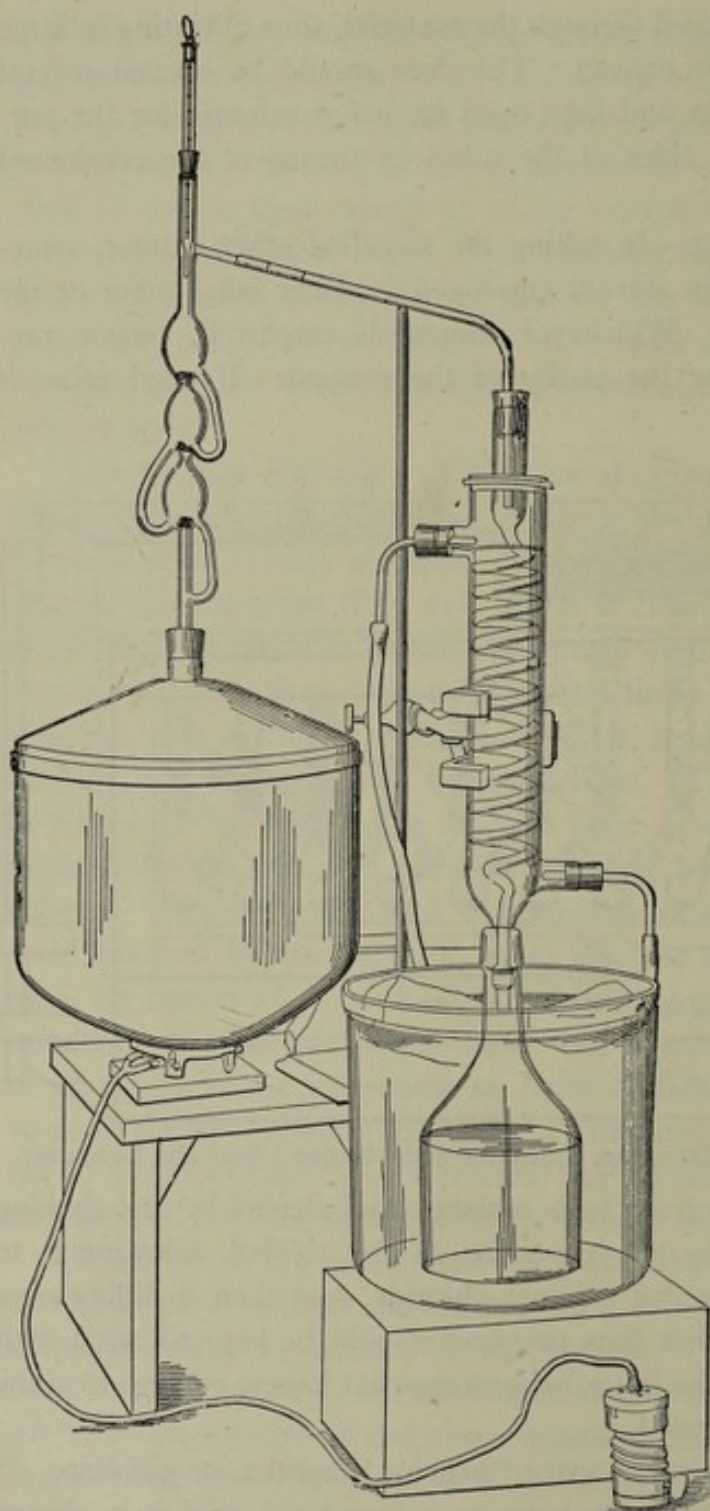


FIG. 23.—Fractionating-still, Arranged for Petroleum Ether.

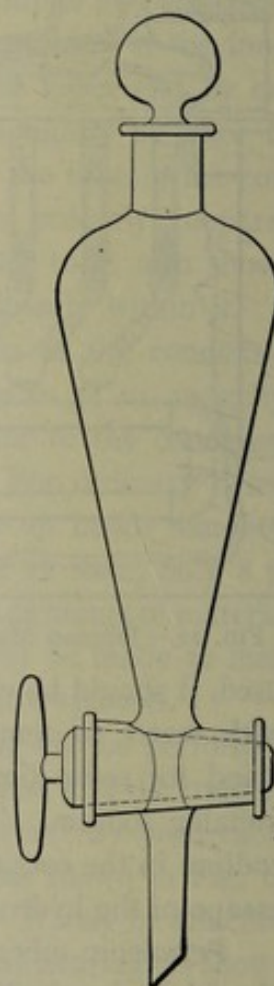


FIG. 24.—A Convenient Form of Separatory Funnel.



be considered. A convenient still for fractionating such substances as petroleum ether is shown in Fig. 23.

**Extraction with Immiscible Solvents.**—It is frequently necessary to dissolve out a substance from a liquid by shaking it with an immiscible solvent, as, for example, in the extraction of certain preservatives from aqueous or acid solutions with ether, petroleum ether, or chloroform. This can be done by shaking in ordinary flasks, but is attended by some difficulty and loss on decantation. A separatory funnel of the type shown in Fig. 24 is almost indispensable for this kind of extraction. The liquid

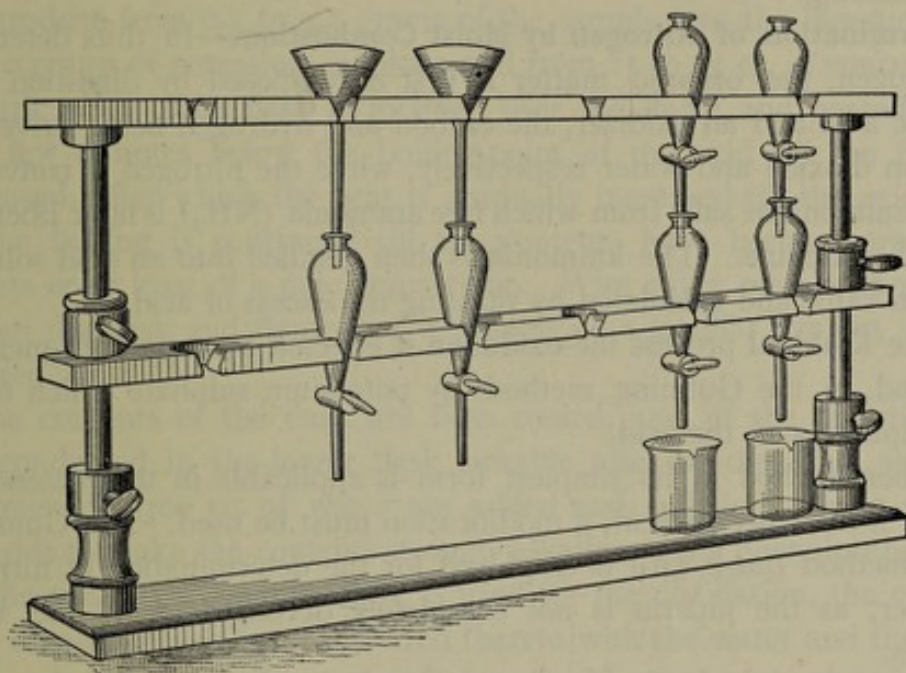


FIG. 25.—Separatory Funnel Support.

and solvent are transferred to the funnel, which is then stoppered and shaken. If the solvent is heavier than water, as in the case of chloroform, it is drawn off from beneath through the outlet-tube of the funnel, or, if the solvent is the lighter, as in the case of ether, the aqueous liquid lying beneath is first drawn off and finally the solvent is poured out through the top. If troublesome emulsions form when shaken, they may frequently be broken up by adding an excess of the solvent and again very gently shaking, or by careful manipulation with a stirring rod, or by centrifuging. If the solvent is ether, and an obstinate emulsion forms, it may frequently be broken by the addition of chloroform. Such a mixture of ether and chloroform sinks to the bottom and may be drawn off as in the case of chloroform alone.



A separatory funnel support, devised by Winton, is shown in Fig. 25. It serves for holding the separatory funnels while drawing from one into another, and also as a support for ordinary funnels. The two shelves are adjustable by means of thumbscrews. The holes in these shelves are somewhat wider than the slots, so that the separatory funnels after being introduced through the latter drop into position and are held firmly while manipulating the stop-cock.

Winton attaches all stop-cocks and stoppers to the funnel by means of small brass chains, thus preventing breaking and interchange of these parts during washing.

**Determination of Nitrogen by Moist Combustion.**—In thus determining nitrogen, the organic matter is first decomposed by digestion with sulphuric acid and an oxidizer, the carbon and hydrogen being driven off as carbon dioxide and water respectively, while the nitrogen is converted into an ammonium salt, from which free ammonia ( $\text{NH}_3$ ) is later liberated by making alkaline. The ammonia is then distilled into an acid solution of known value and calculated by titrating the excess of acid.

In the Kjeldahl process the oxidation is effected by means of a mercury compound, in the Gunning method, by potassium sulphate which forms the bisulphate with the acid.

Neither method in its simplest form is applicable in the presence of nitrates; if these are present, a modification must be used. The Gunning-Arnold method (page 446) is employed for the determination of nitrogen in pepper, as the piperin is not completely decomposed by the usual processes.

*The Gunning Method.—Reagents:*

Standard alkali solution,  $\text{N}/10$   $\text{NaOH}$  or  $\text{NH}_4\text{OH}$ .\*

Pulverized potassium sulphate.

Sulphuric acid, concentrated, free from nitrogen.

Sodium hydroxide, saturated solution.

Standard acid solution,  $\text{N}/10$   $\text{H}_2\text{SO}_4$  or  $\text{HCl}$ .\*

An indicator, cochineal solution (page 28).

Granulated zinc, passing a 1-mm. mesh.

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\* Winton employs standard acid of such a strength that 1 cc. is equivalent to 1% of nitrogen, working on a gram of material, and titrates back with standard alkali two and one-half times weaker than the acid. In order to insure accurate readings, burettes of narrow bore (1 cc. = 2.6 cm.) are employed. The alkali burette is so graduated that a reading of 1 corresponds to 2.5 cc., thus allowing for the greater dilution. The advantage of this system is that the per cent of nitrogen is obtained by simply subtracting the alkali reading from the number of cc. of acid employed.



The digestion and distillation are preferably carried out in the same flask, which should be pear-shaped with flat or round bottom and made of moderately thick Jena glass. A convenient size has the following dimensions: length 29 cm., maximum diameter 10 cm., tapering gradually to a long neck, which near the end is 28 mm. in diameter with a flaring edge. Its capacity is about 550 cc.

If desired, the digestion may be conducted in a smaller hard-glass flask of about 250 cc. capacity and of the same shape as the above, and the distillation in an ordinary round-bottomed flask of 500 cc. capacity.

Introduce from 0.5 to 3.5 grams of the sample into the digestion-flask with 10 grams of potassium sulphate and from 15 to 25 cc. of concentrated sulphuric acid. The flask is inclined over the flame and heated gently for a few minutes below the boiling-point of the acid till the frothing has ceased, after which the heat is gradually increased till the acid boils, and the boiling is continued till the contents have become practically colorless or at least of a pale straw color. Wire gauze may be interposed between the flask and flame, but a triangle or a similiar support is to be preferred.

The contents of the flask are then cooled, and, if the digestion has been conducted in the larger flask suitable also for distilling, as above recommended, 300 cc. of water are added and sufficient strong sodium hydroxide to make the contents strongly alkaline, using phenolphthalein as an indicator. If a separate flask is used for the distillation, the contents of the digestion-flask are transferred thereto with the water and the alkali added. A few pieces of granulated zinc should also be introduced, which by the evolution of gas prevents bumping and the sucking back of the distillate. The flask is then without delay connected with the condenser, the bottom of which is provided with an adapter, dipping below the surface of the standard hydrochloric or sulphuric acid, a measured quantity of which is contained in the receiving-flask. The distillation is then continued till all the ammonia has passed over into the acid, this part of the operation requiring from forty-five minutes to an hour and a half. As a rule the first 250 cc. of the distillate will contain all the ammonia.

The excess of acid in the receiving-flask is then titrated with standard alkali, and the amount of nitrogen absorbed as ammonia is calculated. The reagents, unless known to be absolutely pure and free from nitrates and



ammonium salts, should be tested by conducting a blank experiment with sugar, by means of which any nitrates present are reduced. Any nitrogen due to impurities should be corrected for.

In purchasing sulphuric acid for nitrogen determination it is important to specify that it be "nitrogen-free" as the so-called chemically pure acid often contains a considerable amount of nitrogen.

*Modification of Gunning Method to include Nitrogen of Nitrates.*—In addition to the reagents used in the simpler Gunning method, sodium thiosulphate and salicylic acid are required.

A mixture of salicylic and sulphuric acids is made in the proportion of 30 cc. of concentrated sulphuric to 1 gram of salicylic. From 30 to 35 cc. of

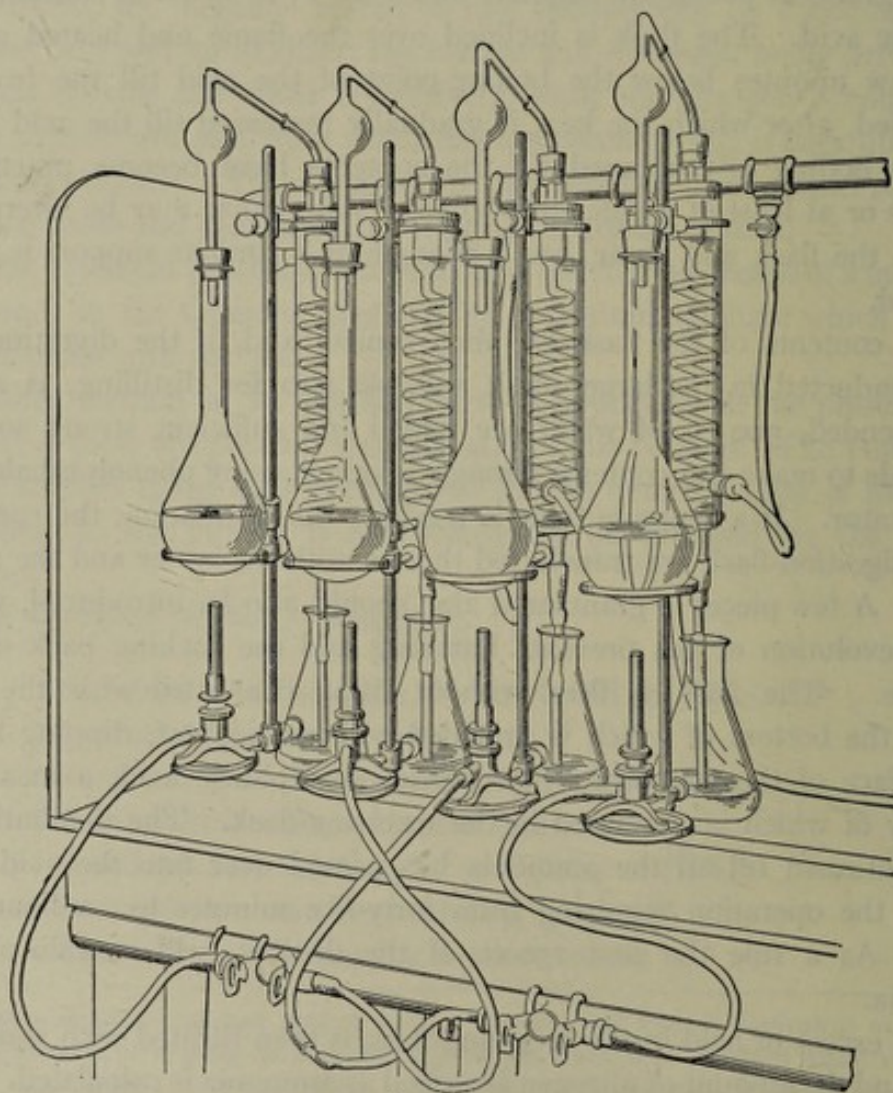


FIG. 26.—Bank of Stills for Nitrogen Determination by Gunning Process.

the mixture are added to the 0.5 to 3.5 grams of the substance in the digestion-flask, the flask is well shaken and allowed to stand a few minutes,



occasionally shaking. Then 5 grams of sodium thiosulphate are added, and 10 grams of potassium sulphate, after which the heat is applied, at first very gently and afterwards increasing slowly till the frothing has ceased. The heating is then continued till the contents have been boiled practically colorless. From this point on, proceed as in the Gunning method.

*The Kjeldahl Method.*—One gram of the air dry substance, or a proportionately larger amount of a moist or liquid substance, and 0.7 gram of mercuric oxide (or an equivalent amount of metallic mercury) are placed

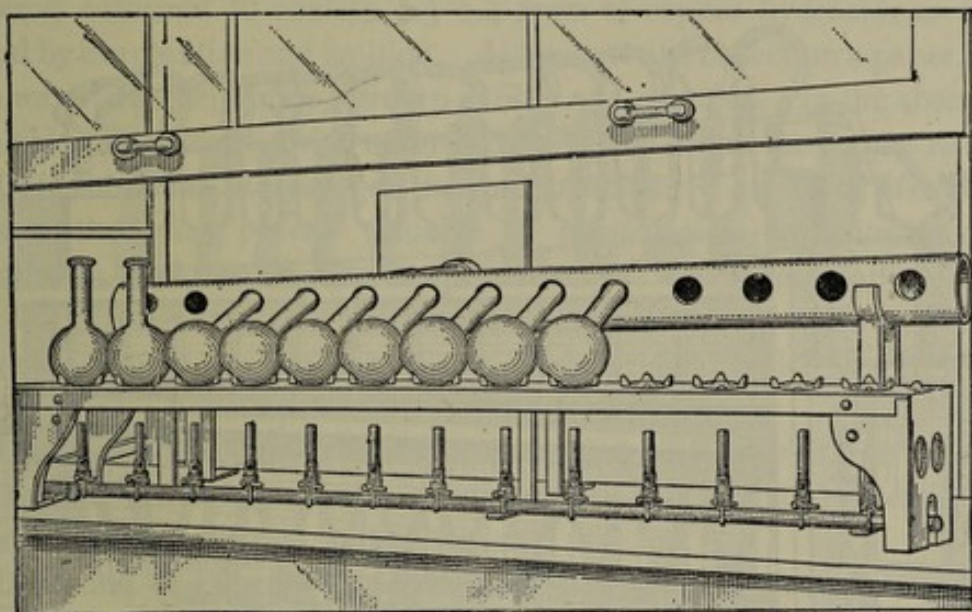


FIG. 27a.—Johnson Digestion Stand for Nitrogen Determination with Lead Pipe for Carrying off Fumes.

in a 550-cc. Jena flask and 20 cc. of sulphuric acid added. The flask is placed in an inclined position over a Bunsen burner, and the mixture heated below boiling for 5 to 15 minutes or until the frothing ceases, after which the heat is raised until the mixture boils briskly. The boiling is continued until the liquid has become nearly colorless and for a half hour in addition. The lamp is then turned out, the flask placed in an upright position, and potassium permanganate slowly added with shaking until the solution takes on a permanent green or purple color.

After cooling, 250 cc. of water are added, then 25 cc. of potassium sulphide solution (40 grams of the commercial salt in 1 liter of water), sufficient saturated sodium hydroxide solution to render the solution alkaline, and finally a few grains of granulated zinc, shaking the flask after each addition. Without delay connect with the distillation apparatus, and proceed as in the Gunning method.



**Apparatus for Nitrogen Determination.**—A bank of stills used by the author in nitrogen determination and in other processes is shown in Fig. 26.

The digestion apparatus shown in Fig. 27a is that devised by Johnson, Winton, and Boltwood. The stand is of cast iron, with holes provided with three projections that support the flask. The lead pipe with holes for receiving the ends of the flasks serves to carry off the acid fumes. Sy has devised apparatus for sucking the fumes from the flask into water by means of a filter pump, thus dispensing with a hood.

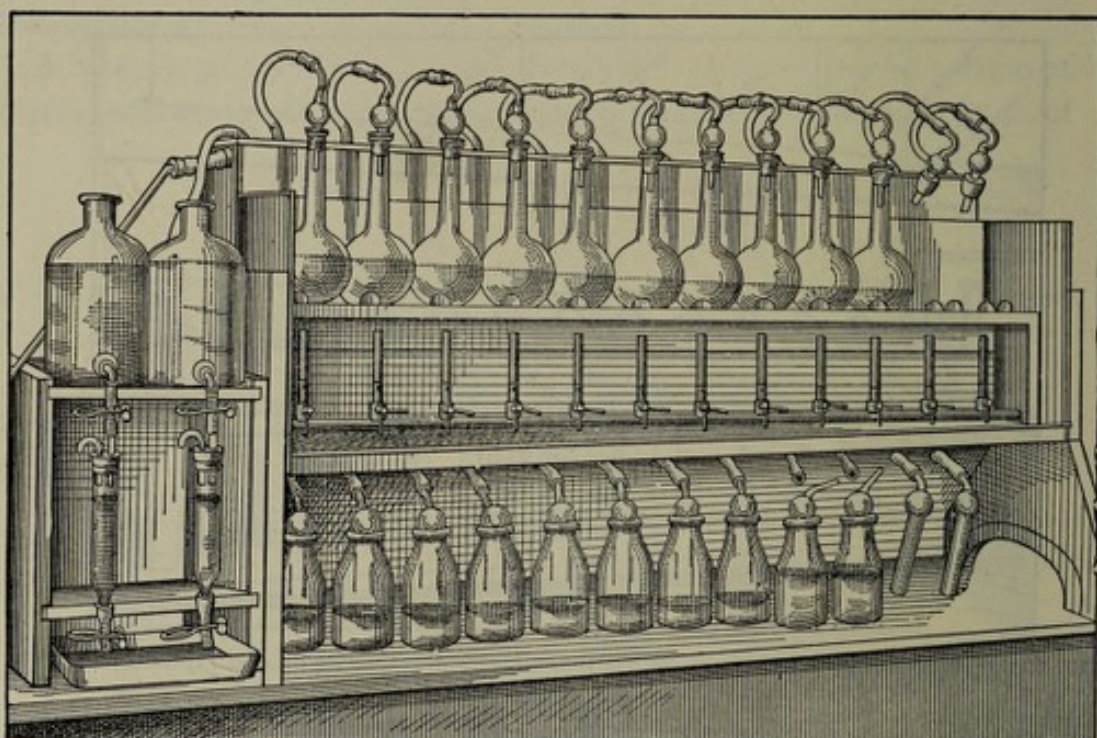


FIG. 27b.—Johnson Distilling Apparatus for Nitrogen Determination.

The Johnson distilling apparatus, with accessories by Winton, is shown in Fig. 27b. The distillation tubes, except for the glass traps and bulb receiver tubes, are of block tin, and are cooled in a copper tank filled with water. The receivers for the distillate are ordinary pint milk bottles.

At the left are two bottles with suspended tubes for measuring the potassium sulphide and sodium hydroxide solutions.

**Determination of Ammonia.**—A weighed quantity of the finely divided sample, treated with ammonia-free water and made alkaline with magnesium oxide free from carbonate, is distilled into a measured quantity of standard acid (tenth-normal hydrochloric or sulphuric acid) and the amount of ammonia determined by titration.



**Determination of Protein Nitrogen.**—*Stutzer Method*.\*—Boil 0.5–2.0 grams of the sample, ground to pass a 1-mm. mesh, with 100 cc. of 1% acetic acid in 95% alcohol, cool, filter, and wash by decantation with warm alcohol. Heat the insoluble matter in the beaker with 100 cc. of water for 10 minutes on a boiling water-bath with stirring, cool, and add copper hydroxide suspension (2% copper sulphate solution containing 0.05% of glycerol, precipitated with an excess of sodium hydroxide, washed by decantation with water containing 0.5% of glycerol, and finally suspended in 10% glycerol) sufficient to contain 0.3–0.4 gram of copper hydroxide as determined by evaporation and ignition. Allow to settle, collect on a paper, wash with water, and determine nitrogen in filter and contents. In the absence of alkaloids heat directly with water and precipitate with the copper reagent.

**Determination of Nitrogen in Amino Acids.**—*Van Slyke Method*.†—This method has proved valuable in physiological investigations and is useful in food examination in special cases. The manipulation is quite simple, but the apparatus is somewhat expensive. For further details reference should be made to Van Slyke's original articles or Mathews' *Physiological Chemistry*.

**Determination of the Various Carbohydrates.**—Under title of "Cereals" in Chapter X are given in detail methods for separation and determination of sugar, starch, dextrin, crude fiber, etc.

**Detection of Poisons.**—Metallic impurities present in foods incidental to their preparation, or as adulterants, are considered under title of foods liable to such adulteration. The detection of highly toxic substances, such as arsenic, corrosive sublimate, and alkaloids, added with criminal intent, comes within the province of the medico-legal chemist or toxicologist and is beyond the scope of this work. The methods involved are fully described in the treatises of Autenrieth ‡ and Blyth,§ only those for arsenic, which occurs also as an accidental impurity, being here considered.

**Detection and Determination of Arsenic.**—*Methods of Solution*.—Syrups, baking powders and other materials soluble in water or acid do not need preliminary treatment. Beer is treated as described in Chapter XV. Other methods of solution are as follows:

1. *Johnson-Chittenden-Gautier Method*.||—This method is suitable for meat, vegetables, and the like, the proportion of acids used being

\* Jour. Landw., 29, 1881, p. 473.

† D. D. Van Slyke, Jour. Biol. Chem., 12, 1912, p. 275; 16, 1913, p. 121; 23, 1915, p. 408.

‡ Detection of Poisons and Strong Drugs, trans. by Warren, Phila., 1905.

§ Poisons, their Effects and Detection, London, 1906.

|| Amer. Chem. Jour., 2, 1880–81, p. 250.



varied to suit conditions. Heat at  $150^{\circ}$ – $160^{\circ}$  C., in a porcelain dish, 100 grams of the finely divided material with 23 cc. of pure concentrated nitric acid, stirring occasionally. When the mixture assumes a deep orange color, remove from the heat, add 3 cc. of pure concentrated sulphuric acid, and stir while nitrous fumes are given off. Heat to  $180^{\circ}$  and add while hot, drop by drop, with stirring, 8 cc. of nitric acid, then heat at  $200^{\circ}$  till sulphuric fumes come off and a dry charred mass remains. Pulverize the mass, exhaust with hot water, filter, evaporate to small volume, take up in cold 20% sulphuric acid and treat by the modified Marsh or Gutzeit method.

2. *Sanger Method*.\*—Digest at room-temperature for some hours 5 to 20 grams of the material in a casserole with about an equal bulk of

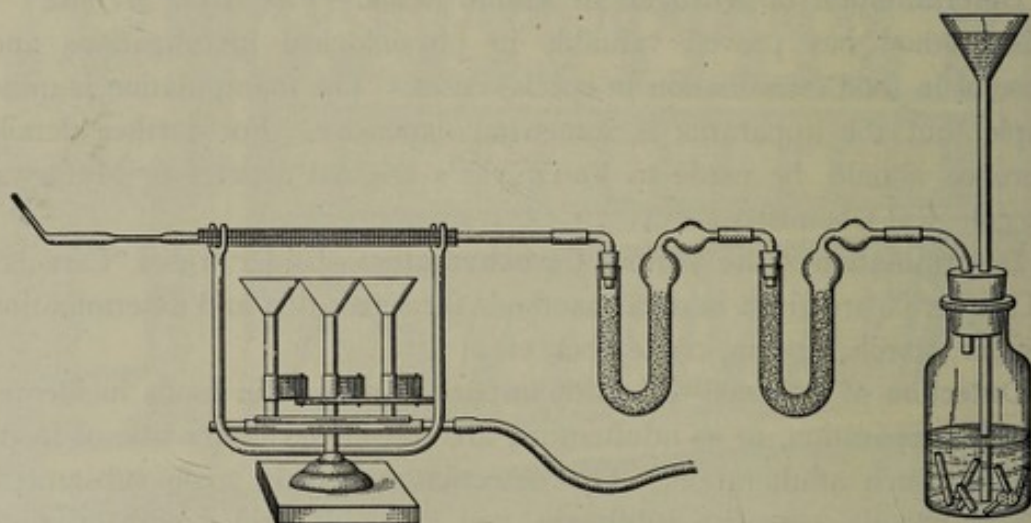


FIG. 28.—Marsh Apparatus for Arsenic.

concentrated nitric acid, add 20 cc. of concentrated sulphuric acid and digest further at a gentle heat until the mixture begins to char. Add about 2 cc. of nitric acid and heat until sulphuric fumes appear, repeating the addition of acid and heating until oxidation appears to be practically complete. Remove all nitric acid by dilution and evaporation to the fuming stage, then dilute with 4 volumes of water. At this point about twice the bulk of saturated sulphurous acid solution may be added and the evaporation repeated, thus reducing to the arsenious condition, but this is not usually necessary.

*Methods of Determination.*—1. *Marsh Test.*—The apparatus (Fig. 28) consists of a generating flask with funnel tube, a U-tube containing cotton

\* Proc. Am. Acad. Arts, Sci., 26, 1891, p. 24.



moistened with 10% lead acetate solution (to remove hydrogen sulphide), a calcium chloride drying tube, and a hard glass tube of 6 mm. bore, drawn down near the end to a uniform constriction about 4 cm. long and 1 mm. inside diameter and also at the very end to a narrow exit tube. The tube is supported over a three-burner furnace the part in contact with the flame being wrapped with wire gauze.

Introduce into the generating flask from 20 to 30 grams of arsenic-free stick zinc and a perforated platinum disk to form an electric couple. Stopper and add through the funnel tube 20% sulphuric acid sufficient to start the reaction and drive out all air. When danger of explosion is over heat the tube to bright redness. After running the current long enough to prove the absence of arsenic in the reagents add slowly from the funnel tube the solution of the material in 20% sulphuric acid or the solution obtained by one of the foregoing methods containing about 20% of that acid, keeping a steady evolution of gas. When the flow slackens add 30% sulphuric acid and later 40% acid until all arsenic has been expelled, which usually requires 2 to 3 hours. If no arsenic mirror forms in the constriction of the tube in one hour, further test may be abandoned.

If more than 0.1 mg. of arsenic appears to be present cut off the constriction from the tube and weigh it on an assay balance; then dissolve the arsenic in a solution of sodium hypochlorite. (antimony being insoluble), wash with water and then with alcohol, dry, cool, and weigh. The loss is arsenic.

If the amount of arsenic is very small Sanger compares the mirror with a series of standard mirrors prepared in the same apparatus using quantities of a standard solution containing from 0.005 to 0.05 mg. of

$\text{As}_2\text{O}_3$ . To prepare the standard solution 1 gram of pure  $\text{As}_2\text{O}_3$  is dissolved in arsenic-free sodium hydroxide, acidified with sulphuric acid, made up to one liter and 10 cc. of this stock solution further diluted to 1 liter; 1 cc. = 0.01 mg.  $\text{As}_2\text{O}_3$ .

2. *Sanger-Black-Gutzeit Method*.\*—The apparatus (Fig. 29), devised by Bishop, consists of a 30 cc. salt-mouth bottle provided with three upright



FIG. 29.—Bishop Apparatus for Arsenic.

\* Jour. Soc. Chem. Ind., 26, 1907, p. 1115.



tubes one above the other. The lower tube is 7 cm. long, 1 cm. in bore, and contains strips of filter-paper previously soaked in 5% lead acetate solution and dried. The middle tube is of the same size as the lower but shorter. It is loosely filled with cotton moistened with 1% lead acetate solution. The upper tube has a uniform bore of 2.5 mm. and is bent twice so that the upper end is vertical. In this tube is placed a strip of cold-pressed drawing paper 2 mm. wide which has been soaked in 5% alcoholic mercuric chloride (or bromide) and dried.

Place in the evolution bottle 10 grams of stick zinc, a few crystals of stannous chloride, a perforated platinum disk and from 2 to 5 grams of the material or else the extract of the charred or digested material prepared as described in the foregoing sections, containing about 20% of sulphuric acid. Add enough 20% (1:4) sulphuric acid to nearly fill the bottle, attach the three tubes and allow to react for 45 minutes. Compare the color on the sensitized strip with that of standard strips obtained with from 0.005 to 0.05 mg. of  $\text{As}_2\text{O}_3$  in the same apparatus, using measured quantities of the standard solution described under the Marsh test.

**Colorimetric Analysis.**—Certain analytical processes depend on the formation of a compound of the substance to be determined having a definite color, and the calculation of the quantity present from the intensity of the color of the solution, compared with that of a solution containing a known amount. The comparisons may be made in a special form of cylinder or in a colorimeter. The latter has the advantage that a single solution of known strength serves within reasonable limits for matching any shade in the unknown solution, and for any number of determinations, the desired depth of the color being secured by varying the length of the column.

**Schreiner's Colorimeter.\***—This apparatus, shown in Fig. 30, consists of two graduated tubes (*B*), containing the standard and unknown colorimetric solutions, the height of the column of liquid in both tubes being changed by two immersion tubes (*A*), which remain stationary while the graduated tubes are raised or lowered in the clamps (*C*). The mirror *D* reflects the light through the tubes, and the mirror *E* reflects it again to the eye of the operator at *F*.

In making the comparisons, the tube containing the solution of either known or unknown strength is set at a definite point, and the other tube is raised or lowered until the colors match. If *R* is the reading of the standard solution of the strength *S*, and *r* the reading of the colorimetric solution of unknown strength *s*, then  $s = \frac{R}{r}S$ .

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\* Jour. Am. Chem. Soc. 27, 1905, p. 1192.



If desired, standard slides of colored glass, such as accompany the Lovibond tintometer, may be used at *G* for matching the solution of unknown strength, the value of these slides being determined by comparison with a standard solution.

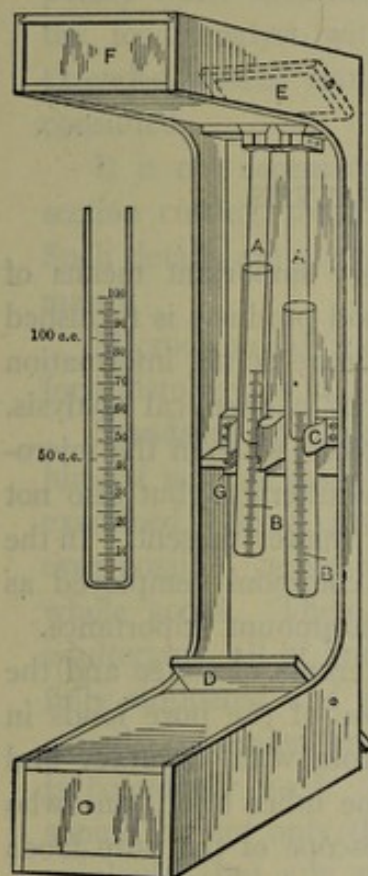


FIG. 30.—Schreiner's Colorimeter with a Tube showing Graduation.

The Lovibond Tintometer may be used for colorimetric chemical analysis, but is not so well suited for this purpose as the Schreiner colorimeter. It is especially designed for determining the color value of liquid and solid technical products, such as beer, wine, oil, flour, paper, etc.

The instrument itself is of simple construction, consisting of an elongated box with an eyepiece at one end and two rectangular openings at the other, one for the solution or substance to be examined, the other for the standard glass slides used for matching the color. Light is reflected through the openings by means of a square piece of opal glass mounted on a jointed standard. Liquids are examined in rectangular cells with glass sides by transmitted light, while powders are pressed into a form and examined by reflected light.

The standard slides used in general work are red, yellow, and blue in even graduation from .006 to 20 tint units which can be combined so as to produce any desired tint or shade of any color. The results are expressed in terms of standard dominant colors (red, yellow, and blue), subordinate colors (orange, green, and violet) obtained by combining equal values of two dominant colors, and neutral tint (black) obtained by combining equal values of the three dominant colors. Thus

$$\begin{aligned} 0.6R + 5.6Y &= 0.6O + 5.0Y \\ 0.08R + 1.5Y + 0.2B &= 0.08N + 0.12G + 1.3Y \\ 1.2R + 1.0B &= 1.0V + 0.2R \end{aligned}$$

in which *R*=red, *Y*=yellow, *B*=blue, *O*=orange, *G*=green, *V*=violet, *N*=neutral tint or black.

Special slides may be obtained for the examination of any desired product. For example, slides of brown shades are furnished for beer, of yellow shades for oils, and so on.



## CHAPTER V.

### THE MICROSCOPE IN FOOD ANALYSIS.

**Microscopical vs. Chemical Analysis.**—A very important means of identification of adulterants in many classes of food products is furnished by the microscope, which in many cases affords more actual information as to the purity of food than can be obtained by a chemical analysis. This is especially true of coffee, cocoa, and the spices, wherein the microscope serves to reveal not only the nature of the adulterants, but also not infrequently the approximate amount of foreign matter present. In the case of the cereal and leguminous products so commonly employed as adulterants, a microscopical examination is of paramount importance.

The chemical constants of many of the adulterants of coffee and the spices do not always differ sufficiently from those of the pure foods in which they appear to be distinguished therefrom with accuracy and confidence by a chemical analysis alone. On the other hand, one who is familiar with the appearance under the microscope of the pure foods and of the starches and various ground substances used as adulterants, can, with certainty, identify very minute quantities of these materials, when present, with the same ease that one can recognize megascopically the most familiar objects about him.

A chemical test may, for example, indicate the presence of starch, but it cannot reveal the particular kind of starch. The microscope will at once show whether the starch present is wheat or corn or potato or arrowroot, since these starches differ almost as much in microscopical appearance as do the physical characteristics of the grains or tubers from which they are obtained. Again, by a chemical analysis an abnormal amount of crude fiber may show the presence of a woody adulterant, but only the microscope will enable one to decide whether the impurity consists of sawdust, chaff, or ground nut shells. Not only in such instances as these is the microscopical examination of greater importance than a chemical analysis, but it is a much quicker guide.

**The Technique of Food Microscopy.**—The recognition of adulterants by the microscope requires some experience but no more than may be acquired by a chemist who will give the subject serious attention. In



the examination of flour, commercial starch, cocoa, coffee, tea, and the spices for adulteration, a careful study of the powdered substance in temporary water mounting will in most cases prove sufficient to familiarize the food analyst with their characteristics under the microscope. In extended studies standard works on the microscopy of foods should be consulted.

It is not necessary for him to familiarize himself with the details of section cutting, dissecting, or permanent mounting unless he so desires. Such details are given by Behrens,\* Chamberlain,† Gage,‡ and Zimmerman.§

Microchemical methods of analysis, a subject quite distinct from food histology, is fully treated by Chamot.||

**Standards of Comparison.**—For standards the analyst should provide himself with as complete a set as possible of the various materials to be examined, taking care that their absolute purity is established. Wherever possible, he should grind the sample himself from carefully selected whole goods. These, together with samples of the starches and other adulterants, all of known purity, should be contained in small vials carefully stoppered and plainly labeled, arranged alphabetically or in some equally convenient manner in the desk or table on which the microscope is commonly used. The adulterants included in this set of standards should be not only those which experience has shown most liable to be employed, but any which, by reason of their character, might in the analyst's opinion be used under certain conditions.

#### APPARATUS.

**The Microscope-stand.**—An expensive or complicated stand is unnecessary. The prime requisites for good work in a microscope-stand are firmness or rigidity, and accuracy in centering. An inexpensive stand possessing these features can be used for the best work, providing the optical parts are satisfactory. It is well, if economy must be practiced, to purchase a simple stand provided with the society screw, and let the larger portion of the allowance go for a high grade of lenses, since many of the attachments inherent in a high-priced stand, though often of convenience, may well be dispensed with.

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\* Guide to the Microscope in Botany.

† Methods in Plant Histology.

‡ The Microscope and Microscopical Methods.

§ Botanical Microtechnique.

|| Elementary Chemical Microscopy, New York, 1915.



A stand of the so-called continental type (having the horseshoe base) is preferable. A square stage is rather more convenient than the circular form, and the jointed pillar possesses advantages over the rigid variety in ease of manipulation that are certainly worth considering.

The smooth working of both the coarse and fine adjustments should not be lost sight of. If the microscope is to be used exclusively for food work, a substage condenser is unnecessary, hence the construction of the

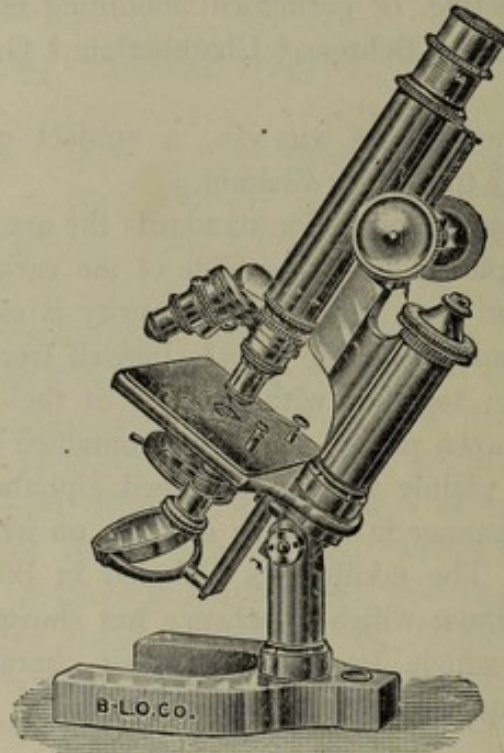


FIG. 31.—Continental Type of Microscope.

substage may be very simple, unless bacteriological work is to be done as well.

A nose-piece, while not indispensable, is a great convenience for the quick transfer of objectives. A double nose-piece carrying two objectives is ample for routine food work.

**The Optical Parts** are by far the most important, and should be of superior quality, though not necessarily of the most expensive makers. The food analyst should have at least two objectives, one for high- and one for low-power work, and preferably two oculars.

For the routine examination of powdered food substances the writer prefers a  $\frac{1}{4}$ -inch objective, used with a medium ocular, the combination giving a magnification of from 240 to 330 diameters, according to the ocular employed. For a low-power objective the  $\frac{3}{4}$ -inch is a conven-



ient size. It is useful as a finder preliminary to examination with the higher power, and, in connection with a low-power eyepiece, is well adapted for the examination of butter and lard, and for use with the polariscope.

An eyepiece micrometer mounted in an one inch ocular is indispensable for measuring starch grains and other elements. It is calibrated by means of a stage micrometer.

**The Micro-polariscope.**—This accessory is useful in the identification of starches and other ingredients, and for ascertaining whether or not fats have been crystallized. The polarizer is held below the stage, while the analyzer is applied above the objective, either in the tube or above the ocular.

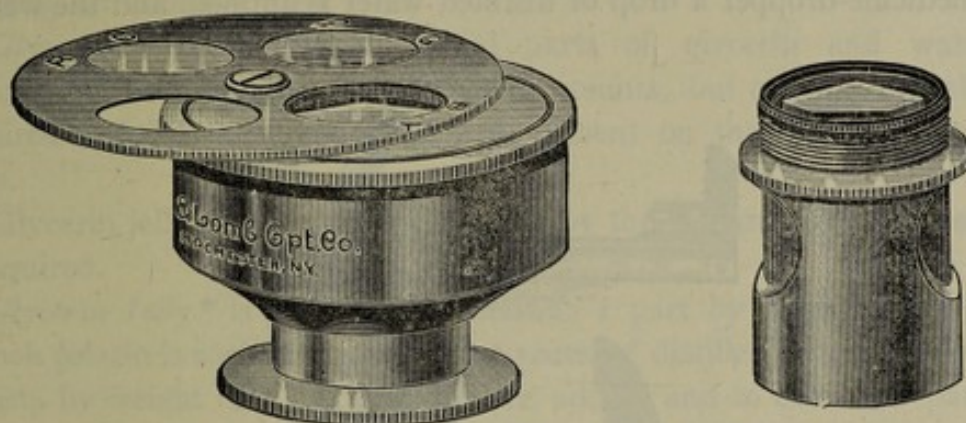


FIG. 32.—Polarizer and Analyzer for the Microscope.

A common form of construction is one in which the substage is adapted to carry interchangeably the diaphragm tube and the polarizer. If the polariscope is much used, it becomes desirable to provide means for quickly changing the polarizer and diaphragm tube below the stage, and for moving the analyzer in and out of place above the objective. Winton\* has devised a microscope-stand with this in view, especially adapted to the needs of the food analyst.

If the polariscope is to be used often, it is convenient to have within easy access two stands, one with the polariscope mounted in place in connection with low-power glasses ready for use, and the other stand provided with the ordinary high- and low-power objectives only.

**Microscope Accessories** include of necessity a large number of slides and cover glasses. The latter should be No. 2 thickness,  $\frac{3}{4}$  inch, either round or square.

One or more dissecting-needles in holders and a small hand magnifying-glass should also be provided.

\* Journal App. Microscopy, 2, p. 550.



Other useful accessories are a mechanical stage, a pair of fine tweezers, knives, scissors, and, if sections are to be cut, a plano-concave razor.

#### MICROTECHNIQUE.

**Preparation of Vegetable Food Products for Microscopical Examination.**—The ground spices and cocoas of commerce are usually of the requisite fineness for direct examination without further treatment. Coffee, chocolate, starches, and similar products should be ground in a mortar fine enough to pass through a sieve with from 60 to 80 meshes to the inch.

A small portion of the powdered sample is taken up on the tip of a clean, dry knife-blade, and placed on the microscope-slide. By means of a medicine-dropper a drop of distilled water is applied, and the wetted

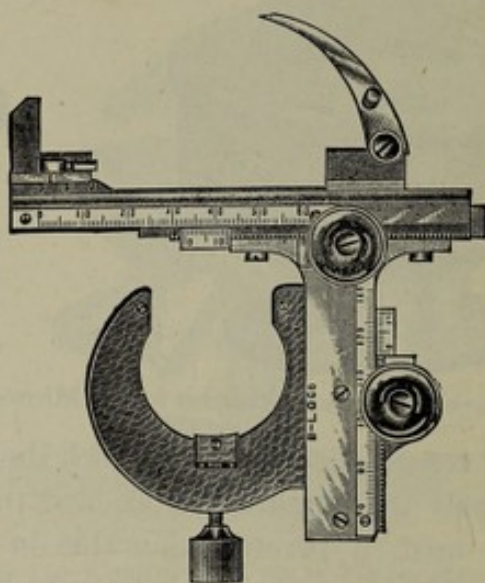


FIG. 33.—Mechanical Stage for Microscope.

powder is then rubbed out under the cover-glass between the thumb and finger to the proper fineness.

The water-mounted slide thus prepared, while useful only for temporary purposes, has proved to be best adapted to the analyst's requirements for routine microscopical examination of powdered food products for adulteration, partly because water is the best medium in most cases for showing up the structural characteristics of these substances and their adulterants, and partly because it serves best for the "rubbing out" process between thumb and finger under the cover-glass, whereby the sample is brought to the requisite degree of fineness.

Experience will soon show how far this rubbing out should be carried for the best effects. Gentle pressure should be applied, care being taken not to break the cover-glass, especially if the sample contain anything of a gritty nature. The rubbing should be continued till the coarser par-



ticles and overlying masses are separated and distributed uniformly, but if too long persisted in, the forms of the tissues, starch grains, and other characteristic portions will be partially destroyed and of too fragmentary a nature to be readily recognizable.

*Canada Balsam in Xylol* is a useful mountant for the examination of starches with polarized light. In this medium, under ordinary illumination, the starches are not plainly visible, since the refractive index of the two are nearly identical, but with crossed nicols the starch grains stand out clearly and distinctly in a dark background. If the material is not perfectly dry it should be soaked in absolute alcohol and then in chloroform or xylol until dehydrated.

*Glycerin*. — A mixture of equal parts of glycerin and water is perhaps the best medium for permanent mounts, but considerable skill is required to finish the preparation with cement on the edge of the cover-glass.

Glycerin jelly is more readily handled by the beginner since no cement is required.

*Glycerin Jelly* \* is prepared as follows: 1 part by weight of the finest French gelatin is soaked two hours in 6 parts of distilled water, after which 7 parts by weight of C. P. glycerin are added, and to each 100 parts of the mixture add 1 part of concentrated carbolic acid. Heat the mixture while stirring till flocculency disappears and filter through asbestos while warm, the asbestos being previously washed and put into the funnel while wet. The jelly is solid at ordinary temperatures, and must be warmed to melt. A small bit of this jelly is removed from the mass by a knife-blade and placed on the clean-slide, which is held over a gas flame till the jelly is melted. The powdered specimen being then shaken into the molten drop, the cover-glass is gently placed upon it (being brought down obliquely to avoid formation of air-bubbles) and pressed down in place.

**Microscopical Diagnosis.**—It is never safe to pass judgment on a spice or other food by the microscopical examination of a single portion. Several slides should be prepared with bits of the powder taken from different parts of the mass, before the character and extent of the adulteration can be safely determined. Care should be taken that the slide, the knife-blade, the water, and the medicine-dropper be perfectly clean and free from contamination with previous specimens.

It should be borne in mind that at best a composite powdered sample

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\* Botan. Centralbl., Bd. 1, p. 25.



is but a mechanical mixture of various tissues, and that no two portions will show exactly the same composition.

**Characteristic Features of Vegetable Foods under the Microscope.—**

The structural features of a powdered spice, examined microscopically, will be found to differ considerably in appearance from those of a thin, carefully mounted section of the same spice. Instead of the beautiful arrangement of cells and cell contents with the perfect order of various parts as seen in the mounted section, one finds in the powdered sample under the microscope what often appears to be a most confusing mass of fragments of various tissues. For this reason the most striking characteristics seem to vary with different observers, and it is a well-known fact that microscopists differ widely as to conceptions of size, shape, and ordinary appearance, even in the case of certain of the well-known starch grains. It is on this account that, irrespective of the description of others, the analyst should familiarize himself with the microscopical appearance of the foods with which he is dealing, as well as of their adulterants, forming his own standards as to what constitute the recognizable features, from specimens prepared by himself.

In the large variety of ground berries, buds, tubers, barks, etc., from which the spices and condiments are prepared, as well as in the grains, legumes, shells, fruit stones, and other materials forming the most familiar adulterants, the kinds of plant tissues and cell contents which, under the microscope, serve as distinguishing marks or guides for identification are comparatively few in number.

The most common of these varieties of cell tissue and of cell contents to be met with by the food microscopist in his work are briefly the following:

*Parenchyma*.—This is most abundant and widely distributed, forming as it does the thin-walled, cellular tissue of nearly all vegetable food substances. The walls of parenchyma cells are, as a rule, colorless and transparent. The forms of the cells are varied and are often sufficiently characteristic in themselves to identify the substance under examination.

*Sclerenchyma*, or stone cells, are the thick-walled woody cells forming the hard part of nut shells, fruit stones, and seed coverings, occurring also in some fruits and barks. These cells are more often colored and of various shapes but almost always irregular, sometimes elongated, as in cocoanut shells and olive stones, occasionally nearly rectangular, as in pepper shells, and sometimes polygonal or nearly circular.

In appearance the sclerenchyma cell commonly has a more or less



deep, central or axial cavity, from which small fissures extend through the thick walls. See Fig. 35.

Various shaped sclerenchyma cells are found in allspice, cassia,

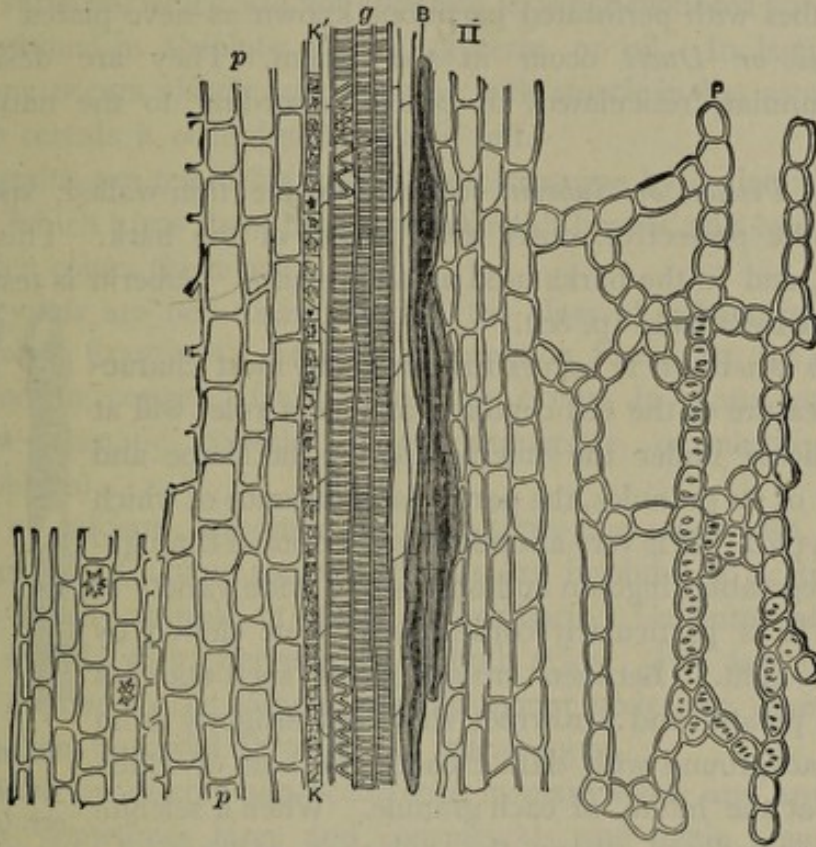


FIG. 34.—Typical Forms of Various Cell Tissues. Longitudinal section through a clove, showing: *Pp*, two forms of parenchyma; *B*, bast fibers; *g*, vascular and sieve tissue; *KK'*, cells with calcium oxalate crystals. (After Vogl.)

pepper, clove stems, nut shells, etc. Stone cells are optically active to polarized light, and between crossed nicols are very conspicuous by their bright appearance.



FIG. 35.—Sclerenchyma, or Stone-cell Tissue. A cross-section through the stone-cell layer of the fruit shell of black pepper. (After Vogl.)

*Fibro-vascular Bundles* are composed of three parts: the bast fibers, or mechanical elements, the phloem, and the xylem.



*Bast Fibers* are elongated, pointed sclerenchyma cells, of which flax fibers are examples.

*Sieve Tubes*, the characteristic elements of the phloem, are thin-walled tubes with perforated partitions known as sieve plates.

*Vessels or Ducts* occur in the xylem. They are designated as spiral, annular, reticulated, or pitted, according to the nature of the walls.

*Corky Tissue, or Suberin*, constitutes the thin-walled, spongy cells forming the protective, outer dead layers of the bark. This is found in cassia, and in the barks used as adulterants. Suberin is tested for by potassium hydroxide (p. 80).

*Starch* wherever it occurs furnishes the most characteristic feature of the cell contents, and, as a rule, will at once indicate under the microscope, by the shape and grouping of its granules, the particular substance of which it forms a part. It is very abundantly distributed throughout the vegetable kingdom and occurs in a wide variety of forms. It is particularly conspicuous when viewed by polarized light. Between crossed nicols such starches as corn, potato, and arrowroot show out brightly from a dark background with dark crosses, the bars of which intersect at the hilum of each granule. When a selenite plate is introduced above the polarizer, a beautiful play of colors is seen with various starches, a phenomenon which Blyth applies as a means of identification and classification, but which more modern microscopists regard as of minor importance to distinguishing the various starches morphologically. Starch is found naturally in the cereals, legumes, and many vegetables, in cassia, allspice, nutmeg, pepper, ginger, cocoa, and turmeric. The cereal and leguminous starches from their inertness and cheapness constitute the most common adulterants of the spices and of powdered foods in general. Starch grains are found in the cells of the parenchyma and in other cellular tissues. Iodine is the special reagent (p. 78).

*Gums and Resins* occur in characteristic forms among the cell contents of some of the spices. As an example, the portwine-colored lumps of gum in allspice furnish one of the most ready means of recognizing that spice microscopically. Resin is tested for microchemically with alkanna tincture (p. 79).

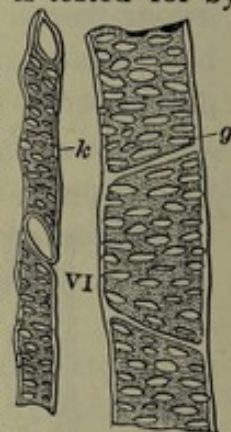


FIG. 36.—Reticulated Ducts of Chicory. (After Vogl.)



*Aleurone or Protein Grains* are found in many seeds, but are not especially characteristic. They somewhat resemble small starch grains. Most varieties of protein grains are soluble in water, but some are insoluble. The soluble varieties, which are not apparent in water-mounted specimens, must be examined in absolute alcohol, glycerin, or oil. In leguminous seeds aleurone occurs closely intermingled with starch in the same cells, while in the cereals it occupies the whole cell.

Protein grains are tested for under the microscope by iodine in potassium iodide, which turns them brown or yellowish brown, and by Millon's reagent, which colors them brick red.

*Plant Crystals* are not uncommon in the class of substances which the food analyst examines. Among the common forms are the piperin crystals found in pepper. Calcium oxalate occurs in many vegetable products as prismatic crystals, crystal aggregates, or needle-shaped crystals (raphides).

Crystals of calcium carbonate are sometimes met with also, as, for example, in hops. Calcium oxalate crystals are insoluble in acetic acid, while being readily soluble in dilute hydrochloric. Calcium carbonate crystals are soluble with effervescence in both acids. The acid reagents are directly applied to the sample in water-mount under the cover-glass, and the reaction observed through the microscope.

*Fat Globules* are of common occurrence in many foods and appear of various sizes, sometimes large and conspicuous, and again almost lost sight of because of their minuteness. They are sometimes colorless, as in mace, and sometimes deeply tinted, as in cayenne. Alkanna tincture is used as a reagent for fat (p. 79).

*Other Cell Contents* of less importance, but which may be identified by the microscope with reagents, are tannic acid (tested for by chloriodide of zinc and ferric acetate (pp. 78 and 79), and various essential oils, for the detection of which alkanna tincture is employed.

#### REAGENTS IN FOOD MICROSCOPY.

Unless a more extended microscopical investigation of vegetable food substances is contemplated than is involved in the mere identification of adulterants, the analyst will have little need for reagents other than iodine in potassium iodide, chloral hydrate solution, and potassium hydroxide solution, the last two for clearing, but will depend almost entirely on the form and appearance of the various tissues or tissue fragments, as well as on the abundance, shape, and distribution of such distinctive cell contents as the starches, fat globules, or crystals.



Analytical reagents are applied to the water-mounted sample by means of a glass rod or pipette, with which a drop of the reagent is deposited on the sample upon the slide, having previously removed the cover, which is afterwards replaced. Or, without removing the cover-glass, a drop of the reagent is placed in contact with one side of it on the slide. Along the opposite side of the cover is then placed a piece of filter-paper. The latter withdraws by capillary attraction a portion of the water from under the cover-glass, and this is replaced by the reagent, which thus intermingles with the particles of the substance.

The following reagents include those needed in routine work as well as some others suited for studies of the general nature of tissues and cell contents.

**A. Analytical Reagents.—Iodine in Potassium Iodide.**—Two grams of crystallized potassium iodide are first dissolved in 100 cc. of distilled water and the solution is saturated with iodine.

This reagent is indispensable for the identification of starch, especially when the latter is present in minute quantities. Starch granules when moistened with water are turned blue by iodine, the reaction being exceedingly delicate under the microscope, even when the starch granules are very minute and insignificant without the reagent.

Iodine in connection with sulphuric acid is also useful in distinguishing pure cellulose from its various modifications, such as lignin and suberin. For this purpose the water-mounted sample is first permeated with the iodine reagent, after which concentrated sulphuric acid is applied, with the result that all pure cellulose is turned a deep-blue color, while the modified forms of cellulose are colored yellow or brown. The cellulose is first converted by the sulphuric acid into a carbohydrate isomeric with starch, known as amyloid.

Protein grains are colored brown or yellow brown by the action of iodine.

**Chloriodide of Zinc.**—Pure zinc is dissolved in concentrated hydrochloric acid to saturation, and an excess of zinc added. The solution is then evaporated to about the consistency of concentrated sulphuric acid, after which it is first saturated with potassium iodide, and finally with iodine.

This reagent may be used instead of sulphuric acid and iodine for the detection of cellulose, since the zinc chloride converts the cellulose into amyloid, which the reagent colors blue.

Chloriodide of zinc is useful for detecting tannic acid in cell contents. For this purpose the above reagent is much diluted by the addition of



a 20% solution of potassium iodide. In this diluted form, when applied to the sample, a reddish or violet coloration is imparted to cell contents having tannin.

*Phenol-hydrochloric Acid* is prepared by saturating concentrated hydrochloric acid with the purest crystallized carbolic acid. Wood fiber, or lignin, when treated with a drop of this reagent under the cover-glass, and exposed for half a minute to the direct sunlight, will be colored an intense green, which quickly fades.

*Indol*.—Several crystals of indol are freshly dissolved in warm water. Lignified cell walls assume a deep-red color, when the specimen to be examined is treated first with a drop of the indol reagent, and afterwards washed with dilute sulphuric acid, 1:4.

*Millon's Reagent*.—This is prepared by dissolving metallic mercury in its weight of concentrated nitric acid, and diluting with an equal volume of water. This reagent, which should be freshly prepared, is of use in testing for protein compounds, which turn brick red when treated with it, especially on gently warming the slide.

*Tincture of Alkanna*.—A 70 or 80% alcoholic extract of alkanna root, when kept in contact with resins, fixed oils, fats, or essential oils for a short time, stains these cell contents a lively red. The staining is hastened by the aid of heat. Essential oils and resins are soluble in strong alcohol, while fixed oils and fats are insoluble, hence the distinction between these classes of cell contents may be made by the application of alcohol to the alkanna-stained specimen.

*Ferric Chloride, Ferric Acetate, or Ferric Sulphate*, used in dilute aqueous solution, are all applicable as reagents for tannic acid, which, when present in appreciable amount, will be colored green or blue by either of these reagents.

**B. Clarifying Reagents.**—Many of the harder cellular tissues are too opaque for careful examination, and may be rendered transparent by clarifying or bleaching. The simplest and for many purposes the most satisfactory method for clearing the tissues is by boiling a water mount, replacing the water lost by evaporation. Proceeding in this manner, there is ordinarily no danger of the slide or cover-glass breaking; if the boiling is carried out without a cover-glass, the slide is almost sure to break. A portion of the powdered sample is either boiled with a drop of the reagent under the cover-glass or is allowed to soak for hours or even days in the reagent, using a drop of the same reagent as a medium for examination on the object-glass, instead of water. The clarifying reagents most commonly used are the following:



*Chloral Hydrate*.—A 60% solution.

*Ammonia*.—Concentrated, or 28% ammonia is commonly used.

*Potassium Hydroxide*, used in various degrees of concentration, often in dilute solution, say 5%. This reagent, added to a water mount, causes swelling of the cell wall, and dissolves intercellular substances and protein. It bleaches most of the coloring matters, destroys the starch, and forms soluble soaps with the fats. Potassium hydroxide is also used in testing for suberin, which is extracted from corky tissue on boiling with the reagent, and appears as yellow drops.

*Schultze's Macerating Reagent* (concentrated nitric acid and chlorate of potassium) is best used by placing the powder or bit of tissue to be treated in a test-tube with an equal volume of potassium chlorate crystals, adding about 2 cc. of concentrated nitric acid, and warming the tube till bubbles are evolved freely, or until the necessary separation of cells is effected. The sample is then removed and washed with water.

By this treatment, bast and wood fibers as well as stone cells are readily separated from other tissues.

*Cuprammonia* (Schweitzer's Reagent).—This is prepared by adding slowly a solution of copper sulphate to an aqueous solution of sodium hydroxide, forming a precipitate of cupric hydroxide, which is separated by filtration, washed, and dissolved in concentrated ammonia. It should be freshly prepared, and is never fit for use unless it is capable of immediately dissolving cotton. Indeed its chief use is as a test for cellulose, which it readily dissolves. In observing this reaction under the microscope, the powdered specimen under the cover-glass should be only slightly damp before a drop of the fresh reagent is applied. The cell walls are seen to swell up and gradually become more and more indistinct, till they finally disappear.

Cuprammonia is also used as a test for pectose, which occurs in many cell walls; often intermixed with cellulose. When treated with this reagent, cellular tissue containing pectose is acted upon in such a manner that a delicate framework of cupric pectate is sometimes left behind, after the dissolution of the cellulose with which it is mingled.\*

#### PHOTOMICROGRAPHY.

The photomicrograph serves as a simple means of keeping permanent records of unusual forms of adulteration encountered in the course of routine examination. Besides this, the photomicrograph has at times proved its usefulness as a means of evidence in court, showing as it does with faithfulness the presence of a contested adulterant. It is true

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\* Poulsen, Botanical Micro-chemistry, p. 15.



that from an artistic and didactic standpoint the photomicrograph of a powdered sample is often disappointing, due to the fact that ordinarily much of the field is out of focus, unless a very simple homogeneous subject is photographed, as, for instance, starch. As compared with the carefully prepared drawing of a section, which shows minute details of structure, the photomicrograph portrays what happens to be in focus.

SUMMARY OF MICROCHEMICAL REACTIONS FOR IDENTIFYING  
CELLULAR TISSUE AND CELL CONTENTS. BASED ON BEHRENS'.\*

	Iodine in Potassium Iodide.	Chlor-iodide of Zinc.	Iodine and Sulphuric Acid.	Cuprammonia.	Potassium Hydroxide.	Concentrated Sulphuric Acid.	Schultze's Mixture.
Cellulose, cell substance.	Yellow to brownish	Violet	Blue	Dissolves	Swells up	Dissolves	Dissolves
Lignin, wood substance.	Yellow	Yellow	Yellow to brownish	Insoluble	Dissolves	Dissolves	Dissolves easily
Middle lamella, inter-cellular substance. . .	Yellow	Yellow	Yellow	Insoluble	.....	.....	Dissolves easily
Suberin, cork substance.	Yellow or brownish	Yellow or brown	Brown	Insoluble	Insoluble in cold. By boiling it comes out in drops	Insoluble	Gives ceric acid reaction†
Starch. ....	Blue	.....	.....	.....	Dissolves	.....	.....
Protein. ....	Brown yellow	.....	.....	.....	Dissolves	.....	.....
Gums and resins. ....	.....	.....	.....	.....	.....	.....	.....
Fat. ....	.....	.....	.....	.....	Saponifies	.....	.....
Essential oils. ....	.....	.....	.....	.....	.....	.....	.....
Tannin. ....	.....	Reddish to violet	.....	.....	.....	.....	.....
Calcium oxalate crystals	.....	.....	.....	.....	.....	.....	.....
Calcium carbonate "	.....	.....	.....	.....	.....	.....	.....

	Phenol-hydrochloric Acid.	Indol.	Ferric Acetate or Sulphate.	Alkanna Tincture.	Hydrochloric Acid.	Acetic Acid.	Millon's Reagent.
Cellulose, cell substance.	Uncolored	Uncolored	.....	.....	.....	.....	.....
Lignin, wood substance.	Green	Cherry red	.....	.....	.....	.....	.....
Middle lamella, inter-cellular substance. ....	Green	Cherry red	.....	.....	.....	.....	.....
Suberin, cork substance.	Uncolored	Uncolored	.....	.....	.....	.....	.....
Starch. ....	.....	.....	.....	.....	.....	.....	.....
Protein. ....	.....	.....	.....	.....	.....	.....	Brick red
Gums and resins. ....	.....	.....	.....	Bright red	.....	.....	.....
Fat. ....	.....	.....	.....	Bright red	.....	.....	.....
Essential oils. ....	.....	.....	.....	Bright red	.....	.....	.....
Tannin. ....	.....	.....	Blue or green	.....	.....	.....	.....
Calcium oxalate crystals	.....	.....	.....	.....	Soluble without effervescence	Insoluble	.....
Calcium carbonate "	.....	.....	.....	.....	Soluble with effervescence	Soluble with effervescence	.....

\* Microscopical Investigation of Vegetable Substances, page 356.

† When treated with the reagent, suberin forms masses of ceric acid, soluble in ether, alcohol, or chloroform.

While the analyst examines microscopically the ordinary powdered spice, for example, he constantly moves with his hand the fine adjustment-screw, bringing into focus different parts of the field successively. This



he does unconsciously, so that he does not realize how far from flat the field actually is till he undertakes to photograph it, when, as a rule, only a small portion is in good focus. It is therefore impossible in one photograph to show successfully many varied forms of tissue or cell contents in the powder, but separate photographs should be made as far as possible with only a little in each. Thus, for example, with a composite subject like powdered cassia bark, it would be very difficult to show starch, stone cells, and bast fibers in one field, all in equally good focus, and, for the best results only, one, or at most two, such varied groups of elements should be shown in one picture.

**Appurtenances and Methods of Procedure.**—The temporary method of water-mounting employed by the analyst in routine examination presents many difficulties from a photographic point of view. The vibrating motion of the particles is very annoying, and some skill is required in using just the right amount of water, in avoiding air-bubbles, in waiting the requisite amount of time before exposing the plate for the vibratory motion to cease, and, on the other hand, avoiding too long delay, which would result in the evaporation of the water, and the consequent breaking up of the field. In the writer's experience, however, in spite of these difficulties, the water-mounting gives decidedly the clearest results, and, with patience on the part of the operator, it is in many ways the most desirable method of mounting for photographic purposes. It is in fact the method employed in making most of the photomicrographs of powdered specimens that appear in the plates at the end of this volume, though a few were mounted in glycerin jelly, and the starches for the polarized-light pictures in Canada balsam. The sections of tissues shown in the plates were mounted some in glycerin and others in glycerin jelly.

Experience has shown that two degrees of magnification well calculated to bring out the chief characteristics of the spices and their adulterants in a photomicrograph are 125 and 250 diameters. The starches, which are the most common of any one class of adulterants, vary very widely in the size of their granules. With these the larger magnification of 250 has been found satisfactory, while the general appearance of the composite ground-spice itself under the microscope, as well as that of such adulterants as ground bark, sawdust, chicory, pea hulls, and the like, is best shown with the lower power of 125.\*

\* The degrees of magnification adopted in the originals of most of the photomicrographs illustrated in the accompanying plates are accordingly 125 and 250, but in the process of lithographing, the photographs were slightly reduced, so that the actual scales in the reproduction are 110 and 220 respectively.



The object, mounted in the manner above described, is best examined when held in a mechanical stage, furnished with micrometer adjustments, in such a manner that a typical field may be selected and held in place long enough to photograph.

**The Camera.**—This need not of necessity be complicated, but may consist simply of a horizontal wooden base on which the microscope

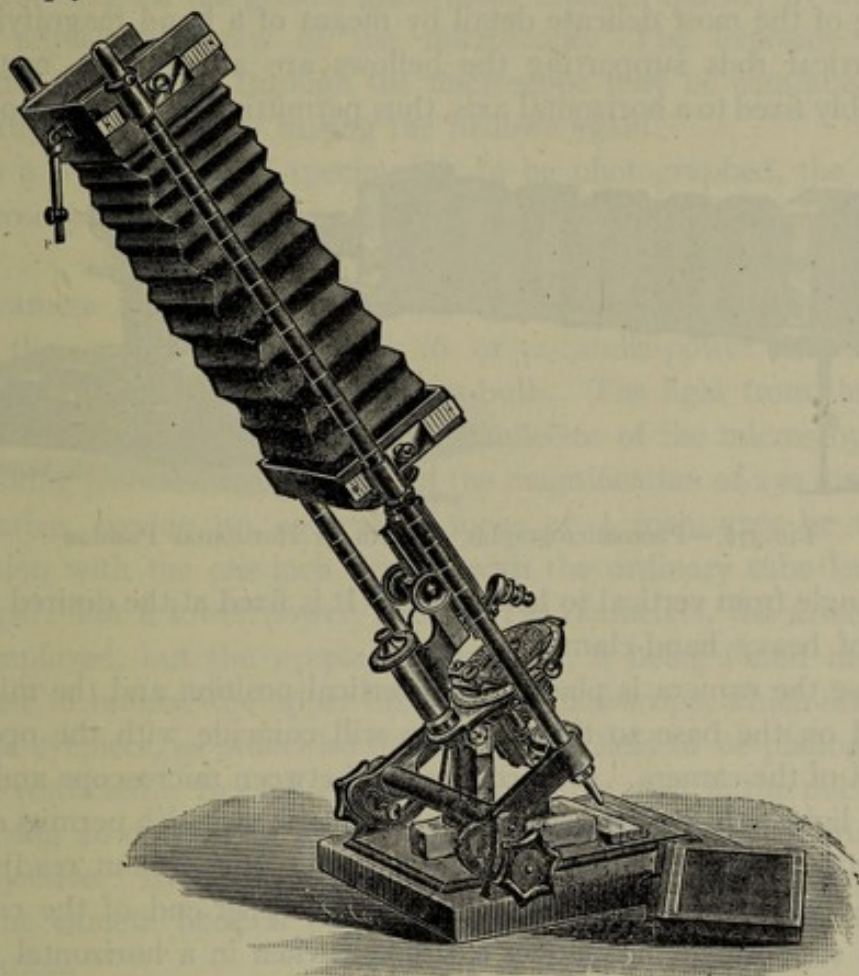


FIG 37a.—A Convenient Photomicrographic Camera.

rests, and an upright board firmly secured to the base, carrying a frame for an interchangeable ground glass and plate-holder, with a rubber gauze skirt hanging from the frame, adapted to be gathered and tied about the top of the microscope-tube. Means are further provided, as by a slotted guide and screw, for adjusting the frame at any desired height on the upright board.\*

A more convenient form of apparatus now employed by the writer is that shown in Figs. 37a and 37b.

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\* Such a contrivance as this was employed in making some of the accompanying photomicrographs.



The base is a solid iron plate upon which the microscope rests (any microscope may be used with this camera), and above which the camera bellows is supported on two solid steel rods. The bellows is supported at either end on wooden frames.

The ground glass is provided with a central transparent area, formed by cementing a cover-glass upon the ground glass, and permits the accurate focusing of the most delicate detail by means of a hand magnifying-glass. The vertical rods supporting the bellows are attached to metal arms, immovably fixed to a horizontal axis, thus permitting the camera to be tilted

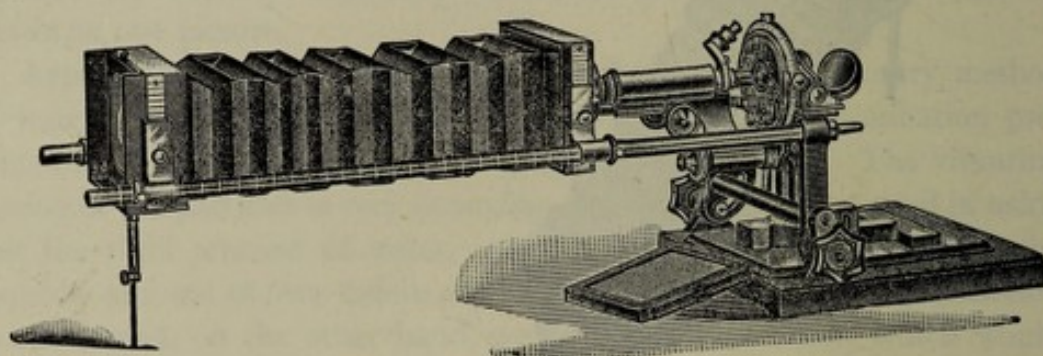


FIG 37b.—Photomicrographic Camera in Horizontal Position

to any angle from vertical to horizontal. It is fixed at the desired angle by means of heavy hand-clamps.

In use the camera is placed in a vertical position and the microscope adjusted on the base so that its tube will coincide with the opening in the front of the camera. The connection between microscope and camera is made light-tight by means of a double chamber, which permits considerable vertical motion of the tube of the microscope without readjustment. A jointed telescoping rod is attached to the upper end of the camera to act as a support, giving perfect steadiness when in a horizontal position, and folding down parallel with the bellows so as to be out of the way when in any other position.

*Amplification.*—The vertical rods are graduated in inches for determining the amount of amplification, and to show when the ground glass is at right angles to the optical axis. The following simple rule for determining the amount of amplification will give sufficiently accurate results. When photographing without the eyepiece, divide the distance of the ground glass from the stage of the microscope in inches, by the focal length in inches of the objective used. When photographing with the eyepiece, proceed as above and multiply the result by the quotient obtained by dividing 10 by the focus in inches of the eyepiece used.



*Adjustment and Manipulation.*—The microscope can be placed in any position desired, and the camera adjusted to it. The bellows can then be raised and the microscope used as though no camera were present. When an object is to be photographed, the bellows may be slid into position without in any way disturbing the arrangement of light or object, the final focusing on the ground glass being effected quickly by means of the fine adjustment-screw of the microscope. The exposure having been made, observation through the microscope may be continued without interruption by simply raising the bellows again.

When a water-mounted specimen is to be photographed, the camera and microscope tube should be vertical instead of inclined as shown in the cut.

The camera is best kept in a dark room where the exposures are to be made, the source of light being a 16- or 32-candle-power electric lamp, preferably provided with a ground-glass bulb. The light from this lamp is first carefully centered by moving the reflector of the microscope.

In making pictures, for instance, of the magnification of 250 diameters, the objective, having an equivalent focus of  $\frac{1}{8}$  inch, may be used in combination with the one-inch ocular, with the ordinary tube length of microscope. For a lower power, such as 125 diameters, the same objective is employed, but the eyepiece is left out, it being found necessary in this case to remove the upper tube of the microscope, which ordinarily carries the eyepiece, as otherwise the size of the field to be photographed would be restricted. In each case a diaphragm is used in the microscope stage, having an opening of about the same size as that of the front lens of the objective. By means of a stage micrometer scale, the proper position of the camera back is previously determined to give the required magnification.



## CHAPTER VI.

### THE REFRACTOMETER.

THE refractive index ranks in importance with the specific gravity as a means of determining the identity and purity of various food products, as well as of estimating the percentage of valuable constituents. Various forms of refractometer are used in food analysis.

The *Abbé refractometer* is of general application in determining the refractive index of fats, fatty oils, waxes, and essential oils, in estimating the solids in saccharine substances, and in other analytical operations. It employs but a few drops of the material, and reads the refractive index directly, using ordinary white light.

The *immersion refractometer*, an instrument of recent introduction, is suited for the examination of milk serum to detect added water therein, the detection and determination of methyl alcohol in ethyl alcohol, and the standardization of a wide variety of solutions. The instrument is immersed directly in the liquid to be examined, the degree of refraction being indicated on an arbitrary scale.

The *Pulfrich* is used with the sodium light, and requires a larger amount of material than the Abbé, the liquid being held in a cylinder above the prism. The scale reading is in angular degrees, from which the index of refraction is calculated by a formula or from a table. The instrument is provided with a temperature-controlling apparatus.

In the *Amagat and Jean* or *oleo-refractometer*, an outer and an inner cylinder are respectively filled with an oil of known value or purity, and with the oil to be examined. By the comparative displacement to the right or left of a beam of white light passing through both cylinders, the displacement being read in degrees on an arbitrary scale, the refraction of an oil may be measured. Two oils may thus be readily compared under the same conditions, one of known purity, for example, with a doubtful sample of the same kind.

The *butyro-refractometer* and the *Wollny milk fat refractometer* (p. 126) are, as their names imply, instruments primarily intended for more restricted fields of work than the foregoing. They involve the same principle as the Abbé, but are simpler in construction and have arbitrary scales.

Unless such widely varying substances as the waxes and the essential oils are to be studied, the Zeiss butyro-refractometer, though primarily



intended for the examination of butter and lard, answers most of the purposes of the Abbé instrument with the advantage of greater simplicity, being equally well adapted for examining all the common edible oils and fats, as well as other materials.

#### THE ZEISS BUTYRO-REFRACTOMETER.

This instrument (shown in Fig. 38) is so constructed that the degree of refraction of a beam of light, which passes obliquely through a thin

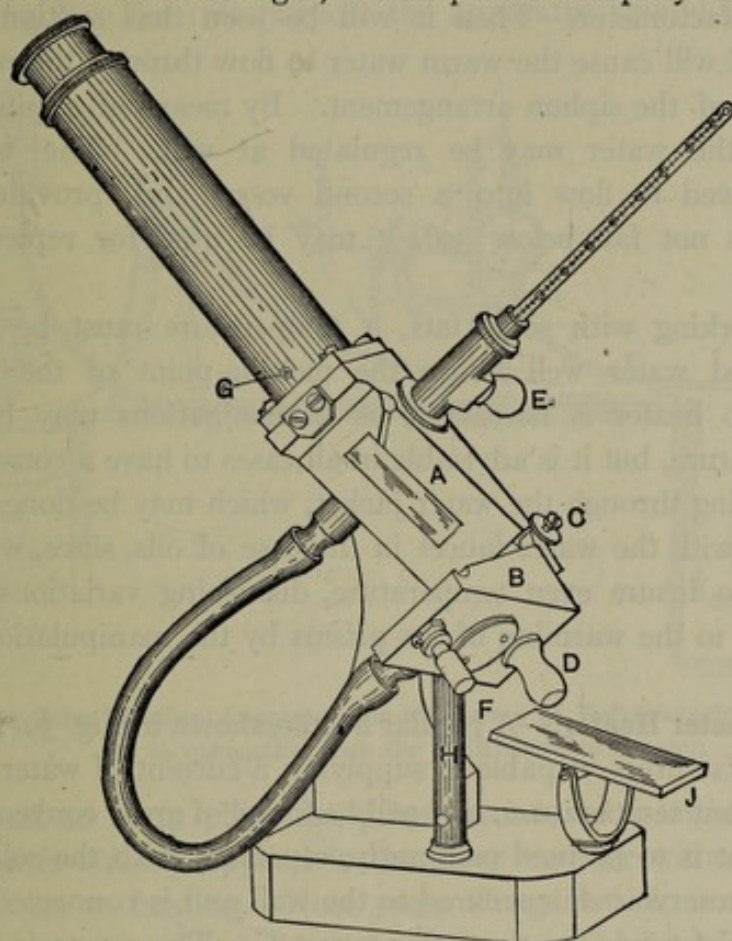


FIG. 38.—The Zeiss Butyro-refractometer.

film of the fat, is read on an arbitrary scale of sufficient extent to cover the widest limits of deviation possible for butter fat and oleomargarine under ordinary temperatures.

The graduation is in divisions from 1 to 100, covering a variation in refractive indices of from 1.4220 to 1.4895. *A* and *B* are the two hinged hollow portions of the prism casing of the instrument, so arranged that when closed together the melted fat is held in a film of sufficient thickness between the two opposed transparent prism surfaces, the beam of light, either diffused daylight or lamplight, being reflected through it by means of the mirror *J*. The transparent scale is within the telescope tube at the height indicated by *G*.



The refractometer is connected to any kind of heating arrangement, which admits of warm water being transmitted through the prism casing, in at *D* and out at *E*. A simple arrangement, which suffices for all ordinary cases, may expeditiously be improvised in the following manner: Fill a vessel of say 2 gallons capacity with water of  $40^{\circ}$  to  $50^{\circ}$  C. Into this vessel dip the free end of an india-rubber tube slipped over the nozzle *D* and let the vessel be placed at a height of about one-half or one yard above the refractometer. Then it will be seen that suction at a tube attached to *E* will cause the warm water to flow through the prism casing by the action of the siphon arrangement. By means of a pinch clip the velocity of the water may be regulated at will. The waste water may be allowed to flow into a second vessel and, provided its temperature does not fall below  $30^{\circ}$ , it may be used for replenishing the upper vessel.

When working with solid fats, a temperature must be maintained by the heated water well above the melting-point of the fat. With liquid oils no heater is necessary, as determinations may be made at room temperature, but it is advisable in all cases to have a constant stream of water passing through the water jacket, which may be done by directly connecting it with the water faucet in the case of oils, since, without such precautions to insure even temperature, disturbing variations are liable to occur, due to the warming of the prisms by the manipulation of cleaning, etc.

**Refractometer Heater.**—A regular heater, shown in Fig. 39, is furnished by the manufacturers, capable of supplying a current of water of approximately constant temperature, and will be found of great convenience when the instrument is to be used constantly, especially with the solid fats.

A supply reservoir *A* is secured to the wall and is connected by means of a rubber inlet tube *G* to the water faucet *C*. The reservoir is provided with a waste overflow pipe and with an outlet tube *D*, the flow through the latter being regulated by the cock *H*. The tube *D* leads to the spiral heater *HS*, which is heated by a Bunsen burner. From the heater the tube *E* conducts the warm water through the refractometer, from which it flows through the tube *F*, either directly into the sink, or into the intermediate vessel *B*. The temperature of the water is regulated by adjusting the cock *H*, and the height of the flame of the Bunsen burner.

**Manipulation of the Butyro-refractometer.**—The prism casing is first opened by giving about half a turn to the right to the pin *F*, Fig. 38, until it meets with a stop. Then simply turn the half *B* of the prism



casing aside. Pillar *H* holds *B* in the position shown in Fig. 38. The prism and metallic surfaces must now be cleaned with the greatest care, the best means for this purpose being soft linen, moistened with a little alcohol or benzine.

If the sample to be examined is a solid fat, maintain the temperature above the melting-point, and apply by a glass rod a drop or two of the clear melted fat (filtered if turbid) to the surface of the prism contained in the casing *B*. For this purpose the apparatus should be raised with

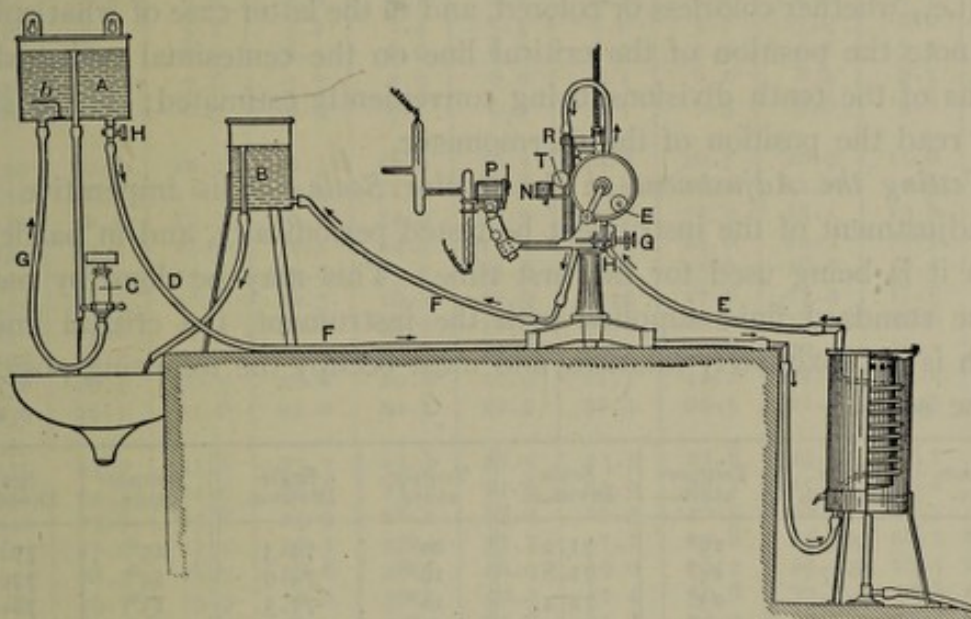


FIG. 39.—The Zeiss Heating Apparatus for all Forms of Refractometer. Shown in the cut in connection with the Pulfrich refractometer.

the left hand so as to place the prism surface in a horizontal position. A liquid oil is directly applied in the same manner without preliminary precautions as to heating. Now press *B* against *A*, and place *F* by turning it in the opposite direction, in its original position; thereby *B* is prevented from falling back, and both prism surfaces are kept in close contact. Place the instrument again upon its sole plate.

While looking into the telescope, give the mirror *J* such a position as to render the critical line, which separates the bright left part of the field from the dark right part, distinctly visible. It may also be necessary to move or turn the instrument about a little. First it will be necessary to ascertain whether the space between the prism surfaces be uniformly filled with oil or fat, failing which the critical line will not be distinct. For this purpose examine the rectangular image of the prism surface formed about 1 cm. above the ocular with a hand magnifier or with the



naked eye, placing the latter at its proper distance from the ocular. Finally adjust the movable front part of the ocular so that the scale becomes clearly visible.

By allowing a current of water of constant temperature to flow through the apparatus some time previous to the taking of the reading, the at first somewhat hazy critical line approaches in a short time, generally after a minute, a fixed position, and quickly attains its greatest distinctness. When this point has been reached, note the appearance of the critical line (i.e., whether colorless or colored, and in the latter case of what color); also note the position of the critical line on the centesimal scale, which admits of the tenth divisions being conveniently estimated; at the same time read the position of the thermometer.

*Testing the Adjustment of the Ocular Scale.*—It is imperative that the adjustment of the instrument be tested periodically, and in particular when it is being used for the first time. This may be done by means of the standard fluid supplied with the instrument, the critical line of which is approximately colorless, and must occupy the following positions in the scale.

Temperature.	Scale Division.	Temperature.	Scale Division.	Temperature.	Scale Division.	Temperature.	Scale Division.
30°	68.1	25°	71.2	20°	74.3	15°	77.3
29°	68.7	24°	71.8	19°	74.9	14°	77.9
28°	69.3	23°	72.4	18°	75.5	13°	78.6
27°	70.0	22°	73.0	17°	76.1	12°	79.2
26°	70.6	21°	73.6	16°	76.7	11°	79.8
25°	71.2	20°	74.3	15°	77.3	10°	80.4

The fractional parts of a degree can accordingly easily be brought into calculation ( $0.1 = 0.06$  scale div.). Deviations of 1 to 2 decimals of the scale divisions are of no consequence, and are in most cases due to inexact determinations of temperature. Should, however, careful tests result in the discovery of greater deviations, readjustment of the scale will be necessary, which may be effected by means of a watch-key supplied with the instrument, in accordance with the values given in the above table. The watch-key is inserted at *G* in Fig. 38, and by its means the position of the objective, and therefore that of the critical line with respect to the scale may be altered.

*Transformation of Scale Divisions into Indices of Refraction.*—The following table, adapted from that of Pulfrich, enables one to convert scale readings on the butyro-refractometer into indices of refraction,  $n_D$ , and vice versa:



## EQUIVALENTS OF INDICES OF REFRACTION AND BUTYRO-REFRACTOMETER READINGS.

Refractive Index. $n_D$ .	Fourth Decimal of $n_D$ .									
	0	1	2	3	4	5	6	7	8	9
	SCALE READINGS.									
I.422	0.0	0.1	0.2	0.4	0.5	0.6	0.7	0.9	1.0	1.1
I.423	1.2	1.4	1.5	1.6	1.7	1.9	2.0	2.1	2.2	2.4
I.424	2.5	2.6	2.7	2.8	3.0	3.1	3.2	3.3	3.5	3.6
I.425	3.7	3.8	4.0	4.1	4.2	4.3	4.5	4.6	4.7	4.8
I.426	5.0	5.1	5.2	5.4	5.5	5.6	5.7	5.9	6.0	6.1
I.427	6.2	6.4	6.5	6.6	6.8	6.9	7.0	7.1	7.2	7.4
I.428	7.5	7.6	7.7	7.9	8.0	8.1	8.2	8.4	8.5	8.6
I.429	8.7	8.9	9.0	9.1	9.2	9.4	9.5	9.6	9.8	9.9
I.430	10.0	10.1	10.3	10.4	10.5	10.6	10.7	10.9	11.0	11.1
I.431	11.3	11.4	11.5	11.6	11.8	11.9	12.0	12.2	12.3	12.4
I.432	12.5	12.7	12.8	12.9	13.0	13.2	13.3	13.5	13.6	13.7
I.433	13.8	14.0	14.1	14.2	14.4	14.5	14.6	14.7	14.9	15.0
I.434	15.1	15.3	15.4	15.5	15.6	15.8	15.9	16.0	16.2	16.3
I.435	16.4	16.6	16.7	16.8	17.0	17.1	17.2	17.4	17.5	17.6
I.436	17.8	17.9	18.0	18.2	18.3	18.4	18.5	18.7	18.8	18.9
I.437	19.1	19.2	19.3	19.5	19.6	19.7	19.8	20.0	20.1	20.3
I.438	20.4	20.5	20.6	20.8	20.9	21.1	21.2	21.3	21.4	21.6
I.439	21.7	21.8	22.0	22.1	22.2	22.4	22.5	22.6	22.7	22.9
I.440	23.0	23.2	23.3	23.4	23.5	23.7	23.8	23.9	24.1	24.2
I.441	24.3	24.5	24.6	24.7	24.8	25.0	25.1	25.2	25.4	25.5
I.442	25.6	25.8	25.9	26.1	26.2	26.3	26.5	26.6	26.7	26.9
I.443	27.0	27.1	27.3	27.4	27.5	27.7	27.8	27.9	28.1	28.2
I.444	28.3	28.5	28.6	28.7	28.9	29.0	29.2	29.3	29.4	29.6
I.445	29.7	29.9	30.0	30.1	30.3	30.4	30.6	30.7	30.8	30.9
I.446	31.1	31.2	31.4	31.5	31.6	31.8	31.9	32.1	32.2	32.3
I.447	32.5	32.6	32.8	32.9	33.0	33.2	33.3	33.5	33.6	33.7
I.448	33.9	34.0	34.2	34.3	34.4	34.6	34.7	34.9	35.0	35.1
I.449	35.3	35.4	35.6	35.7	35.8	36.0	36.1	36.3	36.4	36.5
I.450	36.7	36.8	37.0	37.1	37.2	37.4	37.5	37.7	37.8	37.9
I.451	38.1	38.2	38.3	38.5	38.6	38.7	38.9	39.0	39.2	39.3
I.452	39.5	39.6	39.7	39.9	40.0	40.1	40.3	40.4	40.6	40.7
I.453	40.9	41.0	41.1	41.3	41.4	41.5	41.7	41.8	42.0	42.1
I.454	42.3	42.4	42.5	42.7	42.8	43.0	43.1	43.3	43.4	43.6
I.455	43.7	43.9	44.0	44.2	44.3	44.4	44.6	44.7	44.9	45.0
I.456	45.2	45.3	45.5	45.6	45.7	45.9	46.0	46.2	46.3	46.4
I.457	46.6	46.7	46.9	47.0	47.2	47.3	47.5	47.6	47.7	47.9
I.458	48.0	48.2	48.3	48.5	48.6	48.8	48.9	49.1	49.2	49.4
I.459	49.5	49.7	49.8	50.0	50.1	50.2	50.4	50.5	50.7	50.8
I.460	51.0	51.1	51.3	51.4	51.6	51.7	51.9	52.0	52.2	52.3
I.461	52.5	52.7	52.8	53.0	53.1	53.3	53.4	53.6	53.7	53.9
I.462	54.0	54.2	54.3	54.5	54.6	54.8	55.0	55.1	55.3	55.4
I.463	55.6	55.7	55.9	56.0	56.2	56.3	56.5	56.6	56.8	56.9
I.464	57.1	57.3	57.4	57.6	57.7	57.9	58.0	58.2	58.3	58.5
I.465	58.6	58.8	58.9	59.1	59.2	59.4	59.5	59.7	59.8	60.0
I.466	60.2	60.3	60.5	60.6	60.8	60.9	61.1	61.2	61.4	61.5
I.467	61.7	61.8	62.0	62.2	62.3	62.5	62.6	62.8	62.9	63.1
I.468	63.2	63.4	63.5	63.7	63.8	64.0	64.2	64.3	64.5	64.7
I.469	64.8	65.0	65.1	65.3	65.4	65.6	65.7	65.9	66.1	66.2



## EQUIVALENTS OF INDICES OF REFRACTION AND BUTYRO-REFRACTOMETER READINGS—(Continued).

Refractive Index. $n_D$ .	Fourth Decimal of $n_D$ .									
	0	1	2	3	4	5	6	7	8	9
	SCALE READINGS.									
1.470	66.4	66.5	66.7	66.8	67.0	67.2	67.3	67.5	67.7	67.8
1.471	68.0	68.1	68.3	68.4	68.6	68.7	68.9	69.1	69.2	69.4
1.472	69.5	69.7	69.9	70.0	70.2	70.3	70.5	70.7	70.8	71.0
1.473	71.1	71.3	71.4	71.6	71.8	71.9	72.1	72.2	72.4	72.5
1.474	72.7	72.9	73.0	73.2	73.3	73.5	73.7	73.8	74.0	74.1
1.475	74.3	74.5	74.6	74.8	75.0	75.1	75.3	75.5	75.6	75.8
1.476	76.0	76.1	76.3	76.5	76.7	76.8	77.0	77.2	77.3	77.5
1.477	77.7	77.9	78.1	78.2	78.4	78.6	78.7	78.9	79.1	79.2
1.478	79.4	79.6	79.8	80.0	80.1	80.3	80.5	80.6	80.8	81.0
1.479	81.2	81.3	81.5	81.7	81.9	82.0	82.2	82.4	82.5	82.7
1.480	82.9	83.1	83.2	83.4	83.6	83.8	83.9	84.1	84.3	84.5
1.481	84.6	84.8	85.0	85.2	85.3	85.5	85.7	85.9	86.0	86.2
1.482	86.4	86.6	86.7	86.9	87.1	87.3	87.5	87.6	87.8	88.0
1.483	88.2	88.3	88.5	88.7	88.9	89.1	89.2	89.4	89.6	89.8
1.484	90.0	90.2	90.3	90.5	90.7	90.9	91.1	91.2	91.4	91.6
1.485	91.8	92.0	92.1	92.3	92.5	92.7	92.9	93.0	93.2	93.4
1.486	93.6	93.8	94.0	94.1	94.3	94.5	94.7	94.8	95.0	95.2
1.487	95.4	95.6	95.8	96.0	96.1	96.3	96.6	96.7	96.9	97.0
1.488	97.2	97.4	97.6	97.8	98.0	98.1	98.3	98.5	98.7	98.9
1.489	99.1	99.2	99.4	99.6	99.8	100.0				

*The Critical Line.*—It should be remembered that the instrument is primarily intended for use with butter, and that the prisms are so constructed that the critical line of pure butter is colorless, while various other fats and oils, notably oleomargarine, which have greater dispersive powers than natural butter, show a colored critical line. When too great dispersion is apparent to render possible an accurate reading, or when the critical line presents very broad fringes, as with linseed oil, poppyseed oil, turpentine, etc., use a sodium light, obtained by the application of table salt to the Bunsen gas flame, or the diffused daylight may be reflected in the mirror through a flat bottle filled with a dilute solution of potassium bichromate, to give a yellow light.

The advantages of the refractometer for examination of fats and oils consist in the convenience with which very accurate determinations of the refractive index may be made at any temperature between 10° and 50° C., inclusive of thermal variations of refractive powers, and also in the possibility which it affords of distinguishing substances by their different dispersive powers, rendered visible by the different coloring of the critical line, a red-colored critical line being indicative of a relatively low dispersive power, a blue line of relatively high dispersion.



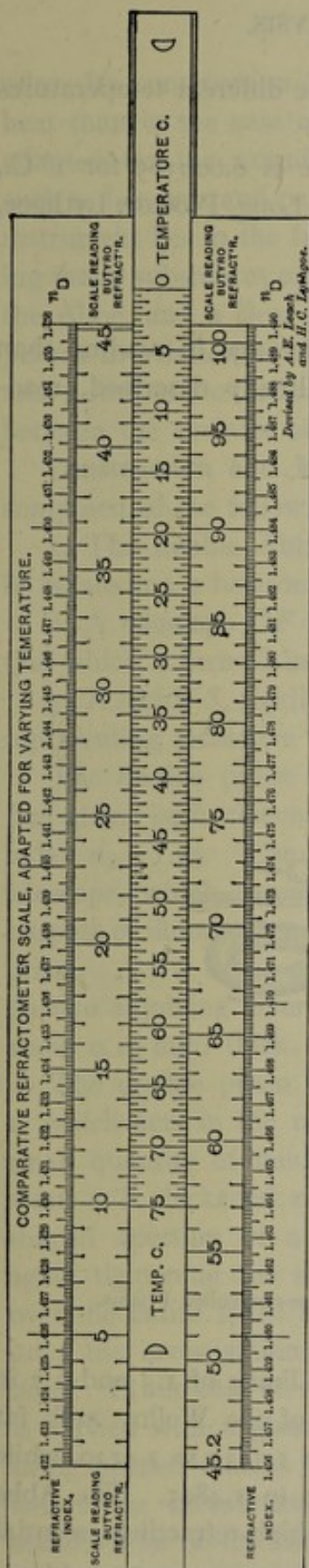


FIG. 40.—Comparative Refractometer Scale.

† Manufactured by Messrs. Baird and Tatlock, L., 14 Cross Street, Hatton Garden, London.

### Variation of Reading with the Temperature.—

No definite temperature has been adopted as a standard for readings of this instrument, but it is easy to reduce readings at any temperature to terms of any other temperature for purposes of comparison. While the change in index of refraction for  $1^{\circ}$  C. is the same whatever the temperature, as Tolman and Munson have pointed out,\* the change in scale reading per  $1^{\circ}$  C. decreases as the temperature increases, and varies slightly with different oils. For correcting reading  $R'$  at a temperature  $T'$  to a reading  $R$  at temperature  $T$ , their formula is  $R = R' - X(T - T')$ ,  $X$  being the change in scale reading due to change of  $1^{\circ}$  C. in temperature.

For butter, oleomargarine, beef tallow, lard, and other fats reading from  $40^{\circ}$  to  $50^{\circ}$  or thereabouts on the scale,  $X = 0.55$ . For oils reading between  $60^{\circ}$  and  $70^{\circ}$ , like olive, mustard, rapeseed, cottonseed, peanut, etc.,  $X = 0.58$ , and for oils reading between  $70^{\circ}$  and  $80^{\circ}$ , like corn oil,  $X = 0.62$ .

The slide rule † shown in Fig. 40, for use with the refractometer, has been jointly devised by H. C. Lythgoe and the writer, to render unnecessary the use of tables or formulas. The extreme upper and lower scale divisions indicate indices of refraction, and adjacent to these are the scale divisions indicating readings on the butyro-refractometer. By comparison, therefore, the values of either the Abbé or the butyro scale may be readily ascertained in terms of the other.

The sliding scale, expressing temperature readings in degrees centigrade, is intended to be used in connection with the adjacent scale of butyro-refractometer readings, to readily express the butyro-scale reading of any fat or oil taken at a given temperature, in terms of that at any other temperature. This is frequently convenient

\* Jour. Am. Chem. Soc., XXIV, p. 755.

† Manufactured by Messrs. Baird and Tatlock, L., 14 Cross Street, Hatton Garden, London.



in comparing the work of various observers, where different temperatures have been employed.

The correction for change in  $n_D$  on the scale is 0.000365 for  $1^\circ \text{C.}$ , being based on the experimental work of Tolman, Long, Proctor, Lythgoe, and the author.

#### THE ABBÉ REFRACTOMETER.

This instrument, Fig. 41, has a much wider range in reading than either the butyro or the Wollny instruments already described, read-

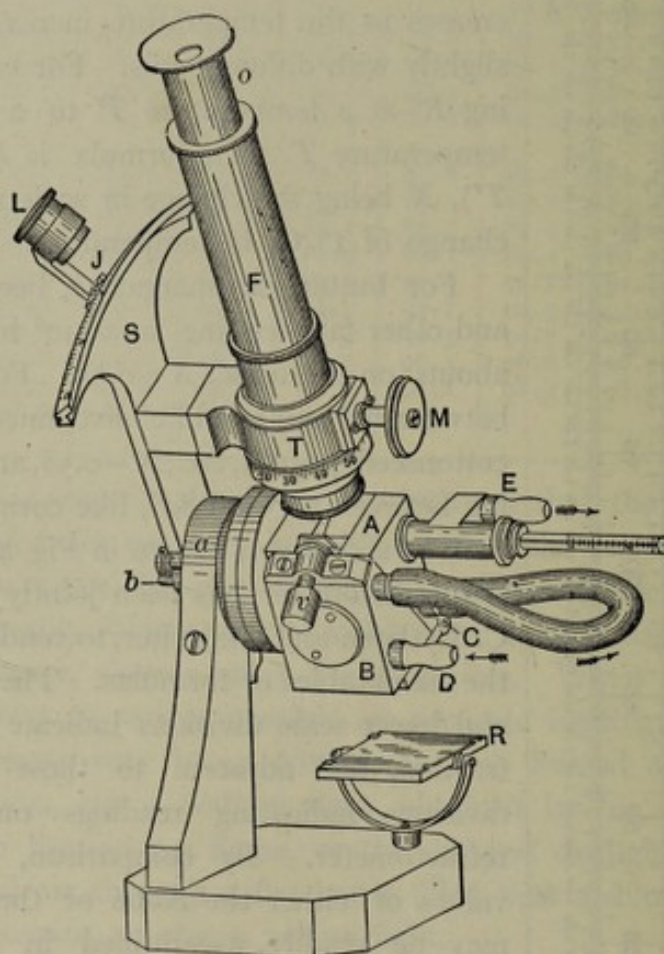


FIG. 41.—The Abbé Refractometer with Temperature-controlled Prisms.

ing as it does to the fourth decimal between the limits of 1.3 and 1.7 in indices of refraction. The equivalent readings of the Wollny milk fat refractometer, in indices of refraction, range from 1.3332 to 1.4220, while those of the butyro instrument run from 1.4220 to 1.4895. The Abbé instrument is thus necessary for use with the high-refracting essential



oils. Its construction is such that the prisms can withstand a higher heat than in the case of the butyro-refractometer, and it is hence better adapted for the examination of samples having a high melting-point, such as beeswax and paraffin. An advantage of the Abbé over the butyro instrument lies in the fact that the wide dispersion, inevitable when reading many substances on the butyro, may be entirely compensated for with the Abbé, and a clear sharp line be obtained. The construction of the prisms in relation to the heating jacket is similar in both instruments, and a film of the substance to be examined is held in the same manner between the surfaces of the prisms.

**Construction and Manipulation.**—The Abbé refractometer is mainly composed of the following parts (see Fig. 41):

1. The double Abbé prism  $AB$ , which contains the fluid and can be rotated on a horizontal axis by means of an alidade.
2. A telescope  $OF$  for observing the border-line of the total reflection which is formed in the prism.
3. A sector  $S$ , rigidly connected with the telescope, on which divisions representing refractive indices are engraved.

The double prism ( $AB$ , Fig. 41) consists of two similar prisms of flint-glass, each cemented into a metal mount and having a refractive index  $n_D = 1.75$ . The former of the two prisms, that farthest from the telescope, which can be folded up or removed, serves solely for the purpose of illumination, while the border-line is formed in the second flint prism. A few drops of the fluid to be investigated is deposited between the two adjoining inner faces of the prisms in the form of a thin stratum, about 0.15 mm. thick.

The double prism is opened and closed by means of a screw-head  $v$ , which acts in the manner of a bayonet catch. In order to apply a small quantity of fluid to the prisms without opening the casing, the screw  $v$  is slackened and a few drops of fluid poured into the funnel-shaped aperture of a narrow passage, not seen in the figure. On again tightening the screw, the fluid is distributed by capillary action over the entire space between the two prisms. This arrangement facilitates the investigation of rapidly evaporating fluids, such as ether solutions. In the case of viscous fluids (resins, etc.), a drop of moderate size is applied with a glass rod to the dull prism surface, the double prism being opened for the purpose. The prisms are then closed again, and before the measurement is proceeded with, the refractometer is left standing for a few minutes in order to compensate for any cooling or heating of the prisms which might occur while they were separated.



The arrangement for controlling the temperature of the prisms of the Abbé refractometer is essentially after Dr. R. Wollny's plan of enclosing the prisms in a metal casing with double walls, through which water of a given temperature is circulated.

The border-line is brought within the field of the telescope  $OF$  by rotating the double prism by means of the alidade in the following manner: Holding the sector, the alidade is moved from the initial position at which the index points to  $n_D = 1.3$ , in the ascending scale of the refractive indices until the originally entirely illuminated field of view is encroached upon from the direction of its lower half by a dark portion; the line dividing the bright and the dark half of the field then is the "border-line." When daylight or lamplight is being employed, the border-line, owing to the total reflection and the refraction caused by the second prism, assumes at first the appearance of a band of color, which is quite unsuitable for any exact process of adjustment. The conversion of this band of color into a colorless line sharply dividing the bright and dark portions of the field is the work of the compensator, which consists of two similar Amici prisms of direct vision for the  $D$ -line, and rotated simultaneously, though in opposite directions, round the axis of the telescope by means of the screw-head  $M$ . The dispersion of the border-line, which appears in the telescope as a band of color, can thus be counteracted by rotating the screw-head  $M$  till the equal but opposite dispersions are neutralized, making the line colorless and sharp.

The border-line is now adjusted upon the point of intersection of the crossed lines by slightly inclining the double prism to the telescope by means of the alidade. The position of the pointer on the graduation of the sector is then read by the aid of the magnifier attached to the alidade. The reading supplies the refractive index  $n_D$  of the substance under investigation without any computation, and with a degree of exactness approaching to within about two units of the fourth decimal. Simultaneously, the reading of the scale on the drum of the compensator ( $T$  in Fig. 41) enables the mean dispersion to be arrived at by means of a special table and a short process of computation.

*Influence of Temperature.*—As the refractive index of fluids varies with their temperature, it is of importance to know the temperature of the fluid contained in the double prism during the process of measurement.

If, therefore, it is desired to measure a fluid with the highest degree of accuracy attainable with the Abbé refractometer (to within one or



two units of the fourth decimal of  $n_D$ ), it is absolutely necessary to bring the fluid, or rather the double prism containing it, to a definite known temperature, and to be able to control this temperature so as to keep it constant to within some tenths of a degree for a period of several hours; hence a refractometer principally required for the investigation of fluids must be provided with heatable prisms.

The type of heater shown in Fig. 39. and described in connection with the butyro-refractometer on page 88, is equally adapted for controlling the temperature of the prisms in the Abbé instrument, the flow of water entering at *D* and passing out at *E*, Fig. 41.

#### THE IMMERSION REFRACTOMETER.

This form of refractometer is of more recent introduction than the others made by Zeiss, and has many features that especially commend it to the use of the food analyst. The construction of the immersion refractometer is such that, as its name implies, it may be immersed directly in an almost endless variety of solutions, the strength of which, within limits, may be determined by the degree of refraction read upon an arbitrary scale. Thus, for example, the strengths of various acids and of a variety of salt solutions used as reagents in the laboratory, as well as of formaldehyde, of sugars in solution, and of alcohol, are all capable of determination by the use of the immersion refractometer.

Figure 42 shows the form used by the writer. *P* is a glass prism fixed in the lower end of the tube of the instrument, while at the top of the tube is the ocular *Oc*, and just below this on a level with the vernier screw *Z* is the scale on which is read the degree of refraction of the liquid in which the prism *P* is immersed. The tube may be held in the hand and directly dipped in the liquid to be tested, this liquid being contained in a vessel with a translucent bottom, through which the light is reflected. The preferable method of use is, however, that shown in the cut.

*A* is a metal bath with inlet and outlet tubes, arranged whereby water is kept at a constant level. The water is maintained at a constant temperature by means of a controller of the same type as the refractometer heater shown in Fig. 39. In the bath *A* are immersed a number of beakers, containing the solutions to be tested. *T* is a frame on which is hung the refractometer by means of the hook *H*, at just the right height to permit of the immersion of the prism *P* in the liquid in any of the beakers in the row beneath. Under this row of beakers the bottom of the tank is composed of a strip of ground glass, through which light is reflected by an adjustable pivoted mirror.



The temperature of the bath is noted by a delicate thermometer immersed therein, capable of reading to tenths of a degree.

Returning to the main refractometer-tube, *R* is a graduated ring or collar which is connected by a sleeve within the tube with a compound prism near the bottom, the construction being such that by turning the collar *R* one way or the other the chromatic aberration or dispersion of any liquid may be compensated for, and a clear-cut shadow or critical line projected across the scale. By the graduation on the collar *R*, the degree of

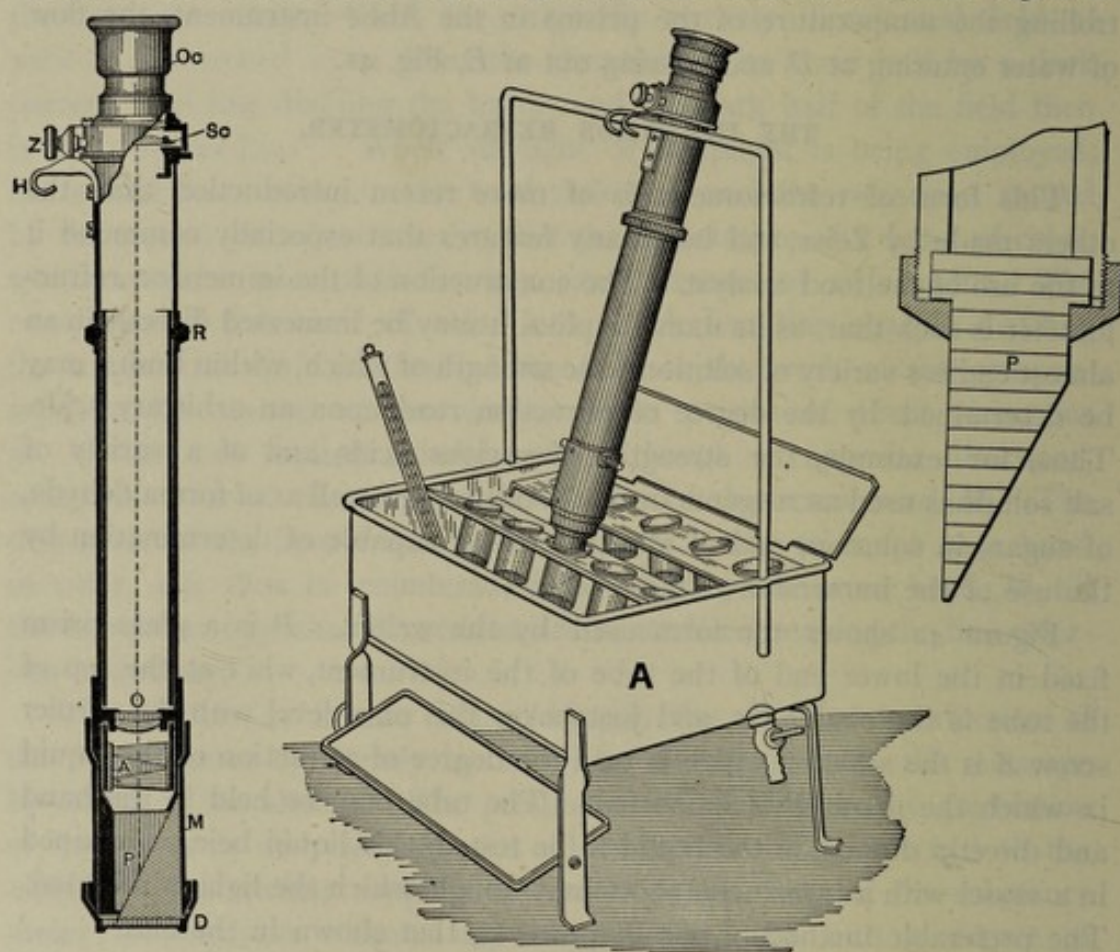


FIG. 42.—The Zeiss Immersion Refractometer.

dispersion may be read. Tenths of a degree on the main scale of the instrument may be read with great accuracy by means of the vernier screw *Z*, graduated along its circumference, the screw being turned in each case till the critical line on the scale coincides with the nearest whole number.

The scale of the instrument reads from  $-5$  to  $105$ , corresponding to indices of refraction of from  $1.32539$  to  $1.36640$ . It should be noted that the index of refraction may be read with a greater degree of accuracy on the immersion refractometer than on the Abbé instrument.



**Manipulation of the Immersion Refractometer.**—Before using the instrument for the first time, it is necessary to see that the refractometer is correctly adjusted. For this purpose the bath *A* is placed with its long side parallel to the window and the mirror turned towards a bright sky, the bath is half filled with tap-water, and a beaker filled with distilled water is then placed in one of the five holes in the front row immediately above the mirror. Finally, the refractometer is hung by its hook *H* upon the wire frame, the prism being totally submerged in the water contained in the beaker.

The whole apparatus is now allowed to stand for ten minutes, or until the distilled water has acquired the exact temperature of the bath, and the ocular is focussed upon the divisions of the scale by turning the milled zone of the ocular shell until the lines and numbers are seen quite distinctly, the mirror being adjusted so that the light of the bright sky is seen directly through the beaker. The upper part of the field from  $-5$  to about  $15$  appears bright, and it is separated from the lower dark part by a sharp line of demarcation, if the index on the ring of the compensator stands at  $5$ .

SCALE READING AND INDEX OF REFRACTION OF DISTILLED WATER  
AT  $10-30^{\circ}$  C., ACCORDING TO WAGNER.

Temperature C.	Scale Reading.	Index of Refraction, $n_D$ .	$n_D$ Difference for $1^{\circ}$ C.	Temperature C.	Scale Reading.	Index of Refraction, $n_D$ .	$n_D$ Difference for $1^{\circ}$ C.
30	11.8	1.33196	...	19	14.7	1.333075	8.5
29	12.1	1.33208	12.0	18	14.9	1.33316	8.5
28	12.4	1.332195	11.5	17.5	15.0	1.33320	4 } 8.0
27	12.7	1.33231	11.5	17	15.1	1.33324	4 } 8.0
26	13.0	1.33242	11.0	16	15.3	1.333315	7.5
25	13.25	1.332525	10.5	15	15.5	1.33339	7.5
24	13.5	1.332625	10.0	14	15.7	1.33346	7.0
23	13.75	1.33272	9.5	13	15.85	1.333525	6.5
22	14.0	1.33281	9.0	12	16.0	1.33359	6.5
21	14.25	1.33290	9.0	11	16.15	1.33365	6.0
20	14.5	1.33299	9.0	10	16.3	1.333705	5.5

The reading is taken and the temperature of the distilled water noted. Reference to the above table will show if the refractometer is correctly adjusted. Should the average of several careful readings at a given temperature deviate from that contained in the table, the following should be resorted to:

**Readjustment of the Scale.**—The ocular end of the refractometer hanging on the wire frame is grasped from behind with the thumb and forefinger of the left hand, the micrometer drum set to 10, and the steel



spike, housed in the case of the apparatus, inserted into one of the holes of the nicked cross-holed screw lying on the inner side of the micrometer drum. The spike is then turned anti-clockwise, as seen from the rear, whereupon the nicked milled nut, which governs the micrometer, becomes loosened. The temperature of the distilled water in the beaker is taken once more to see that it has remained constant, and then the table (page 99) is consulted to find the "adjusting number" properly belonging to the temperature indicated. By turning the spike, the borderline is brought exactly upon the integer scale division appertaining to the adjusting number, and the loose micrometer drum is turned so that the index accords with the decimal portion of the adjusting number. The drum is now held firmly with the thumb and forefinger of the left hand, while the nut is screwed up tight again by the right hand, taking care, however, that the drum does not wander off the index. Finally, the new adjustment is tested by repeated readings.

*Regulating the Temperature.*—In many cases it suffices to allow water at the temperature of the room to flow slowly from a tank suspended high upon the wall through the bath. Should it be required, however, to maintain a given temperature (say 20° C.) for hours together constant to a tenth of a degree, which is frequently desirable if not actually necessary, a more elaborate temperature-regulating device should be employed. In cold weather, or when the tap-water has a lower temperature than that desired, a refractometer heater of the type shown in Fig. 39, and described on page 88, is convenient.

When, as in the summer, the tap-water temperature is higher than that desired for the refractometer bath, there are various ways of successfully controlling the temperature at a lower degree. An ice-water tank placed above the level of the bath may be employed, the flow from which through the bath is carefully controlled by a pinch-cock or otherwise, or is allowed to mingle, under careful regulation before entering the bath, with the water from the tap direct or with that from the heater.

*Investigation of Solutions in Beakers in Bulk.*—The first ten solutions are poured into beakers until two-thirds full, and the latter are immersed and brought to the temperature of the bath *A*. When the first five solutions have been measured, they are taken out of the water-bath and the second series of five beakers inserted in their place, bringing at the same time a third series into the water-bath. The second series are measured and so on. Small gummed labels on the outside prove quite satisfactory for numbering the beakers. It is absolutely necessary to



compare the temperature of the solutions in the beakers with the water-bath from time to time.

After each immersion, the prism should be wiped dry with a clean, soft piece of old linen.

*Investigations of Solutions Excluded from Air.*—Quickly evaporating liquids, for instance ether solutions, should be treated individually by means of the metal beaker adapted to fit the prism end of the refractometer. To fill the beaker, the refractometer is held in the left hand with the prism pointing upwards, and the metal beaker (*M*, Fig. 42) is set and securely fastened by means of the bayonet joint. It is now filled quite full and the cover *D* carefully fitted and locked.

The solution is now enclosed, air and water tight. The refractometer as before is hung upon the wire frame of the bath, with the metal beaker submerged in the bath.

It is expedient to place the solutions before investigation in closed flasks in the nine unoccupied holes in the bath.

After the measurement, the refractometer is held in the left hand with the prism pointing downwards, and the beaker together with its cover detached by giving a slight turn with the right hand. The solution can be used for other purposes, since it has undergone no change in constitution. Finally, the cover is detached from the beaker, and cover, beaker, and prism cleaned by simple means, and the refractometer made ready for the reception of the next solution, as above.

*Investigations of Small Quantities of Solutions with the Auxiliary Prism.*—When the solution does not occur in sufficiently large quantities for investigation in the glass beaker, or when the solution is too deeply colored, as in dark beers, molasses, etc., the auxiliary prism is used. As described under "Solutions Excluded from Air," the metal beaker without cover is fitted on the refractometer. The auxiliary prism is held horizontally, and, a few drops of the solution having been applied to the hypotenuse face, the prism is inserted into the metal beaker, held conveniently for the purpose, with its hypotenuse face laid upon the polished elliptical face of the refractometer prism, and then locked in by the cover. If an insufficient quantity of the solution has been taken, the margins of the out-spread drops lying between the two prisms can be recognized by looking through the window of the cover on which abuts the square polished end of the auxiliary prism. It is strongly recommended, wherever possible, to apply a sufficiency of the solution, so that the space between the prisms is completely filled, otherwise a loss in brilliancy occurs, and, under certain circumstances, an unavoidable



TABLE OF INDICES OF REFRACTION,  $n_D$   
 (Compared with Scale Readings of Zeiss Immersion Refractometer, according to Wagner.)

Scale Read- ing.	$n_D$	Scale Read- ing.	$n_D$	Scale Read- ing.	$n_D$	Scale Read- ing.	$n_D$	Scale Read- ing.	$n_D$
0.0	1.327360	5.0	1.329320	10.0	1.331260	15.0	1.333200	20.0	1.335168
0.1	1.327399	5.1	1.329350	10.1	1.331299	15.1	1.333238	20.1	1.335168
2	438	2	398	2	388	2	276	2	206
3	477	3	437	3	377	3	314	3	244
4	516	4	476	4	416	4	352	4	282
5	555	5	515	5	455	5	390	5	320
6	594	6	554	6	494	6	428	6	358
7	633	7	593	7	533	7	466	7	396
8	672	8	632	8	572	8	504	8	434
9	711	9	671	9	611	9	542	9	472
1.0	750	6.0	710	11.0	650	16.0	580	21.0	510
1.1	1.327789	6.1	1.329749	11.1	1.331689	16.1	1.333619	21.1	1.335549
2	828	2	788	2	728	2	658	2	588
3	867	3	827	3	767	3	697	3	627
4	906	4	866	4	806	4	736	4	666
5	945	5	905	5	845	5	775	5	705
6	984	6	944	6	884	6	814	6	744
7	1.328023	7	982	7	922	7	853	7	783
8	062	8	1.330022	8	962	8	892	8	822
9	101	9	061	9	1.332001	9	931	9	861
2.0	140	7.0	100	12.0	040	17.0	970	22.0	900
2.1	1.328180	7.1	1.330139	12.1	1.332078	17.1	1.334008	22.1	1.335938
2	220	2	178	2	116	2	046	2	976
3	657	3	217	3	154	3	084	3	1.336014
4	300	4	256	4	192	4	122	4	052
5	340	5	295	5	230	5	160	5	090
6	380	6	334	6	268	6	198	6	128
7	420	7	373	7	304	7	236	7	166
8	460	8	412	8	344	8	274	8	204
9	500	9	451	9	382	9	312	9	242
3.0	540	8.0	490	13.0	420	18.0	350	23.0	280
3.1	1.328579	8.1	1.330528	13.1	1.332459	18.1	1.334389	23.1	1.336319
2	618	2	566	2	498	2	428	2	358
3	657	3	604	3	537	3	467	3	397
4	696	4	642	4	576	4	506	4	436
5	735	5	680	5	615	5	545	5	475
6	774	6	718	6	654	6	584	6	514
7	813	7	756	7	693	7	623	7	553
8	852	8	794	8	732	8	662	8	592
9	891	9	832	9	771	9	701	9	631
4.0	930	9.0	870	14.0	810	19.0	740	24.0	670
4.1	1.328969	9.1	1.330909	14.1	1.332849	19.1	1.334779	24.1	1.336708
2	1.329008	2	948	2	888	2	818	2	746
3	047	3	987	3	927	3	857	3	784
4	085	4	1.331026	4	966	4	896	4	822
5	125	5	104	5	1.333005	5	935	5	860
6	164	6	104	6	044	6	974	6	898
7	203	7	143	7	083	7	1.335013	7	936
8	242	8	182	8	122	8	052	8	974
9	281	9	221	9	161	9	091	9	1.337012
5.0	320	10.0	260	15.0	200	20.0	130	25.0	050



TABLE OF INDICES OF REFRACTION,  $n_D$ —(Continued).

Scale Reading.	$n_D$	Scale Reading.	$n_D$	Scale Reading.	$n_D$	Scale Reading.	$n_D$	Scale Reading.	$n_D$
25.0	1.337050	30.0	1.338960	35.0	1.340860	40.0	1.342750	45.0	1.344630
25.1	1.337088	30.1	1.338998	35.1	1.340898	40.1	1.342788	45.1	1.344667
2	126	2	1.339036	2	936	2	826	2	704
3	164	3	074	3	974	3	864	3	741
4	202	4	112	4	1.341012	4	902	4	778
5	240	5	150	5	050	5	940	5	818
6	278	6	188	6	088	6	978	6	852
7	316	7	226	7	126	7	1.343016	7	889
8	354	8	264	8	164	8	054	8	926
9	392	9	302	9	202	9	092	9	963
26.0	430	31.0	340	36.0	240	41.0	130	46.0	1.345000
26.1	1.337468	31.1	1.339378	36.1	1.341278	41.1	1.343167	46.1	1.345037
2	506	2	416	2	316	2	204	2	074
3	544	3	454	3	354	3	241	3	111
4	582	4	492	4	392	4	278	4	148
5	620	5	530	5	430	5	315	5	185
6	658	6	568	6	468	6	352	6	222
7	696	7	606	7	506	7	389	7	259
8	734	8	644	8	544	8	426	8	296
9	772	9	682	9	582	9	463	9	333
27.0	810	32.0	720	37.0	620	42.0	500	47.0	370
27.1	1.337849	32.1	1.339758	37.1	1.341657	42.1	1.343538	47.1	1.345408
2	888	2	796	2	694	2	576	2	446
3	927	3	834	3	731	3	614	3	484
4	966	4	872	4	768	4	652	4	522
5	1.338005	5	910	5	805	5	690	5	560
6	044	6	948	6	842	6	728	6	598
7	083	7	986	7	879	7	766	7	636
8	122	8	1.340024	8	916	8	804	8	674
9	161	9	062	9	953	9	842	9	712
28.0	200	33.0	100	38.0	990	43.0	880	48.0	750
28.1	1.338238	33.1	1.340138	38.1	1.342028	43.1	1.343918	48.1	1.345787
2	276	2	176	2	066	2	956	2	824
3	314	3	214	3	104	3	994	3	861
4	352	4	252	4	142	4	1.344032	4	898
5	390	5	290	5	180	5	070	5	935
6	428	6	328	6	218	6	108	6	972
7	466	7	366	7	256	7	146	7	1.346009
8	504	8	404	8	294	8	184	8	046
9	542	9	442	9	332	9	222	9	083
29.0	580	34.0	480	39.0	370	44.0	260	49.0	120
29.1	1.338618	34.1	1.340518	39.1	1.342408	44.1	1.344297	49.1	1.346158
2	656	2	556	2	446	2	334	2	196
3	694	3	594	3	484	3	371	3	234
4	732	4	632	4	522	4	408	4	272
5	770	5	670	5	560	5	445	5	310
6	808	6	708	6	598	6	482	6	348
7	846	7	746	7	636	7	519	7	386
8	884	8	784	8	674	8	556	8	424
9	922	9	822	9	712	9	593	9	462
30.0	960	35.0	860	40.0	750	45.0	630	50.0	500



TABLE OF INDICES OF REFRACTION,  $n_D$ —(Continued).

Scale Reading.	$n_D$ .	Scale Reading.	$n_D$ .	Scale Reading.	$n_D$ .	Scale Reading.	$n_D$ .	Scale Reading.	$n_D$ .
50.0	1.346500	55.0	1.348360	60.0	1.350210	65.0	1.352050	70.0	1.353880
50.1	1.346537	55.1	1.348397	60.1	1.350247	65.1	1.352087	70.1	1.353917
2	574	2	434	2	284	2	124	2	954
3	611	3	471	3	321	3	161	3	991
4	648	4	508	4	358	4	198	4	1.354028
5	685	5	545	5	395	5	235	5	065
6	722	6	582	6	432	6	272	6	102
7	759	7	619	7	469	7	309	7	139
8	796	8	656	8	506	8	346	8	176
9	833	9	693	9	543	9	383	9	213
51.0	870	56.0	730	61.0	580	66.0	420	71.0	250
51.1	1.346907	56.1	1.348767	61.1	1.350617	66.1	1.352457	71.1	1.354286
2	944	2	804	2	654	2	494	2	322
3	981	3	841	3	691	3	531	3	358
4	1.347018	4	878	4	728	4	568	4	394
5	055	5	915	5	765	5	605	5	430
6	092	6	952	6	802	6	642	6	466
7	129	7	989	7	839	7	679	7	502
8	166	8	1.349026	8	876	8	716	8	538
9	203	9	063	9	913	9	753	9	574
52.0	240	57.0	100	62.0	950	67.0	790	72.0	610
52.1	1.347277	57.1	1.349137	62.1	1.350987	67.1	1.352827	72.1	1.354646
2	314	2	174	2	1.351024	2	864	2	682
3	351	3	211	3	061	3	901	3	718
4	388	4	248	4	098	4	938	4	754
5	425	5	285	5	135	5	975	5	790
6	462	6	312	6	172	6	1.353012	6	826
7	499	7	359	7	209	7	049	7	862
8	536	8	396	8	246	8	086	8	898
9	573	9	433	9	283	9	123	9	934
53.0	610	58.0	470	63.0	320	68.0	160	73.0	970
53.1	1.347647	58.1	1.349507	63.1	1.351357	68.1	1.353196	73.1	1.355006
2	684	2	544	2	394	2	232	2	042
3	721	3	581	3	431	3	268	3	078
4	758	4	618	4	468	4	304	4	114
5	795	5	655	5	505	5	340	5	150
6	832	6	692	6	542	6	376	6	186
7	869	7	729	7	579	7	412	7	222
8	906	8	766	8	616	8	448	8	258
9	943	9	803	9	653	9	484	9	294
54.0	980	59.0	840	64.0	690	69.0	520	74.0	330
54.1	1.348018	59.1	1.349877	64.1	1.351726	69.1	1.353556	74.1	1.355366
2	056	2	914	2	762	2	592	2	402
3	094	3	951	3	798	3	628	3	438
4	132	4	988	4	834	4	664	4	474
5	170	5	1.350025	5	870	5	700	5	510
6	208	6	062	6	906	6	736	6	546
7	246	7	099	7	942	7	772	7	582
8	284	8	136	8	978	8	808	8	618
9	322	9	173	9	1.352014	9	844	9	659
55.0	360	60.0	210	65.0	050	70.0	880	75.0	690



TABLE OF INDICES OF REFRACTION,  $n_D$ —(Continued).

Scale Read- ing.	$n_D$ .	Scale Read- ing.	$n_D$ .	Scale Read- ing.	$n_D$ .	Scale Read- ing.	$n_D$ .	Scale Read- ing.	$n_D$ .
75.0	1.355690	80.0	1.357500	85.0	1.359300	90.0	1.361090	95.0	1.362870
75.1	1.355727	80.1	1.357536	85.1	1.359336	90.1	1.361126	95.1	1.362906
2	764	2	572	2	372	2	162	2	942
3	801	3	608	3	408	3	198	3	978
4	838	4	644	4	444	4	234	4	1.363014
5	875	5	680	5	480	5	270	5	050
6	912	6	716	6	516	6	306	6	086
7	949	7	752	7	552	7	342	7	122
8	986	8	788	8	588	8	378	8	158
9	1.356023	9	824	9	624	9	414	9	194
76.0	060	81.0	860	86.0	660	91.0	450	96.0	230
76.1	1.356096	81.1	1.357896	86.1	1.359696	91.1	1.361486	96.1	1.363256
2	132	2	932	2	732	2	522	2	292
3	168	3	968	3	768	3	558	3	328
4	204	4	1.358004	4	804	4	594	4	364
5	240	5	040	5	840	5	630	5	400
6	276	6	076	6	876	6	666	6	436
7	312	7	112	7	912	7	702	7	472
8	348	8	148	8	948	8	738	8	518
9	384	9	184	9	984	9	774	9	554
77.0	420	82.0	220	87.0	1.360020	92.0	810	97.0	590
77.1	1.356456	82.1	1.358256	87.1	1.360056	92.1	1.361846	97.1	1.363625
2	492	2	292	2	092	2	882	2	660
3	528	3	328	3	128	3	918	3	695
4	564	4	364	4	164	4	954	4	730
5	600	5	400	5	200	5	990	5	765
6	636	6	436	6	236	6	1.362026	6	800
7	672	7	472	7	272	7	062	7	835
8	708	8	508	8	308	8	098	8	870
9	744	9	544	9	344	9	134	9	905
78.0	780	83.0	580	88.0	380	93.0	170	98.0	940
78.1	1.356816	83.1	1.358616	88.1	1.360416	93.1	1.362205	98.1	1.363975
2	852	2	652	2	452	2	240	2	1.364010
3	888	3	688	3	488	3	275	3	045
4	924	4	724	4	524	4	310	4	080
5	960	5	760	5	560	5	345	5	115
6	996	6	796	6	596	6	380	6	160
7	1.357032	7	832	7	632	7	415	7	195
8	068	8	868	8	668	8	450	8	230
9	104	9	904	9	704	9	485	9	265
79.0	140	84.0	940	89.0	740	94.0	520	99.0	290
79.1	1.357176	84.1	1.358976	89.1	1.360775	94.1	1.362555	99.1	1.364325
2	212	2	1.359012	2	810	2	590	2	360
3	248	3	048	3	845	3	625	3	395
4	284	4	084	4	880	4	660	4	430
5	320	5	120	5	915	5	695	5	465
6	356	6	156	6	950	6	730	6	500
7	392	7	192	7	985	7	765	7	535
8	428	8	228	8	1.361020	8	800	8	570
9	464	9	264	9	055	9	835	9	605
80.0	500	85.0	300	90.0	090	95.0	870	100.0	640



degradation of the sharpness of the border-line. On the other hand, with a sufficient quantity of solution, the border-line is surprisingly sharp.

The refractometer is now suspended on the frame, and the measurement proceeded with as before described. After measurement, the cover is first removed, and the prism allowed to fall into the hollow of the hand, then the beaker is removed to enable the refractometer to be conveniently cleaned.

**Strengths of Various Solutions.**—The most extensive work on the quantitative determination of the strength of a large number of common aqueous solutions with the immersion refractometer has been done by Wagner, who has published a large number of tables. These tables show the percentage strength (grams per 100 cc. at 17.5° C.) of a large number of salt solutions and of acids, corresponding to the range of scale readings of the instrument, as well as of cane sugar, dextrose, formalde-

SCALE READINGS ON IMMERSION REFRACTOMETER OF VARIOUS STANDARD REAGENTS USED IN VOLUMETRIC ANALYSIS.\*

	Temperature C.								
	15°.	16°.	17°.	17.5°.	18°.	19°.	20°.	21°.	22°.
Hydrochloric acid:									
Normal. ....	37.45	37.20	36.95	36.85	36.70	36.45	36.20	35.95	35.70
Tenth-normal. ....	17.80	17.60	17.40	17.30	17.20	17.00	16.80	16.55	16.30
Sulphuric acid:									
Normal. ....	30.60	30.40	30.20	30.10	29.95	29.75	29.50	29.25	29.00
Fifth-normal. ....	18.75	18.60	18.40	18.30	18.20	18.00	17.80	17.55	17.30
Tenth-normal. ....	17.15	16.95	16.75	16.65	16.55	16.35	16.15	15.90	15.65
Oxalic acid:									
Half-normal. ....	22.45	22.30	22.10	22.00	21.90	21.70	21.50	21.25	21.00
Tenth-normal. ....	17.15	16.95	16.75	16.65	16.55	16.35	16.15	15.90	15.65
Potassium bitartrate:									
Tenth-normal. ....	17.75	17.55	17.35	17.25	17.15	16.95	16.75	16.50	16.25
Potassium hydroxide:									
Normal. ....	43.90	43.65	43.40	43.25	43.10	42.80	42.50	42.20	41.95
Tenth-normal. ....	18.45	18.30	18.10	18.00	17.90	17.70	17.50	17.25	17.00
Sodium hydroxide:									
Tenth-normal. ....	18.50	18.35	18.15	18.05	17.95	17.75	17.55	17.30	17.05
Sodium thiosulphate:									
Tenth-normal. ....	24.20	24.05	23.85	23.75	23.65	23.45	23.20	22.95	22.70
Potassium bichromate:									
Tenth-normal. ....	17.75	17.55	17.35	17.25	17.15	16.95	16.75	16.50	16.25
Silver nitrate:									
Tenth-normal. ....	20.20	20.05	19.85	19.75	19.65	19.45	19.25	19.00	18.75
Sodium chloride:									
Tenth-normal. ....	18.20	18.00	17.80	17.70	17.60	17.40	17.20	16.95	16.70
Ammonium sulphocyanate:									
Tenth-normal. ....	20.60	20.45	20.25	20.15	20.05	19.85	19.65	19.40	19.15

\* According to Wagner, all these solutions were made up at 17.5° C. Readings at different temperatures are given for convenience.



hyde, alcohol, etc. All these observations have been based on the Mohr liter, at a temperature of  $17.5^{\circ}$ . More convenient for the American analyst would be tables based on the use of a higher temperature, say  $20^{\circ}$ , and the analyst is recommended to work out his own standards for comparison, at the temperature best suited to his special locality and convenience. The instrument is especially useful in preparing normal and tenth-normal solutions.

The table on page 106, from Wagner, shows the strength of various common laboratory reagents.

SCALE READINGS AT TEMPERATURES FROM  $10-30^{\circ}$  C.  
CORRECTED TO  $17.5^{\circ}$ , ACCORDING TO WAGNER.

No.	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.	12 & 13.	No.
Tempera- ture C.	Scale Reading at $17.5^{\circ}$ C.												Tempera- ture C.
	15.	20.	25.	30.	35.	40.	45.	50.	60.	70.	80.	90 & 100.	
30	3.20	3.15	3.25	3.40	3.55	3.65	3.90	4.05	4.20	4.60	4.80	5.25	30
29	2.90	2.85	2.95	3.10	3.25	3.35	3.55	3.75	3.90	4.25	4.45	4.85	29
28	2.60	2.55	2.65	2.80	2.95	3.05	3.25	3.45	3.60	3.90	4.10	4.50	28
27	2.30	2.25	2.35	2.50	2.65	2.75	2.95	3.15	3.30	3.50	3.75	4.10	27
26	1.00	1.95	2.05	2.20	2.35	2.45	2.55	2.80	2.95	3.10	3.30	3.65	26
25	1.75	1.75	1.80	1.90	2.05	2.15	2.25	2.45	2.60	2.70	2.95	3.20	25
24	1.50	1.45	1.55	1.60	1.75	1.85	1.95	2.10	2.25	2.35	2.55	2.75	24
23	1.25	1.25	1.30	1.35	1.45	1.55	1.65	1.75	1.90	2.00	2.15	2.35	23
22	1.00	1.00	1.05	1.10	1.15	1.25	1.30	1.40	1.55	1.65	1.75	1.90	22
21	0.75	0.75	0.80	0.85	0.90	0.95	1.05	1.05	1.20	1.25	1.35	1.45	21
20	0.50	0.50	0.55	0.60	0.65	0.65	0.75	0.75	0.85	0.90	0.95	1.05	20
19	0.30	0.30	0.30	0.35	0.40	0.40	0.45	0.45	0.45	0.55	0.55	0.60	19
18	0.10	0.10	0.10	0.15	0.15	0.15	0.15	0.15	0.15	0.20	0.20	0.20	18
17.5	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	17.5
17	-0.10	0.10	0.10	0.10	0.10	0.10	0.15	0.15	0.15	0.15	0.20	0.20	17
16	0.30	0.30	0.30	0.30	0.35	0.35	0.40	0.45	0.45	0.50	0.55	0.55	16
15	0.50	0.45	0.45	0.50	0.60	0.60	0.65	0.75	0.75	0.80	0.85	0.90	15
14	0.70	0.60	0.60	0.70	0.80	0.85	0.90	0.95	1.05	1.10	1.25	1.25	14
13	0.85	0.75	0.75	0.85	1.00	1.10	1.15	1.20	1.35	1.40	1.55	1.60	13
12	1.00	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	12
11	1.15	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	11
10	1.25	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
No.	1.	2.	3.	4.	5.	6.	7.	8.	9.	10.	11.	2 & 13.	No.



## CHAPTER VII.

### MILK AND ITS PRODUCTS.

#### MILK.

**CHARACTERS AND CONSTITUENTS.**—Milk is the secretion of the mammary glands of female mammals for the nourishment of their young. Containing as it does all the requisites for a complete food, i.e., sugar, fat, proteins, and mineral ingredients, combined in appropriate proportion, there is ample reason why it occupies so high a place in the scale of human foods. It is a yellowish-white opaque fluid, denser than water, containing in complete solution the sugar, soluble albumin, and mineral content, and, in less complete solution, the casein, while the fat-globules are held in suspension in the serum, forming an emulsion.

The specific gravity of pure milk ranges from 1.027 to 1.035.

Milk from various animals has the same general physical properties and the same ingredients, differing, however, in percentage composition. Of all the varieties, the milk of the cow is by far the most important from its universal use, and, unless otherwise qualified, the term milk wherever it occurs in this volume will be understood to mean cow's milk.

**Acidity.**—When perfectly fresh, milk of carnivorous mammals is, as a rule, acid to litmus, while human milk is alkaline. Cow's milk, when freshly drawn, reacts acid to phenolphthalein, and alkaline to methyl orange and lacmoid, but amphoteric to litmus, i.e., reacts acid to blue and alkaline to red litmus paper. Bordas and Touplain\* believe the original acidity to phenolphthalein is due to casein; as a result of the decomposition of lactose, before lactic acid appears, casein is separated from calcium caseinate and monocalcium phosphate is formed from the dicalcium salt. It soon becomes distinctly acid, and the acidity increases as the lactose gradually becomes converted into lactic acid.

**Microscopical Appearance.**—Under the microscope pure milk shows a conglomeration of various-sized fat globules having a pearly lustre.

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\* Compt. rend., 152, 1911, p. 1274.



These globules vary from 0.001 to 0.01 mm. in diameter, averaging about 0.005 mm., although the size varies greatly with the breed of cow (p. 115), period of lactation, and other conditions. Whether or not the globules have a special membranous coating is still in dispute.

Leucocytes, identical with or related to the white corpuscles of the blood, are present in all milk,\* but according to Doane are indicative of udder diseases when in masses. Some authors believe that the leucocyte count is of diagnostic value.

When examined under very high powers, it is possible to distinguish bacteria in the milk, the number to be seen depending greatly on the time that has elapsed since the milk was drawn from its source, as well as on the surroundings, the conditions of handling, exposure, etc.

Certain bacteria are present in the milk before it is drawn from the udder, but these are few in number and kind compared with those introduced during or after milking.

**Color.**—The yellow color of milk is imparted to it by the fat globules, and varies greatly in milk from different breeds of cattle, as well as in milk from the same cow at different seasons, being, as a rule, paler during the winter or stall-fed months, and having its greatest intensity soon after the cow is put out to pasture. The nature of the color is considered in the section on butter (Chapter XIII).

**Fat.**—This is the most variable constituent of milk, being present in amounts ranging from less than 2.5 to nearly 7%. For the chemical composition and characteristics of milk fat, see Butter.

**Lactose or Milk Sugar,** the carbohydrate of milk, is normally present in amounts varying from 3 to 5 per cent.

**Proteins of Milk.**—*Casein*, a phosphoprotein, constitutes about 80% of the entire proteins of milk, being present in an average sample to the extent of about 3%. It exists in combination as calcium caseinate, and probably does not form a perfect solution in the milk, but is rather diffused therein in a somewhat colloidal form, being so finely divided, however, as to be incapable of separation by filtration while the milk is fresh. On coagulation with rennet paracasein is formed.

Pure casein is a white, odorless, and tasteless solid, sparingly soluble in water, and insoluble in ether and alcohol. It is readily soluble in dilute alkalies. Strong acids also dissolve it, but its character is changed. From

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\* Doane, Md. Agric. Exp. Sta., Bul. 102, 1905, p. 205; Savage, Jour. Hyg. (Cambridge), 6, 1906, p. 173.



alkaline solution it is precipitated without change by neutralizing with acid. Its specific rotation in aqueous solutions is  $[\alpha]_D = -80$  (Hoppe-Seyler).

*Lactalbumin* is the soluble albumin of milk, existing therein to the extent of about 0.6% and forming about 15% or more of the milk proteins. It much resembles the albumin of eggs, being coagulated at 72° to 84° C. It is readily soluble in water. Its specific rotation is  $[\alpha]_D = -37$ .

*Lactoglobulin* has been discovered by Emmerling as a constituent in milk, but exists in traces only. According to Babcock, it may be separated from milk whey by carefully neutralizing with sodium hydroxide, and afterwards saturating with magnesium sulphate. It much resembles the globulin of blood serum, being coagulated at 67° to 76° C.

*Fibrin*.—Babcock has discovered in milk very minute traces of a substance analogous to the fibrin of blood. This substance, it is claimed, forms a part of the slime found in the separator-bowl of a centrifugal skimmer.

*Other Nitrogenous Substances*.—Besides the above normal constituents of milk, certain bodies may be formed by proteolytic action during fermentation, such, for example, as caseoses and peptones, formed for the most part by the decomposition of a part of the casein. Galactin is a gelatin-like body of the nature of peptone, occurring in traces in milk. Besides these, minute traces of amino-bodies, such as creatin, urea, and allantoin, are sometimes present, and also ammonia.

*Citric Acid* has been found to exist in milk, probably in combination with certain of the mineral constituents, being present to the extent of about 0.1%.

*Other Organic Constituents* reported in milk are lecithin, cephalin; cholesterol, acetone, and thiocyanates.

*The Enzymes of Milk* have been subjects of extensive investigation. The presence or amount of four of these, namely, peroxidase, reductase, aldehyde reductase, and catalase, are of considerable importance in sanitary milk examination.

*Peroxidase* originates in the mammary glands. Since it is destroyed at about 80° C., it is the basis of various tests for distinguishing raw from heated milk. Peroxidase acts in the presence of hydrogen peroxide, from which it splits off an atom of active oxygen, the latter combining with various chromogens (page 173).

*Reductase* is indicated by the decolorizing power of milk for certain dyes. Since reductase is believed to be a product of bacteria, the tests



TABLE SHOWING THE COMPOSITION OF MILK.\*

Milk. .... 100.0	Butter fat ..... 3.6	Olein. ....	Glycerides of insoluble and non-volatile acids. .... 3.3	Fat..... 3.6	Total solids. ... 12.7
		Palmitin.....			
		Stearin. ....			
		Myristin.....			
		Butin (trace).....			
		Butyrin. ....			
		Caproin.....			
		Caprylin (trace).....			
		Caprinin (trace).....			
		Casein. .... 3.00			
		Albumin. .... 0.60			
		Lactoglobulin			
		Galactin			
		Fibrin (trace)			
		Milk sugar ..... 4.5			
		Citric acid. .... 0.1			
		Potassium oxide. .... 0.175			
		Sodium oxide. .... 0.070			
		Calcium oxide..... 0.147			
		Magnesium oxide..... 0.017			
		Iron oxide..... 0.001†			
		Sulphur trioxide..... 0.027			
		Phosphoric pentoxide. .... 0.170			
		Chlorine..... 0.100			
		Ash..... 0.7			
		Water.....			
					87.3
					100.0

\* According to S. M. Babcock.

† Other authors find much smaller amounts.



are used for detecting pollution as an adjunct to bacteriological examination.

*Aldehyde Reductase* is a term applied to the enzyme which decolorizes methylene blue in the presence of formaldehyde, the latter inhibiting, it is believed, the action of bacteria so that the decolorizing action is due largely to the enzyme. The test furnishes information as to whether or not milk has been properly pasteurized at 63° C. (page 174).

*Catalase* splits up two molecules of hydrogen peroxide into two molecules of water and one of oxygen. The latter may be measured or otherwise determined. The enzyme has been shown to be derived from leucocytes and the test is therefore useful in detecting milk from diseased udders (mastitis, etc.).

**Average Composition of Milk.**—On page 111 is given in schematic form the average percentages of the principal constituents as arranged by Babcock.

The following table shows the forms in which, according to Van Slyke and Bosworth,\* the constituents are probably combined:

Fat.....	3.90
Lactose .....	4.90
Proteins combined with calcium.....	3.20
Dicalcium phosphate ( $\text{CaHPO}_4$ ).....	0.175
Calcium chloride ( $\text{CaCl}_2$ ).....	0.119
Monomagnesium phosphate ( $\text{MgH}_4\text{P}_2\text{O}_8$ ) .....	0.103
Sodium citrate ( $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$ ).....	0.222
Potassium citrate ( $\text{K}_3\text{C}_6\text{H}_5\text{O}_7$ ) .....	0.052
Dipotassium phosphate ( $\text{K}_2\text{HPO}_4$ ).....	0.230
Total solids.....	12.901

**Composition of the Ash of Milk.**—The ash of milk does not truly represent the mineral content, since, in the process of incineration, the character of some of the constituents is altered by oxidation and otherwise.

The composition of the ash of the typical milk sample, the full analysis of which is given on page 111, would be about as follows:

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\* N. Y. State Agric. Exp. Sta., Tech. Bul. 39, 1914.



Potassium oxide.....	25.02
Sodium oxide.....	10.01
Calcium oxide.....	20.01
Magnesium oxide.....	2.42
Iron oxide.....	0.13
Sulphur trioxide.....	3.84
Phosphoric pentoxide.....	24.29
Chlorine.....	14.28
	<hr/>
	100.00

**Composition of Milk of Different Animals.**—A summary of analyses of human, goat's, ewe's, mare's, and ass's milk, as well as of cow's milk for comparison, is given in the following table, the figures for human milk being compiled by Richmond,\* those for the milk of other animals by König:

	No. of Anal-yses.	Specific Gravity.	Water.	Fat.	Lactose	Total Pro-tein.	Casein.	Albu-min.	Ash.	Fuel Value per Lb., Calories
Cow's milk....	800									
Maximum....	....	1.0370	90.32	6.47	6.12	6.40	6.29	1.44	1.21	
Minimum....	....	1.0264	80.32	1.67	2.11	2.07	1.79	0.25	0.35	
Average.....	....	1.0315	87.27	3.64	4.88	3.55	3.02	0.53	0.71	310
Human milk....	94									
Maximum....	....	1.0426	.....	9.05	8.89	5.56	....	....	0.50	
Minimum....	....	1.0240	.....	0.47	4.22	0.85	....	....	0.09	
Average.....	....	1.0313	88.20	3.30	6.80	1.50	....	....	0.20	295
Goat's milk....	200									
Maximum....	....	1.0360	90.16	7.55	5.77	....	3.94	2.01	1.06	
Minimum....	....	1.0280	82.02	3.10	3.26	....	2.44	0.78	0.39	
Average.....	....	1.0305	85.71	4.78	4.46	4.29	3.20	1.09	0.76	364
Ewe's milk....	32									
Maximum....	....	1.0385	87.02	9.80	7.95	....	5.69	1.77	1.72	
Minimum....	....	1.0298	74.47	2.81	2.76	....	3.59	0.83	0.13	
Average.....	....	1.0341	80.82	6.86	4.91	6.52	4.97	1.55	0.89	502
Mare's milk....	47									
Average.....	....	1.0347	90.78	1.21	5.67	1.99	1.24	0.75	0.35	194
Ass's milk.....	5									
Average.....	....	1.0364	89.64	1.64	5.99	2.22	0.67	1.55	0.51	222

**Fore Milk and Strippings.**—Unless a portion drawn from the well-mixed or whole complete milking of an animal is taken for analysis, one

\* Dairy Chemistry, London, 1914, p. 394.



does not get a fair representative sample of the milk, for it is a well-known fact that the first portion of milk drawn from the udder, termed the "fore milk," is very low in fat, while the last portions or "strippings" contain a very high fat content, sometimes exceeding 10% fat. The following analyses show the difference between fore milk and strippings in two cases:

	Per Cent Water.	Per Cent Solids.	Per Cent Fat.
(1) Fore milk.....	88.17	11.83	1.32
Strippings.....	80.82	19.18	9.63
(2) Fore milk.....	88.73	11.27	1.07
Strippings.....	80.37	19.63	10.36

The percentages of protein, lactose, and ash are nearly the same in both fore milk and strippings.

**Colostrum.**—The milk given by cows and other mammals for two or three days after the birth of young is termed colostrum, and differs materially in composition from normal milk. It is yellow in color, of an oily consistency, and has a pungent taste. It acts as a purge upon the young. Under the microscope may be seen fat globules, which are larger than at subsequent stages of lactation, and circular cells (0.005–0.025 mm.) related to, if not identical with, the leucocytes or white corpuscles of the blood. It is very high in albumin, which seems to be similar to blood albumin. The following analyses were made by Engling, showing the composition of colostrum from a cow eight years old:

Time after Calving.	Specific Gravity.	Fat.	Casein.	Albumin.	Lactose.	Ash.	Total Solids.
Immediately.....	1.068	3.54	2.65	16.56	3.00	1.18	26.93
After 10 hours.....	1.046	4.66	4.28	9.32	1.42	1.55	21.23
" 24 ".....	1.043	4.75	4.50	6.25	2.85	1.02	19.37
" 48 ".....	1.042	4.21	3.25	2.31	3.46	0.96	14.19
" 72 ".....	1.035	4.08	3.33	1.03	4.10	0.82	13.36

The average of twenty-two analyses of colostrum from different cows by Engling showed total solids 28.31, fat 3.37, casein 4.83, albumin 15.85, lactose 2.48, ash 1.78.

**Changes in Composition During Lactation.**—Crowther and Ruston\* in Scotland, and Eckles and Shaw† in the United States have found

\* Trans High. Agric. Soc. Scotland, v. 23, 1911, p. 93.

† U. S. Dept. of Agric., Bur. of Anim. Ind., Bul. 155, 1913.



that fat, protein, and total solids are highest in the earliest and latest stages of lactation, while ash remains practically constant throughout the period. Lactose according to Crowther and Ruston decreases steadily after the first month or so, while according to Eckles and Shaw the only change attributable to the stage of lactation is a slight decline toward the end. The last named authors note that the fat globules decrease sharply in size during the first 6 weeks, remain practically constant 5 to 6 months, then decrease more rapidly to the end of the period.

**Milk of Young and Old Cows.**—Less data are available on this point than on the other common factors influencing composition. La Cour\* found that the milk from young cows was generally higher in fat content than that of old cows.

**Milk of Different Breeds.**—The following table and the table on page 152 give analyses showing the general characteristics of the milk of single cows of several well-known breeds. The following results by Eckles and Shaw† are the averages of determinations made throughout the whole period of lactation.

AVERAGE COMPOSITION OF MILK OF INDIVIDUAL COWS REPRESENTING FOUR BREEDS (ECKLES AND SHAW).

Breed.	Age of Cow.	Total Solids.	Fat.	Total Protein.	Casein.	Lactose.	Relative Vol. of Fat Globules.	Total Milk Yield.
	Yrs. Mos.							lbs.
Holstein.....	5 3	12.12	3.23	3.00	2.49	5.05	127	8684
Holstein.....	5 0	10.73	2.93	2.70	2.11	4.26	164	8994
Holstein.....	3 8	11.35	3.10	3.21	2.49	4.25	134	8814
Average.....	4 8	11.38	3.09	2.93	2.36	4.51	142	8831
Ayrshire.....	3 8	12.08	3.51	3.11	2.62	4.85	141	6275
Ayrshire.....	4 8	12.71	3.85	3.33	2.81	4.96	160	6382
Average.....	4 2	12.41	3.68	3.25	2.70	4.90	150	6329
Jersey.....	6 10	14.09	4.87	3.70	2.93	4.85	309	5429
Jersey.....	8 1	13.34	4.64	3.27	2.65	4.95	336	6115
Jersey.....	11 4	15.02	5.36	3.97	3.13	4.80	338	5733
Average.....	8 9	14.09	4.95	3.64	2.93	4.87	328	5759
Shorthorn.....	4 4	13.08	3.89	3.40	2.74	5.04	311	5172
Shorthorn.....	4 11	13.01	4.13	3.49	2.87	4.91	353	4449
Shorthorn.....	6 0	12.17	3.37	3.28	2.62	4.98	211	6539
Average.....	5 1	12.69	3.73	3.38	2.74	4.99	282	5387

\* Tidskr. Landökon, 13, 1894, p. 303.

† U. S. Dept. of Agric., Bur. of Anim. Ind., Bul. 156, 1913.



**Influence of Feed on Composition.**—A great amount of work has been done on this subject, but the results are conflicting and not such as can be readily summarized. In general it may be stated that the influence exerted by the feed, except under abnormal conditions or in the case of a few special feeds, is slight compared with that of breed. This point is worthy of consideration in view of the common practice of attributing to feed abnormalities really due to adulteration.

**Intervals Between Milking.**—Several observers have shown that the longer the intervals between milkings the lower the percentage of fat. When the milking night and morning is at the same hour there is little difference.

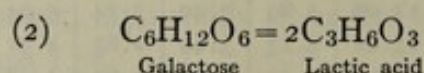
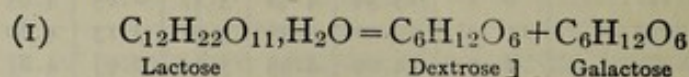
**Frozen Milk.**—Since it is the water that freezes, it follows that in partially frozen milk the unfrozen portion becomes concentrated. This is borne out by the following figures of Richmond: \*

	Frozen Portion.	Unfrozen Portion.
Specific gravity. ....	1.0090	1.0345
Water. ....	96.23	85.62
Fat. ....	1.23	4.73
Lactose. ....	1.42	4.95
Protein. ....	.91	3.90
Ash. ....	.21	.80

The freezing point of milk is considered on page 153.

**Fermentations of Milk.**—These are due to the action of bacteria of various kinds, the most common being the lactic bacilli.

*The Souring of Milk* is caused by the action of a large number of species of acid-forming bacteria, chief among which is the *Bacillus acidilactici*, which multiplies faster than other bacteria in raw milk under favorable conditions of temperature. Part of the milk sugar is acted on and transformed, first into dextrose and galactose, the latter sugar subsequently forming lactic acid, as follows:



In experiments by Van Slyke and Bosworth,† 22% of the lactose was decomposed, of which amount 88.5% went into lactic acid. The citric

\* Analyst, 18, 1893, p. 53.

† N. Y. State Agric. Exp. Sta., Tech. Bul. 18, 1916.



acid was entirely decomposed into acetic acid and carbon dioxide. Albumin was so changed as to pass completely through a porcelain filter. Calcium caseinate reacted with the lactic acid, forming the free protein, which precipitated, and calcium lactate, which remained in solution.

*Abnormal Fermentation.*—Through the agency of micro-organisms that may develop under certain conditions, various changes are produced in milk that to some extent alter its character. Thus *bitter milk* is sometimes produced as the result of some organism as yet but little understood.

Occasionally milk is found possessing a peculiarly thick and slimy consistency, whereby it may be drawn out in threads, by dipping a spoon into the milk and withdrawing it therefrom. This is termed *ropy* milk, and is more often met with in warm weather. It is undoubtedly produced as a result of bacterial action.

*Enzyme-forming Bacteria* are not uncommonly developed in milk, causing various proteolytic changes, whereby the casein is partially transformed into peptones, caseoses, etc.

*Chromogenic Bacteria* are the agencies that produce peculiar pigments in milk. Thus red milk is due to *Bacillus erythrogenes*; yellow milk to *Bacillus xynxanthus*; blue milk to *Bacillus cyanagenes*. The latter is quite common, appearing ordinarily in patches in the milk.

#### CHEMICAL ANALYSIS OF MILK.

Ordinarily, in ascertaining the nutritive value of milk, one determines its specific gravity, total solids, fat, protein, lactose, and ash. Occasionally it is thought desirable to make a distinction in the case of protein between the casein and the albumin. Rarely is it necessary to further subdivide the nitrogenous bodies in milk, unless in connection with a special study of the proteolytic changes which it undergoes.

The total solids, fat, and ash are usually all determined directly, and, in the case of the lactose and the protein, a determination of either one may be directly made (whichever is most convenient), the other being calculated by difference.

When foreign ingredients or adulterants are present in milk, special methods are employed to detect them.

**Preparation of the Sample.**—In procuring a sample for analysis, the greatest care is necessary to insure a homogeneous sample. By far the best method in every case, where possible, is to pour the milk back and forth from one vessel to another (i.e., pour from the original container



into an empty vessel and back at least once). Where this is impossible from the size of the container or for any other reason, the milk should be thoroughly mixed with a dipper. A "sampler," of which the Scovell

sampling-tube (Fig. 43, *A*) is a convenient form, also aids in securing a representative sample, and is invaluable when it is desirable to secure a definite fraction of the whole for a composite sample.

This instrument consists of a brass or copper tube made in two parts which telescope accurately together as shown in Fig. 43, *A*, the lower part being closed at the bottom, but provided with three or more lateral slits. The sampler, drawn out to its full length, is carefully inserted in the tank containing the milk and lowered to the bottom, after which the upper part is pressed down over the lower so as to close the slits, and the tube is then lifted out of the tank, containing a fairly representative sample of the milk.

Samples may be preserved in condition suitable for determination of fat and solids for several days by adding to each quart 1 gram of potassium bichromate, 0.2 gram of mercuric chloride mixed with a coal-tar color to show its poisonous nature, or 1 cc. of 40 % formaldehyde. If other determinations are to be made, the analyst should make certain that the results on the preserved samples are the same as those on the fresh material.

In all operations to which a milk sample is submitted during the process of analysis, it should invariably be poured into a clean empty vessel and back, or shaken, whenever it has been at rest for an appreciable time, in order to insure a homogeneous mixture.

**Determination of Specific Gravity.**—This is most readily obtained with the aid of a hydrometer, accurately graduated within the limits of the widest possible variation in the specific gravity of milk. Hydrometers for special use with milk are known as lactometers, and are graduated variously. One of the most convenient forms of this instrument is the Quevenne lactometer, graduated from 15° to 40°, corresponding to specific gravity 1.015 to 1.040. This instrument, shown in Fig. 43, *B*, has a thermometer combined with it, the stem con-

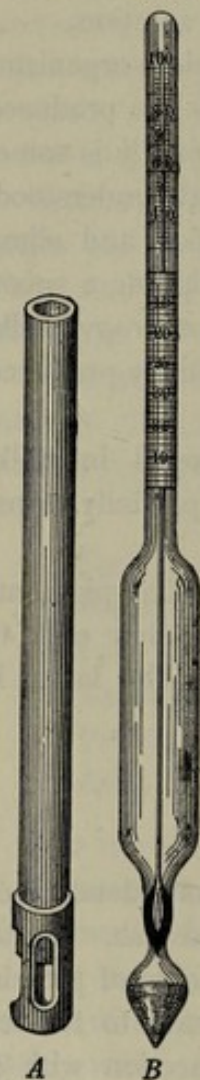


FIG. 43.

*A*, Scovell Milk-Sampling Tube.  
*B*, Quevenne Lactometer.



taining a double scale, on the lower part of which the specific gravity is read, while the temperature is read from the upper part.

Another form of instrument is termed the New York Board of Health lactometer, which is not graduated to read the specific gravity directly, but has an arbitrary scale divided into 120 equal parts, the zero being equal to the specific gravity of water, while 100 corresponds to a specific gravity of 1.029. Deghuée\* has devised a special form requiring only 4 ounces of milk. To convert readings on the New York Board of Health scale to Quevenne degrees they must be multiplied by .29.

QUEVENNE LACTOMETER DEGREES CORRESPONDING TO NEW YORK BOARD OF HEALTH LACTOMETER DEGREES.

Board of Health Degrees.	Quevenne Scale.	Board of Health Degrees.	Quevenne Scale.	Board of Health Degrees.	Quevenne Scale.
60	17.4	81	23.5	101	29.3
61	17.7	82	23.8	102	29.6
62	18.0	83	24.1	103	29.9
63	18.3	84	24.4	104	30.2
64	18.6	85	24.6	105	30.5
65	18.8	86	24.9	106	30.7
66	19.1	87	25.2	107	31.0
67	19.4	88	25.5	108	31.3
68	19.7	89	25.8	109	31.6
69	20.0	90	26.1	110	31.9
70	20.3	91	26.4	111	32.2
71	20.6	92	26.7	112	32.5
72	20.9	93	27.0	113	32.8
73	21.2	94	27.3	114	33.1
74	21.5	95	27.6	115	33.4
75	21.7	96	27.8	116	33.6
76	22.0	97	28.1	117	33.9
77	22.3	98	28.4	118	34.2
78	22.6	99	28.7	119	34.5
79	22.9	100	29.0	120	34.8
80	23.2				

If extreme accuracy is desired, the Westphal balance or the pycnometer should be used for the determination of specific gravity. For ordinary cases, however, the lactometer, if carefully made, is sufficiently accurate.

With any other form of lactometer than the Quevenne, a separate thermometer is necessary in order to determine the temperature, the common practice being to standardize all such instruments at 60° F. (15.6° C.).

Readings at temperatures other than 60° may be corrected to that temperature by the aid of the table on page 133.

**DETERMINATION OF TOTAL SOLIDS.—Dish Method.**—For purposes of milk analysis, platinum dishes are by far the most desirable. These, if made for the purpose, should be of the shape shown in Fig. 51, measur-

\* Jour. Ind. Eng. Chem., 3, 1911, p. 405.



FOR CORRECTING THE SPECIFIC GRAVITY OF MILK ACCORDING TO  
TEMPERATURE (BY DR. PAUL VIETH).

Degrees of Lactom- eter.	Degrees of Thermometer (Fahrenheit).															
	45	46	47	48	49	50	51	52	53	54	55	56	57	58	59	60
20.....	19.0	19.0	19.1	19.1	19.2	19.2	19.3	19.4	19.4	19.5	19.6	19.7	19.8	19.9	19.9	—
21.....	19.0	20.0	20.0	20.1	20.2	20.2	20.3	20.3	20.4	20.5	20.6	20.7	20.8	20.9	20.9	—
22.....	20.0	21.0	21.0	21.1	21.2	21.2	21.3	21.3	21.4	21.5	21.6	21.7	21.8	21.9	21.9	—
23.....	21.0	22.0	22.0	22.1	22.2	22.2	22.3	22.3	22.4	22.5	22.6	22.7	22.8	22.8	22.9	—
24.....	22.0	22.9	23.0	23.1	23.2	23.2	23.3	23.3	23.4	23.5	23.6	23.6	23.7	23.8	23.9	—
25.....	23.8	23.9	24.0	24.0	24.1	24.1	24.2	24.3	24.4	24.5	24.6	24.6	24.7	24.8	24.9	—
26.....	24.8	24.9	24.9	25.0	25.1	25.1	25.2	25.2	25.3	25.4	25.5	25.6	25.7	25.8	25.9	—
27.....	25.8	25.9	25.9	26.0	26.1	26.1	26.2	26.2	26.3	26.4	26.5	26.6	26.7	26.8	26.9	—
28.....	26.7	26.8	26.8	26.9	27.0	27.0	27.1	27.2	27.3	27.4	27.5	27.6	27.7	27.8	27.9	—
29.....	27.7	27.8	27.8	27.9	28.0	28.0	28.1	28.2	28.3	28.4	28.5	28.6	28.7	28.8	28.9	—
30.....	28.6	28.7	28.7	28.8	28.9	29.0	29.1	29.1	29.2	29.3	29.4	29.6	29.7	29.8	29.9	—
31.....	29.5	29.6	29.6	29.7	29.8	29.9	30.0	30.1	30.2	30.3	30.4	30.5	30.6	30.8	30.9	—
32.....	30.4	30.5	30.5	30.6	30.7	30.9	31.0	31.1	31.2	31.3	31.4	31.5	31.6	31.7	31.9	—
33.....	31.3	31.4	31.4	31.5	31.6	31.8	31.9	32.0	32.1	32.3	32.4	32.5	32.6	32.7	32.9	—
34.....	32.2	32.3	32.3	32.4	32.5	32.7	32.9	33.0	33.1	33.2	33.3	33.5	33.6	33.7	33.9	—
35.....	33.0	33.1	33.2	33.4	33.5	33.6	33.8	33.9	34.0	34.2	34.3	34.5	34.6	34.7	34.9	—

	61	62	63	64	65	66	67	68	69	70	71	72	73	74	75
20.....	20.1	20.2	20.2	20.3	20.4	20.5	20.6	20.7	20.9	21.0	21.1	21.2	21.3	21.5	21.6
21.....	21.1	21.2	21.3	21.4	21.5	21.6	21.7	21.8	22.0	22.1	22.2	22.3	22.4	22.5	22.6
22.....	22.1	22.2	22.3	22.4	22.5	22.6	22.7	22.8	23.0	23.1	23.2	23.3	23.4	23.5	23.7
23.....	23.1	23.2	23.3	23.4	23.5	23.6	23.7	23.8	24.0	24.1	24.2	24.3	24.4	24.6	24.7
24.....	24.1	24.2	24.3	24.4	24.5	24.6	24.7	24.9	25.0	25.1	25.2	25.3	25.5	25.6	25.7
25.....	25.1	25.2	25.3	25.4	25.5	25.6	25.7	25.9	26.0	26.1	26.2	26.4	26.5	26.6	26.8
26.....	26.1	26.2	26.3	26.5	26.6	26.7	26.8	27.0	27.1	27.2	27.3	27.4	27.5	27.7	27.8
27.....	27.1	27.3	27.4	27.5	27.6	27.7	27.8	28.0	28.1	28.2	28.3	28.4	28.6	28.7	28.9
28.....	28.1	28.3	28.4	28.5	28.6	28.7	28.8	29.0	29.1	29.2	29.4	29.5	29.7	29.8	29.9
29.....	29.1	29.3	29.4	29.5	29.6	29.8	29.9	30.1	30.2	30.3	30.4	30.5	30.7	30.9	31.0
30.....	30.1	30.3	30.4	30.5	30.7	30.8	30.9	31.1	31.2	31.3	31.5	31.6	31.8	31.9	32.1
31.....	31.2	31.3	31.4	31.5	31.7	31.7	31.8	32.0	32.2	32.4	32.5	32.6	32.8	33.0	33.1
32.....	32.2	32.3	32.5	32.6	32.7	32.9	33.0	33.2	33.3	33.4	33.6	33.7	33.9	34.0	34.2
33.....	33.2	33.3	33.5	33.6	33.8	33.9	34.0	34.2	34.3	34.5	34.6	34.7	34.9	35.1	35.2
34.....	34.2	34.3	34.5	34.6	34.8	34.9	35.0	35.2	35.3	35.5	35.6	35.8	36.0	36.1	36.3
35.....	35.2	35.3	35.5	35.6	35.8	35.9	36.1	36.2	36.4	36.5	36.7	36.8	37.0	37.2	37.3

ing about  $2\frac{3}{4}$  inches in diameter at the top, and  $2\frac{1}{4}$  inches in diameter at the bottom, having carefully rounded rather than square edges, and being  $\frac{1}{2}$  inch deep. The bottom is not perfectly flat, but slightly crowned outward. Such a dish will hold about 35 cc.

For purposes of economy it is best to have these dishes spun out with a thick bottom, but with thin sides, not so thin, however, as to be too readily bent.

If platinum dishes cannot be afforded, dishes of porcelain, glass, aluminum, nickel, or even tin may be used, but in all cases should be as thin as practicable.

About 5 cc. of the thoroughly mixed sample of milk are carefully



transferred by means of a pipette to a tared dish on the scale-pan, and its weight accurately determined. The dish with its contents is then transferred to a water-bath, being placed over an opening preferably but little smaller than the diameter of the bottom of the dish, so that as large a surface as possible is in contact with the live steam of the bath. Here it is kept for at least two hours, after which the dish is wiped dry while still hot, transferred to a desiccator, cooled, and weighed.\*

**Babcock Asbestos Method.**†—Provide a hollow cylinder of perforated sheet metal, 60 mm. long and 20 mm. in diameter, closed 5 mm. from one end by a disk of the same material. The perforations should be about 0.7 mm. in diameter and about 0.7 mm. apart. Fill loosely with from 1.5 to 2.5 grams of freshly ignited, woolly asbestos, free from fine and brittle material, cool in a desiccator, and weigh. Introduce a weighed quantity of milk (between 3 and 5 grams), and dry in a water-oven to constant weight, which is usually reached after four hours' heating.

**DETERMINATION OF ASH.**—The platinum dish containing the milk residue, obtained in the determination of total solids by the dish method described above, is next placed upon a suitable support above a Bunsen flame (a platinum triangle or a ring stand is convenient for this), and the residue is ignited at a dull-red heat to a perfectly white ash, after which it is cooled and weighed.

**DETERMINATION OF FAT.**—**Babcock Asbestos Method.**—Extract the residue from the determination of water by the Babcock asbestos method with anhydrous ether in a continuous extraction apparatus, until all the fat is removed, which usually requires two hours. Evaporate the ether, dry the fat in the extraction flask at the temperature of boiling water, and weigh. The fat may also be determined by difference, drying the extracted cylinders at the temperature of boiling water.

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\* It is a common practice to transfer the milk residue, after a preliminary drying on the water-bath, to an air-oven, kept at a temperature of from 100° to 105°, where it is dried to a constant weight; but after an experience in analyzing over 30,000 samples of milk, the author is prepared to state that in his opinion the results obtained by the above method of procedure, using the water-bath alone, are more satisfactory. It is impossible to keep a milk residue at a temperature above 100° for any length of time without its undergoing decomposition; especially as to its sugar content, as is shown by the darkening in color. A milk residue should be nearly pure white, a brownish color showing incipient decomposition. Hence, by continued heating, especially at the temperature of 105°, the residue would continue to lose weight almost indefinitely. If it is thought best to give a final drying in the air-oven, the time should be short and the temperature employed should not in any case exceed 100°.

† U. S. Dept. of Agric., Bur. of Chem., Bul. 38, p. 100.



**The Adams Method.**—For this method a strip of fat-free filter-paper about  $2\frac{1}{2}$  inches wide and 22 inches long is rolled into a coil and held in place by a wire as shown in Fig. 44. Schleicher and Schüll furnish fat-free strips especially for this work, but it is very easy to prepare the strips and extract them with the Soxhlet apparatus.

About 5 grams of milk are run into a beaker with a pipette, and the weight of the beaker and milk are determined. The coil is then introduced into the beaker, holding it by the wire in such a manner that as much as possible of the milk is absorbed by the paper. It is often possible to take up almost the last drop of the milk. By then weighing the beaker,

the amount of milk absorbed by the coil is determined by difference, and the paper coil is hung up and dried, first in the air and then in the oven at a temperature not exceeding  $100^{\circ}$ . Another method of charging the paper coil consists in suspending it by the wire and gradually delivering upon it 5 cc. of the milk from a pipette, the density of the milk being known.



FIG. 44.—The Adams Milk-fat Coil.

The coil containing the dried residue is then transferred to the Soxhlet extraction apparatus (see p. 53) and subjected to continuous extraction with anhydrous ether for at least two hours, the receiving-flask being first accurately weighed. The tared flask with its contents is freed from all remaining ether, first on the water-bath and finally in the air-oven. It is then cooled and weighed, the increase in weight representing the fat in the amount of milk absorbed by the coil. If there is any doubt about all the fat having been extracted at first, the process of extraction may be continued till there is no longer a gain in weight of the flask. Experience soon shows the length of time necessary for the complete extraction, which of course depends on the degree of heat employed, and the frequency with which the extracting-tube overflows. Two hours is ample for most cases, in which the conditions are such that the ether siphons over from the extraction-tube ten times per hour.

**FAT METHODS BASED ON CENTRIFUGAL SEPARATION.**—These methods are the most practicable for commercial work and for use by the public analyst, since they are much more rapid, and, if carefully carried out, practically as accurate as the Adams method. They all depend upon the use of a centrifuge usually having hinged pockets in which are carried graduated bottles, into each of which a measured



quantity of milk is introduced. The milk is then subjected to the action of a suitable reagent, which dissolves the casein and liberates the fat in a pure state, after which, by whirling at a high speed, the pockets are thrown out horizontally and the milk fat driven into the neck of each bottle, where the amount is directly read.

**The Babcock Test**, although devised originally for the use of creameries and dairymen, is now extensively employed for fat determination in the laboratory. Leach found that the results by the Babcock test and the Adams method, obtained from time to time during ten years, agreed within narrow limits. The following figures show the results of such comparative determinations made in duplicate on three samples of milk, viz., a pure whole milk, (1) and (2); a watered milk, (3) and (4), and a milk centrifugally skimmed, (5) and (6).

COMPARATIVE FAT DETERMINATION BY ADAMS-SOXHLET AND BY BABCOCK PROCESSES.

	Per Cent of Fat by the Adams-Soxh- let Process.	Per Cent of Fat by the Babcock Process.
A whole milk... (1).....	4.27	4.30
(2).....	4.28	4.35
A watered milk (3).....	2.70	2.70
(4).....	2.74	2.80
A skimmed milk (5).....	0.16	0.15
(6).....	0.14	0.15

Equally satisfactory results were obtained by Winton, using the Babcock asbestos method for comparison.

*The Centrifuge.*—Various styles of centrifuge, carrying from 2 to 40 bottles, are in use for this process.

Two forms of hand machine are shown in Fig. 45, one (*D*), for two bottles, arranged to screw on the edge of a table, the other for twelve bottles inclosed in a cast-iron case.

The number of revolutions of the revolving frame for each turn of the crank and the number of turns per minute necessary to secure the requisite number of revolutions of the frame should be determined once for all for each machine and the latter adhered to in making all tests.



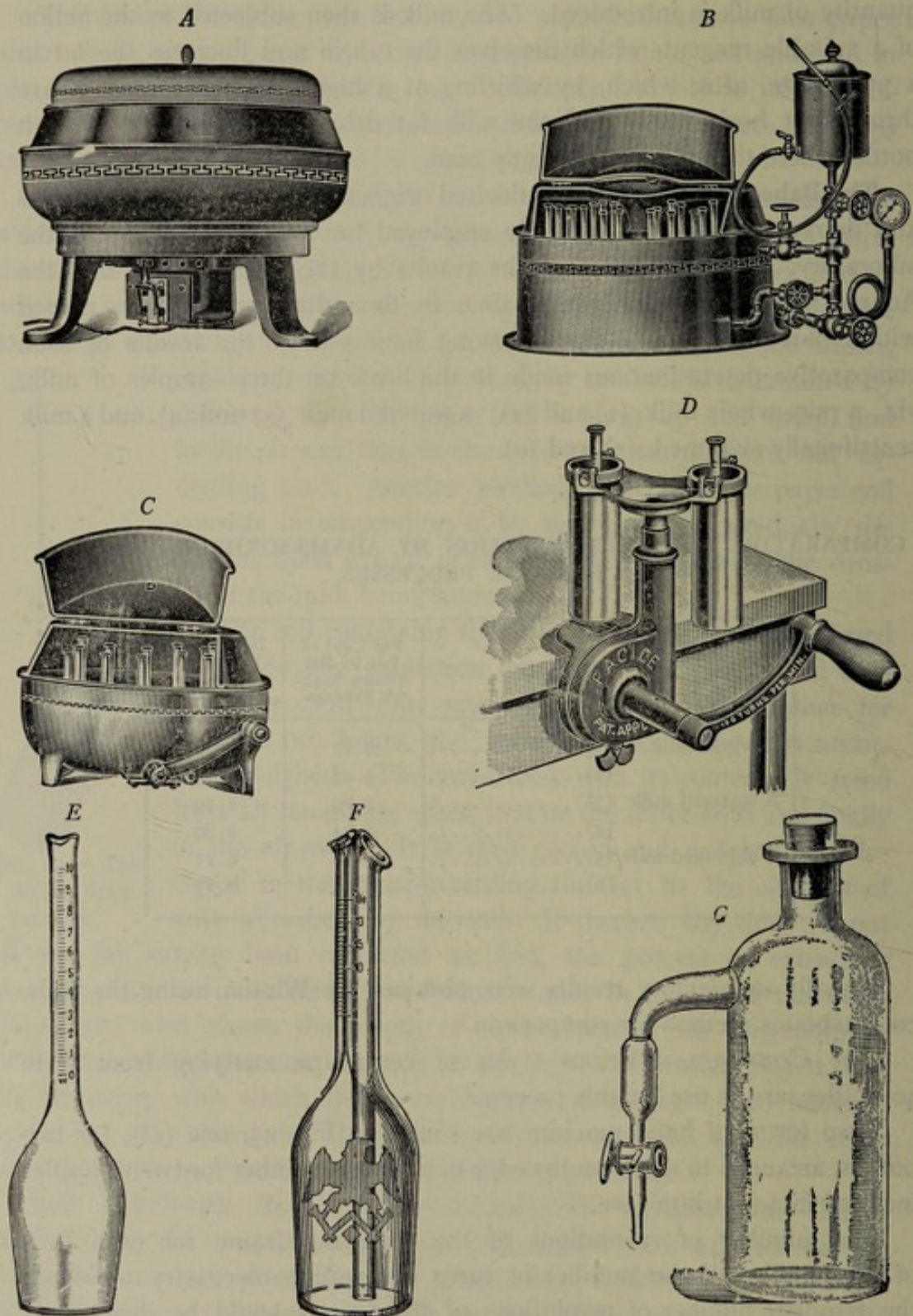


FIG. 45.—Apparatus for Babcock Test.

*A*, Burrell's electric centrifuge; *B*, Burrell's steam turbine centrifuge; *C* and *D*, Burrell's hand centrifuges; *E*, milk bottle; *F*, Wagner's skim-milk bottle; *G*, Swedish or combined acid bottle.



The steam turbine machines (Fig. 45, *B*) are simple in construction and the steam serves to keep the bottles warm as well as to furnish power. The steam impinges against a series of paddles on the outer periphery of the revolving frame, driving it like a horizontal water-wheel. A reverse steam jet, steam gauge, and hot-water tank for filling the bottles are also provided.

Fig. 45, *A* shows an electric machine for 24 to 36 bottles. Laboratory centrifuges are also provided with frames for Babcock bottles.

*Glassware.*—The ordinary test bottle for milk is shown in Fig. 45, *E*. It has graduations corresponding to from 0 to 10% of fat, using 17.6 cc. of milk. One of various forms of skim milk bottle is also shown (*F*). The graduated tube has a capacity corresponding to only 0.25% for its entire length, hence the need of a second tube of larger bore for filling.

The pipettes are graduated to hold 17.6 cc., which for average milk weighs 18 grams. The lower tube should be of such a size as to enter the neck of the test bottle.

A 17.5 cc. cylinder is provided for measuring the acid, but where considerable numbers of tests are made some special measuring device is desirable. Fig. 45, *G* shows a combined acid bottle and pipette, the latter being filled by tipping up the bottle.

*Manipulation.*—Pipette 17.6 cc. (corresponding to 18 grams) of the milk into the test bottle and add 17.5 cc. of commercial sulphuric acid. (sp.gr. 1.82–1.84). Mix thoroughly by a vigorous rotatory movement holding the neck of the bottle between the fingers and at a slight angle away from the body. The lumps of curd which at first form disappear upon shaking; much heat is developed during the mixing and the color changes to deep brown.

Place the test bottles in the pockets of the centrifuge (symmetrically arranged to keep the revolving frame in balance) and whirl at the rate of 800 to 1000 revolutions per minute, according to the diameter of the frame, for 5 minutes. Stop the machine, fill each bottle up to the neck with boiling water and whirl for two minutes longer. Add boiling water up to near the top of the graduation and whirl finally for two minutes.

Remove the bottles from the machine and take the readings of the bottom and the very top of the fat column, the difference being the per cent of fat. If desired, the percentage may be obtained directly by means of calipers. To avoid danger of cooling it is well to immerse the bottles nearly to the top of the neck in water at 60° C., removing one at a time for reading.



**The Werner-Schmidt Method.**—Ten cc. of milk are introduced by means of a pipette into a large test-tube of 50 cc. capacity, and 10 cc. of concentrated hydrochloric acid are added. The mixture is shaken and heated till the liquid turns a dark brown, either by direct boiling for a minute or two, or by immersing the tube in boiling water for from five to ten minutes. The tube is then cooled by immersion in cold water, and 30 cc. of washed ether is added. The tube is closed by a cork provided with tubes similar to a wash-bottle, the larger tube being adapted to slide up and down in the cork, and preferably being turned up slightly at the bottom. The contents of the tube are shaken, the ether layer allowed to separate, and the sliding-tube arranged so that it terminates slightly above the junction of the two layers. The ether is then blown out into a weighed flask. A second and a third portion of ether of 10 cc. each are successively shaken with the acid liquid and added to the contents of the weighed flask, from which the ether is subsequently evaporated and the weight of the fat easily obtained.

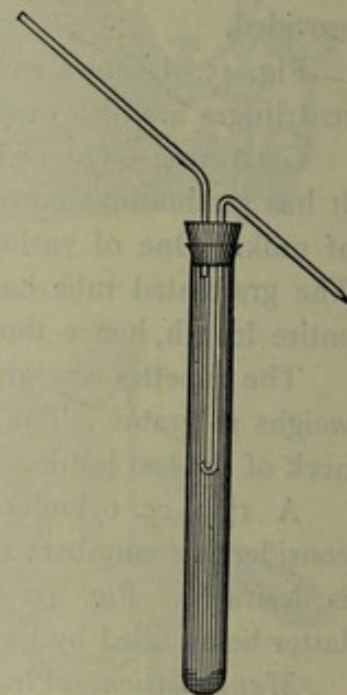


FIG. 46.—The Werner-Schmidt Fat Apparatus.

Instead of measuring the milk into the testing-tube, a known weight of milk may be operated on. A sour milk may be readily tested in this way, provided it is previously well mixed.

**Determination of Fat by the Wollny Milk-fat Refractometer.\***—This instrument presents the same appearance as the butyro-refractometer, Fig. 38, with an arbitrary scale reading from 0 to 100, the equivalent readings in indices of refraction of the Wollny instrument varying from 1.3332 to 1.4220. Exactly 30 cc. of the milk to be tested are measured into the stoppered flask *A*, Fig. 47. This may be done by the use of the automatic pipette, which holds exactly  $7\frac{1}{2}$  cc., removing four pipettes full of the milk. *B* is a numbered tin sampling-tube in which the milk sample is kept for convenience, and into which the automatic pipette readily fits. Having measured 30 cc. of the milk into the flask *A*, 12 drops of a solution of 70 grams potassium bichromate and 312.5 cc. of stronger ammonia in one liter of water may be added as a preservative,

\* Milch Zeit., 1900, pp. 50-53.



if the sample is to be kept for some time before finishing the test. Twelve drops of glacial acetic acid are added to curdle the milk. The flask is then corked and shaken for one to two minutes in a mechanical shaker, after which 3 cc. of a standard alkaline solution are added, and the flask corked and shaken for ten minutes in the mechanical shaker, the temperature being kept at  $17.5^{\circ}\text{C}$ . The standard alkaline solution is prepared

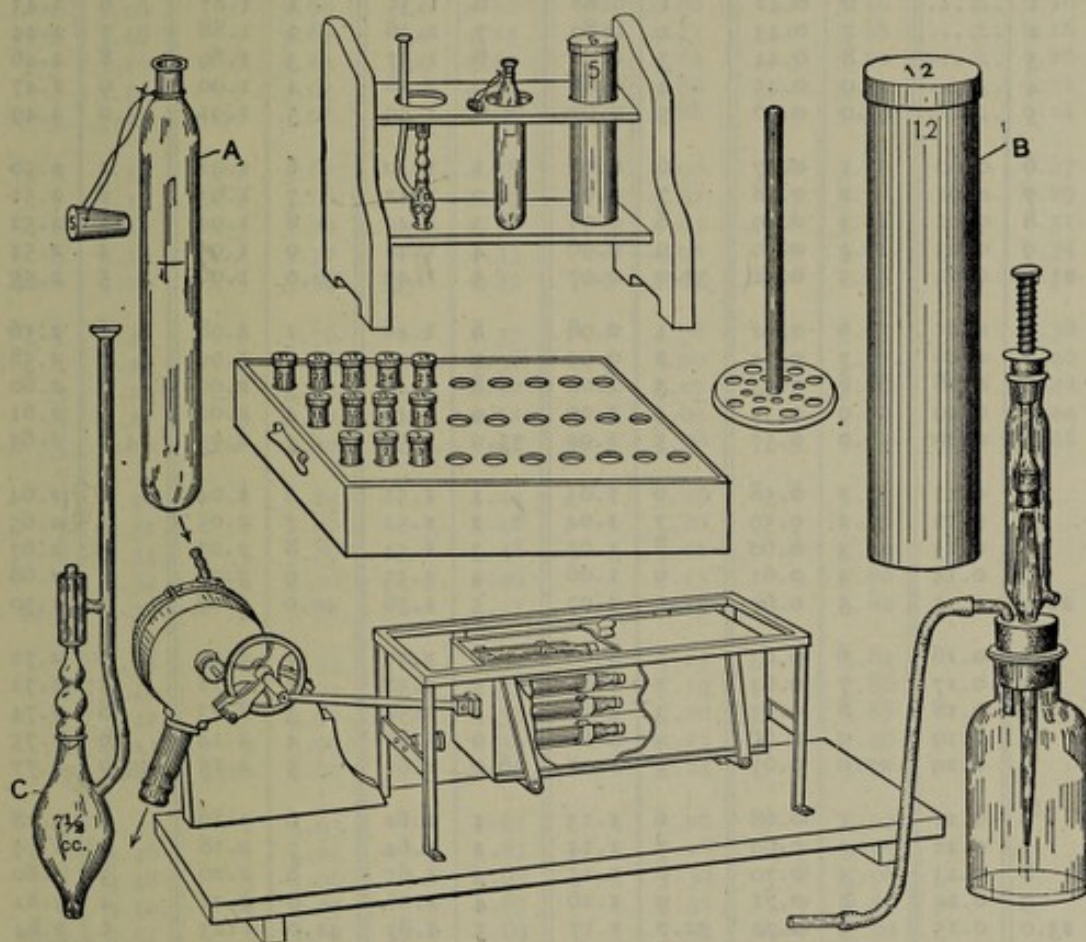


FIG. 47.—Accessories for Carrying Out the Wollny Milk-fat Process.

by dissolving 800 cc. of potassium hydroxide in a liter of water, adding 600 cc. of glycerin and 200 grams pulverized copper hydrate, the mixture being allowed to stand for several days before using, shaking at intervals. Finally 6 cc. of water-saturated ether are added to the mixture in the flask, using for convenience the automatic pipette fitted in the corked bottle as shown. The flask is again shaken for fifteen minutes in the mechanical shaker, and whirled for three minutes in the centrifuge, after which a few drops of the ether solution are transferred to the refractometer, and the reading taken. The percentage of fat is obtained by means of the following table:



PERCENTAGES OF FAT CORRESPONDING TO SCALE READINGS ON THE  
WOLLNY REFRACTOMETER.

Scale Read- ing.	Per Cent Fat.	Scale Read- ing.	Per Cent Fat.	Scale Read- ing.	Per Cent Fat.	Scale Read- ing.	Per Cent Fat.	Scale Read- ing.	Per Cent Fat.	Scale Read- ing.	Per Cent Fat.
20.0	.....	24.5	0.41	29.0	0.87	33.5	1.34	38.0	1.85	42.5	2.41
1	.....	6	0.42	1	0.88	6	1.35	1	1.87	6	2.43
2	.....	7	0.43	2	0.89	7	1.36	2	1.88	7	2.44
3	.....	8	0.44	3	0.90	8	1.37	3	1.89	8	2.46
4	.....	9	0.45	4	0.91	9	1.38	4	1.90	9	2.47
5	.....	25.0	0.46	5	0.92	34.0	1.39	5	1.91	43.0	2.49
6	0.00	1	0.47	6	0.93	1	1.40	6	1.92	1	2.50
7	0.01	2	0.48	7	0.94	2	1.42	7	1.93	2	2.51
8	0.02	3	0.49	8	0.95	3	1.43	8	1.94	3	2.52
9	0.03	4	0.50	9	0.96	4	1.44	9	1.95	4	2.54
21.0	0.04	5	0.51	30.0	0.97	5	1.45	39.0	1.96	5	2.55
1	0.05	6	0.52	1	0.98	6	1.46	1	1.98	6	2.56
2	0.06	7	0.53	2	0.99	7	1.47	2	1.99	7	2.58
3	0.08	8	0.54	3	1.00	8	1.48	3	2.00	8	2.60
4	0.09	9	0.55	4	1.01	9	1.49	4	2.02	9	2.61
5	0.10	26.0	0.57	5	1.02	35.0	1.50	5	2.03	44.0	2.63
6	0.11	1	0.58	6	1.03	1	1.51	6	2.04	1	2.64
7	0.12	2	0.59	7	1.04	2	1.52	7	2.05	2	2.65
8	0.13	3	0.60	8	1.05	3	1.54	8	2.07	3	2.67
9	0.14	4	0.61	9	1.06	4	1.55	9	2.08	4	2.68
22.0	0.15	5	0.62	31.0	1.07	5	1.56	40.0	2.09	5	2.70
1	0.16	6	0.63	1	1.08	6	1.57	1	2.10	6	2.71
2	0.17	7	0.64	2	1.09	7	1.58	2	2.12	7	2.72
3	0.18	8	0.65	3	1.10	8	1.59	3	2.13	8	2.74
4	0.19	9	0.66	4	1.11	9	1.60	4	2.14	9	2.75
5	0.20	27.0	0.67	5	1.12	36.0	1.61	5	2.15	45.0	2.77
6	0.21	1	0.68	6	1.13	1	1.62	6	2.16	1	2.78
7	0.22	2	0.69	7	1.14	2	1.64	7	2.18	2	2.79
8	0.23	3	0.70	8	1.15	3	1.65	8	2.20	3	2.80
9	0.24	4	0.71	9	1.16	4	1.66	9	2.21	4	2.82
23.0	0.25	5	0.72	32.0	1.17	5	1.67	41.0	2.23	5	2.84
1	0.26	6	0.73	1	1.18	6	1.68	1	2.24	6	2.85
2	0.27	7	0.74	2	1.19	7	1.69	2	2.25	7	2.87
3	0.28	8	0.75	3	1.20	8	1.70	3	2.26	8	2.88
4	0.29	9	0.76	4	1.22	9	1.71	4	2.27	9	2.89
5	0.30	28.0	0.77	5	1.23	37.0	1.72	5	2.28	46.0	2.90
6	0.31	1	0.78	6	1.24	1	1.73	6	2.30	1	2.92
7	0.32	2	0.79	7	1.25	2	1.75	7	2.32	2	2.93
8	0.33	3	0.80	8	1.26	3	1.76	8	2.33	3	2.94
9	0.34	4	0.81	9	1.27	4	1.78	9	2.34	4	2.96
24.0	0.36	5	0.82	33.0	1.28	5	1.79	42.0	2.35	5	2.98
1	0.37	6	0.83	1	1.29	6	1.80	1	2.37	6	3.00
2	0.38	7	0.84	2	1.30	7	1.81	2	2.38	7	3.01
3	0.39	8	0.85	3	1.31	8	1.82	3	2.39	8	3.02
4	0.40	9	0.86	4	1.32	9	1.84	4	2.40	9	3.03
5	0.41	29.0	0.87	5	1.34	38.0	1.85	5	2.41	47.0	3.05



PERCENTAGES OF FAT CORRESPONDING TO SCALE READINGS ON THE  
WOLLNY REFRACTOMETER—(Continued).

Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.	Scale Reading.	Per Cent Fat.
47.0	3.05	50.5	3.59	54.0	4.18	57.5	4.78	61.0	5.44	64.5	6.14
1	3.06	6	3.60	1	4.20	6	4.80	1	5.46	6	6.16
2	3.08	7	3.61	2	4.22	7	4.82	2	5.48	7	6.18
3	3.10	8	3.63	3	4.23	8	4.84	3	5.50	8	6.20
4	3.12	9	3.64	4	4.25	9	4.86	4	5.52	9	6.22
5	3.14	51.0	3.66	5	4.26	58.0	4.88	5	5.54	65.0	6.24
6	3.15	1	3.67	6	4.28	1	4.90	6	5.56	1	6.27
7	3.16	2	3.68	7	4.29	2	4.92	7	5.58	2	6.29
8	3.17	3	3.70	8	4.31	3	4.94	8	5.60	3	6.31
9	3.18	4	3.72	9	4.33	4	4.95	9	5.61	4	6.34
48.0	3.20	5	3.74	55.0	4.35	5	4.97	62.0	5.63	5	6.36
1	3.21	6	3.76	1	4.37	6	4.98	1	5.65	6	6.38
2	3.23	7	3.78	2	4.38	7	5.00	2	5.66	7	6.40
3	3.25	8	3.80	3	4.40	8	5.02	3	5.68	8	6.42
4	3.27	9	3.82	4	4.42	9	5.04	4	5.70	9	6.44
5	3.28	52.0	3.84	5	4.43	59.0	5.06	5	5.72	66.0	6.46
6	3.30	1	3.85	6	4.44	1	5.08	6	5.74		
7	3.32	2	3.87	7	4.46	2	5.10	7	5.76		
8	3.33	3	3.89	8	4.48	3	5.11	8	5.78		
9	3.34	4	3.90	9	4.49	4	5.13	9	5.80		
49.0	3.36	5	3.92	56.0	4.51	5	5.15	63.0	5.82		
1	3.38	6	3.93	1	4.53	6	5.17	1	5.84		
2	3.40	7	3.95	2	4.55	7	5.19	2	5.86		
3	3.42	8	3.97	3	4.57	8	5.20	3	5.88		
4	3.43	9	3.99	4	4.59	9	5.22	4	5.90		
5	3.44	53.0	4.01	5	4.60	60.0	5.24	5	5.92		
6	3.45	1	4.03	6	4.61	1	5.26	6	5.94		
7	3.46	2	4.04	7	4.63	2	5.28	7	5.96		
8	3.48	3	4.06	8	4.65	3	5.30	8	5.98		
9	3.50	4	4.07	9	4.67	4	5.32	9	6.00		
50.0	3.51	5	4.09	57.0	4.69	5	5.34	64.0	6.02		
1	3.53	6	4.10	1	4.71	6	5.36	1	6.04		
2	3.55	7	4.12	2	4.73	7	5.38	2	6.07		
3	3.56	8	4.14	3	4.75	8	5.40	3	6.09		
4	3.57	9	4.16	4	4.76	9	5.42	4	6.12		
5	3.59	54.0	4.18	5	4.78	61.0	5.44	5	6.14		

The following table is of use for those who wish to employ the Wollny method, but have the Abbé refractometer instead of the milk-fat refractometer.



INDICES OF REFRACTION ( $n_D$ ) CORRESPONDING TO SCALE READINGS OF  
THE WOLLNY MILK-FAT REFRACTOMETER.

Refractive Index, $n_D$ .	Fourth Decimal of $n_D$ .									
	0	1	2	3	4	5	6	7	8	9
	Scale Readings.									
1.333	.....	.....	0.0	0.1	0.2	0.3	0.4	0.5	0.5	0.6
1.334	0.7	0.8	0.9	1.0	1.1	1.2	1.3	1.4	1.5	1.6
1.335	1.7	1.8	1.9	2.0	2.1	2.1	2.2	2.3	2.4	2.5
1.336	2.8	2.7	2.8	2.9	3.0	3.1	3.2	3.3	3.4	3.5
1.337	3.6	3.7	3.7	3.8	3.9	4.0	4.1	4.2	4.3	4.4
1.338	4.5	4.6	4.7	4.8	4.9	5.0	5.1	5.2	5.3	5.4
1.339	5.5	5.6	5.7	5.8	5.9	6.0	6.1	6.2	6.3	6.4
1.340	6.5	6.6	6.7	6.8	6.9	6.9	7.0	7.1	7.2	7.3
1.341	7.4	7.5	7.6	7.7	7.8	7.9	8.0	8.1	8.2	8.3
1.342	8.4	8.5	8.6	8.7	8.8	8.9	9.0	9.1	9.2	9.3
1.343	9.4	9.5	9.6	9.7	9.8	9.9	10.0	10.1	10.2	10.3
1.344	10.4	10.5	10.6	10.7	10.8	10.9	11.0	11.1	11.2	11.3
1.345	11.4	11.5	11.5	11.6	11.7	11.8	11.9	12.0	12.1	12.2
1.346	12.3	12.4	12.5	12.6	12.7	12.8	12.9	13.0	13.1	13.2
1.347	13.3	13.4	13.5	13.6	13.7	13.8	13.9	14.0	14.1	14.2
1.348	14.3	14.4	14.5	14.6	14.7	14.8	14.9	15.0	15.1	15.2
1.349	15.3	15.4	15.5	15.6	15.7	15.8	15.9	16.0	16.1	16.2
1.350	16.3	16.4	16.5	16.6	16.7	16.8	16.9	17.0	17.1	17.2
1.351	17.3	17.4	17.5	17.6	17.7	17.8	17.9	18.0	18.1	18.2
1.352	18.3	18.4	18.5	18.6	18.7	18.8	18.9	19.0	19.1	19.2
1.353	19.3	19.4	19.5	19.6	19.7	19.8	19.9	20.0	20.1	20.2
1.354	20.3	20.4	20.5	20.6	20.7	20.8	20.9	21.0	21.1	21.2
1.355	21.3	21.4	21.5	21.6	21.7	21.8	21.9	22.0	22.1	22.2
1.356	22.3	22.4	22.5	22.6	22.7	22.8	22.9	23.0	23.1	23.2
1.357	23.3	23.4	23.5	23.6	23.7	23.8	23.9	24.0	24.1	24.2
1.358	24.3	24.4	24.5	24.6	24.7	24.8	24.9	25.0	25.1	25.2
1.359	25.3	25.4	25.5	25.6	25.7	25.8	25.9	26.0	26.1	26.2
1.360	26.3	26.4	26.5	26.6	26.7	26.8	26.9	27.0	27.1	27.3
1.361	27.4	27.5	27.6	27.7	27.8	27.9	28.0	28.1	28.2	28.3
1.362	28.4	28.5	28.6	28.7	28.8	28.9	29.0	29.1	29.2	29.3
1.363	29.4	29.5	29.6	29.7	29.8	29.9	30.0	30.1	30.2	30.3
1.364	30.4	30.5	30.6	30.7	30.8	31.0	31.1	31.2	31.3	31.4
1.365	31.5	31.6	31.7	31.8	31.9	32.0	32.1	32.2	32.3	32.4
1.366	32.5	32.7	32.8	32.9	33.0	33.1	33.2	33.3	33.4	33.5
1.367	33.6	33.7	33.8	33.9	34.0	34.2	34.3	34.4	34.5	34.6
1.368	34.7	34.8	34.9	35.0	35.1	35.2	35.3	35.4	35.5	35.6
1.369	35.7	35.8	36.0	36.1	36.2	36.3	36.4	36.5	36.6	36.7
1.370	36.8	36.9	37.0	37.1	37.2	37.3	37.4	37.6	37.7	37.8
1.371	37.9	38.0	38.1	38.2	38.3	38.4	38.5	38.6	38.7	38.8
1.372	38.9	39.0	39.2	39.3	39.4	39.5	39.6	39.7	39.8	39.9
1.373	40.0	40.1	40.2	40.3	40.4	40.5	40.7	40.8	40.9	41.0
1.374	41.1	41.2	41.3	41.4	41.5	41.6	41.8	41.9	42.0	42.1
1.375	42.2	42.3	42.4	42.5	42.6	42.7	42.8	42.9	43.0	43.1
1.376	43.2	43.3	43.4	43.6	43.7	43.8	43.9	44.0	44.1	44.2
1.377	44.3	44.4	44.6	44.7	44.8	44.9	45.0	45.1	45.2	45.3
1.378	45.4	45.6	45.7	45.8	45.9	46.0	46.1	46.2	46.3	46.4
1.379	46.6	46.7	46.8	46.9	47.0	47.1	47.2	47.3	47.4	47.6



Refractive Index, $n_D$ .	Fourth Decimal of $n_D$ .									
	0	1	2	3	4	5	6	7	8	9
	Scale Readings.									
I. 380	47.7	47.8	47.9	48.0	48.1	48.2	48.3	48.4	48.6	48.7
I. 381	48.8	48.9	49.0	49.1	49.2	49.3	49.4	49.6	49.7	49.8
I. 382	49.9	50.0	50.1	50.2	50.3	50.4	50.6	50.7	50.8	50.9
I. 383	51.0	51.1	51.2	51.3	51.4	51.6	51.7	51.8	51.9	52.0
I. 384	52.1	52.2	52.3	52.4	52.6	52.7	52.8	52.9	53.0	53.1
I. 385	53.2	53.3	53.4	53.6	53.7	53.8	53.9	54.0	54.1	54.2
I. 386	54.3	54.4	54.6	54.7	54.8	54.9	55.0	55.1	55.2	55.3
I. 387	55.4	55.6	55.7	55.8	55.9	56.0	56.1	56.2	56.3	56.5
I. 388	56.6	56.7	56.8	56.9	57.1	57.2	57.3	57.4	57.6	57.7
I. 389	57.8	57.9	58.0	58.1	58.2	58.3	58.4	58.6	58.7	58.8
I. 390	58.9	59.0	59.1	59.2	59.4	59.5	59.6	59.8	59.9	60.0
I. 391	60.1	60.2	60.3	60.4	60.6	60.7	60.8	60.9	61.0	61.1
I. 392	61.3	61.4	61.5	61.6	61.8	61.9	62.0	62.1	62.2	62.3
I. 393	62.4	62.6	62.7	62.8	62.9	63.0	63.2	63.3	63.4	63.5
I. 394	63.6	63.8	63.9	64.0	64.1	64.2	64.4	64.5	64.6	64.7
I. 395	64.8	65.0	65.1	65.2	65.3	65.4	65.6	65.7	65.8	65.9
I. 396	66.0	66.2	66.3	66.4	66.5	66.6	66.8	66.9	67.0	67.1
I. 397	67.2	67.4	67.5	67.6	67.7	67.8	67.9	68.1	68.2	68.3
I. 398	68.4	68.6	68.7	68.8	68.9	69.0	69.1	69.3	69.4	69.5
I. 399	69.6	69.8	69.9	70.0	70.1	70.2	70.4	70.5	70.6	70.8
I. 400	70.9	71.0	71.1	71.2	71.4	71.5	71.6	71.8	71.9	72.0
I. 401	72.1	72.2	72.4	72.5	72.6	72.8	72.9	73.0	73.1	73.2
I. 402	73.4	73.5	73.6	73.8	73.9	74.0	74.1	74.2	74.4	74.5
I. 403	74.6	74.8	74.9	75.0	75.1	75.2	75.4	75.5	75.6	75.8
I. 404	75.9	76.0	76.1	76.2	76.4	76.5	76.6	76.8	76.9	77.0
I. 405	77.1	77.2	77.4	77.5	77.7	77.8	77.9	78.1	78.2	78.3
I. 406	78.5	78.6	78.7	78.8	79.0	79.1	79.2	79.4	79.5	79.6
I. 407	79.8	79.9	80.0	80.1	80.2	80.4	80.5	80.6	80.8	80.9
I. 408	81.0	81.1	81.2	81.4	81.5	81.6	81.7	81.9	82.0	82.1
I. 409	82.3	82.4	82.5	82.6	82.8	82.9	83.0	83.2	83.3	83.4
I. 410	83.6	83.7	83.8	84.0	84.1	84.2	84.4	84.5	84.6	84.8
I. 411	84.9	85.0	85.2	85.3	85.4	85.5	85.6	85.7	85.9	86.1
I. 412	86.2	86.3	86.5	86.6	86.7	86.9	87.0	87.1	87.3	87.4
I. 413	87.5	87.7	87.8	87.9	88.1	88.2	88.3	88.5	88.6	88.7
I. 414	88.9	89.0	89.1	89.3	89.4	89.6	89.7	89.9	90.0	90.1
I. 415	90.2	90.4	90.5	90.6	90.8	90.9	91.0	91.2	91.3	91.5
I. 416	91.6	91.7	91.9	92.0	92.					



**DETERMINATION OF PROTEINS.**—For determination of the total nitrogen in milk, 5 cc. are measured direct into a Kjeldahl digestion-flask, or a known weight from a weighing-bottle may be used, and the regular Gunning method is employed as described on page 69, proceeding with the digestion at once without evaporation.

The total nitrogen, multiplied by 6.38, gives the total proteins. By many the old factor of 6.25 is still employed, but in view of the fact that both casein and albumin have been found to contain 15.7% of nitrogen, there would seem to be the best reasons for employing 6.38 as a factor  $\left(\frac{100}{15.7}\right)$ .

**Ritthausen's Method.**—Ten grams of milk are measured into a beaker and diluted with water to about 100 cc. Five cc. of a solution of copper sulphate (strength of Fehling's copper solution, 34.64 grams  $\text{CuSO}_4$  in 500 cc. of water) are added and the mixture stirred. A solution of sodium hydroxide (25 grams to the liter) is added cautiously a little at a time, till the liquid is nearly, but not quite neutral, avoiding an excess of alkali, as this would prevent the complete precipitation of the proteins. Allow the precipitate to settle, and pour off the supernatant liquid through a weighed filter, previously dried at  $130^\circ \text{C}$ . Wash a number of times by decantation, and transfer the precipitate to the filter, being careful to remove the portions adhering to the sides of the beaker with a rubber-tipped rod. Wash thoroughly with water, and drain dry, after which the precipitate is washed with strong alcohol, dried, extracted with ether, preferably in a Soxhlet extractor, and then transferred on the filter to the oven, dried at  $130^\circ \text{C}$ ., and weighed. The filter and precipitate are then burnt to an ash in a porcelain crucible, and the weight of the residue subtracted from the first weight gives that of the proteins.

Richmond\* recommends modifying this process to the extent of neutralizing the milk, using phenolphthalein as an indicator, before adding the copper sulphate solution, and using only 2.5 cc. of the latter.

**Determination of Casein.**—*Van Slyke Method.*†—Ten grams of the milk sample are placed in a beaker, and made up with water to about 100 cc. at  $40^\circ$  to  $42^\circ \text{C}$ . One and one-half cc. of a 10% solution (by weight) of acetic acid are added, the mixture stirred, warmed to the above temperature, and allowed to stand for from three to five minutes, till a floc-

\* Dairy Chemistry, London, 1914, p. 127.

† U. S. Dept. of Agric., Div. of Chem., Bul. 43, p. 189; Bul. 51, p. 108.



culent precipitate separates, leaving a clear supernatant liquid. Decant upon a filter, wash with cold water two or three times by decantation, and finally transfer the whole of the precipitate to the filter, and, after filtering, wash two or three times. The filtrate should be clear or nearly so. If not, it can generally be made so by repeated filtrations, and the washing done afterwards. The filter containing the washed precipitate is transferred to the Kjeldahl digestion-flask and the nitrogen obtained by the Gunning process.  $N \times 6.38 = \text{casein}$ .

**Determination of Albumin.**—*Van Slyke Method.*—To the filtrate from the direct determination of casein by the acetic acid method as described in the preceding section, exactly neutralized with sodium hydroxide, 0.3 cc. of a 10% solution of acetic acid is added, and the mixture is boiled till the albumin is completely precipitated. The precipitate is collected on a filter and washed, the nitrogen being determined in the precipitate, and the factor 6.38 used in calculating the albumin therefrom.

*Leffman and Beam Modification of the Sebelien Method.\**—Owing to the tedious processes of washing and filtering incidental to the above method for determining casein, the following is suggested. Mix 10 cc. of the milk with saturated magnesium sulphate solution, and saturate the mixture with the powdered salt. Make up to 100 cc. with the same solution, mix, and allow the precipitate to settle, leaving a clear, supernatant layer. Withdraw as much as possible of the clear portion by a pipette and filter through a dry filter. Precipitate the albumin in an aliquot portion by Almén's reagent (4 grams tannin in 1900 cc. of 50% alcohol mixed with 8 cc. of 25% acetic acid), filter, wash, and determine nitrogen.  $N \times 6.38 = \text{albumin}$ .

To obtain the casein, subtract the albumin from the total protein.

**Determination of Nitrogen as Caseoses, Amino-compounds, Peptones, and Ammonia.**—Van Slyke† proceeds as follows: The filtrate from the determination of the albumin, as above, is heated to 70° C., 1 cc. of 50% sulphuric acid is first added, and afterwards chemically pure zinc sulphate to saturation. The mixture is allowed to stand at 70° until the caseoses separate out and settle. Cool, filter, wash with a saturated zinc sulphate solution slightly acidified with sulphuric acid, and determine the nitrogen of the caseoses in the precipitate.

*For Amino-compounds and Ammonia* treat 50 grams of the milk in a

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\* Allen's Commercial Organic Analysis, 4th Ed., Phila., 1914, 8, p. 156.

† N. Y. Exp. Sta. Bul. 215, p. 102.



250-cc. graduated flask with 1 gram sodium chloride and a 12% solution of tannin, added drop by drop till no further precipitate is formed. Dilute to the 250-cc. mark, shake, and filter. Determine the nitrogen in 50 cc. of the filtrate, the result being the combined nitrogen of the amino-compounds and ammonia.

Distil with magnesium oxide 100 cc. of the filtrate from the tannin salt solution, receiving the distillate in a standardized acid, and titrating in the usual way for the ammonia.

Calculate the nitrogen of the *peptones* by subtracting from the total nitrogen that due to all other forms.

Van Slyke has furnished the following unpublished analysis of a sample of milk three months old, kept under antiseptic conditions by chloroform.

Per Cent Total N.	Per Cent Sol. Nitrogen.	Per Cent N as Paranuclein, Caseoses, and Peptones.	Per Cent N as Amino- compounds.
0.561	0.099	0.074	0.025

**DETERMINATION OF MILK SUGAR.**—If a polariscope is available, the sugar of milk can most readily and conveniently be determined by optical methods. In the absence of a polariscope, the reducing power of milk sugar on copper salts may be utilized quite accurately in determining the sugar, using either volumetric or gravimetric methods as desired.

**Determination by Polarization.**—*Wiley Method.\**—1. *Reagents.*—*Mercuric Nitrate.*—This solution is prepared by dissolving metallic mercury in twice its weight of nitric acid of specific gravity 1.42, and adding to the solution an equal volume of water. One cc. of this reagent will be found sufficient to precipitate the proteins and fat completely from 65 grams of milk, but if more is employed the result of the analysis is not affected.

*Mercuric Iodide Solution.*—33.2 grams of potassium iodide are mixed with 13.5 grams of mercuric chloride, 20 cc. of acetic acid, and 64c cc. of water.

*Subacetate of Lead Solution, U. S. P.* See page 610.

**NOTES.**—For the Laurent polariscope, in which the normal weight for sucrose is 16.19 grams, the corresponding normal weight for lac-

\* Am. Chem. Jour., 6, 1884, p. 289.



tose is 20.496, while for the Soleil-Ventzke instrument, in which the sucrose normal weight is 26.048 grams, the corresponding lactose normal weight is 32.975.\*

It is customary to employ three times the normal weight of milk in the case of the Laurent instrument (viz., 61.48 grams) and twice the normal weight in the case of the Soleil-Ventzke (viz., 65.95 grams).

As it is more convenient to measure the milk than to weigh it, and as the volume varies with the specific gravity, the following table is useful, showing the quantity to be measured in any case, having first determined the specific gravity.

Specific Gravity.	Volume of Milk to be Used.	
	For Polariscopes of which the Sucrose Normal Weight is 16.19 Grams.	For Polariscopes of which the Sucrose Normal Weight is 26.048 Grams.
1.024	60.0 cc.	64.4 cc.
1.026	59.9 cc.	64.3 cc.
1.028	59.8 cc.	64.15 cc.
1.030	59.7 cc.	64.0 cc.
1.032	59.6 cc.	63.9 cc.
1.034	59.5 cc.	63.8 cc.
1.035	59.35 cc.	63.7 cc.

For ordinary work it is sufficiently close to have a pipette graduated to deliver 59.7 cc. if the Laurent instrument is used, and 64 cc. for the Soleil-Ventzke.

2. *Process.*—Measure as above, the equivalent of 61.48 grams of the milk for the Laurent, or 65.95 grams for the Soleil-Ventzke, instrument into a 100-cc. graduated flask, add, in order to clarify, 2 cc. of acid nitrate of mercury solution, or 30 cc. of mercuric iodide solution, or 10 cc. of lead subacetate solution. Shake gently and fill to the mark with water, then add from a pipette 2.5 cc. of water to make up for the volume of the precipitated proteins and fat, insuring 100 cc. of sugar solution. Shake thoroughly, filter through a dry paper, and polarize the filtrate, which must be perfectly clear, in a 200-mm. tube. Divide the reading by 3 for the Laurent and by 2 for the Soleil-Ventzke instrument. The quotient is the percentage of lactose.

3. *Allowance for the Volume of the Precipitate.*—This of course varies with the content in proteins, and fat, and while the above allowance gives

\*  $[\alpha]_D$  for lactose = 52.53,  $[\alpha]_D$  for sucrose = 66.5, hence for the Laurent instrument

52.53 : 66.5 :: 16.19 : 20.496,

and for the Soleil-Ventzke instrument 52.53 : 66.5 :: 26.048 : 32.975.



in most cases sufficiently close results, it is not exact. Leffmann\* recommends that the amount of water to be added above 100 cc. be calculated in each case from the percentage of proteins and fat previously found by analysis, multiplying the actual weight of the fat in grams in the sample taken by 1.075, and the weight of proteins by 0.8, the sum of the two results being the volume in cubic centimeters occupied by the precipitate.

All calculations are avoided by employing the double-dilution method, which is to be recommended when very particular results are required.

**Wiley and Ewell's Double-dilution Method.**†—Two flasks are employed graduated at 100 and 200 cc. respectively, into each of which are introduced 65.95 grams of milk, if the Soleil-Ventzke instrument is used (or 61.48 grams in case the Laurent is used) and 4 cc. of the mercuric nitrate solution are added, both flasks being filled to the mark and shaken. The contents are filtered and the polarization is made in each case in a 400-mm. tube.

The second reading (that of the more dilute solution) is multiplied by 2, and the product subtracted from the first reading; the remainder is then multiplied by 2, and the product subtracted from the first reading (that of the stronger or 100 cc. solution). The result is the corrected reading, which, divided by 4, gives the exact per cent of milk sugar in the sample. This method depends on the fact that within ordinary limits the polarizations of two solutions of the same substance are inversely proportional to their volumes.

**DETERMINATION OF MILK SUGAR BY FEHLING'S SOLUTION.**—Twenty-five grams of the milk (24.2 cc.) are transferred to a 250-cc. flask, 0.5 cc. of a 30% solution of acetic acid are added and the contents well shaken. After standing for a few minutes, about 100 cc. of boiling water are run in, the contents again shaken, 25 cc. of alumina cream are next added, the flask shaken once more, and set aside for at least ten minutes. The supernatant liquid is then poured upon a previously wetted ribbed filter, and finally the whole contents of the flask are brought thereon, and the filtrate and washings made up to 250 cc. The filtrate must be perfectly clear. The milk sugar in a solution thus precipitated would ordinarily not exceed  $\frac{1}{2}$  of 1 per cent. Scheibe‡ after precipitating with copper sulphate, adds 2 cc. of saturated sodium fluoride solution to precipitate the lime which otherwise would cause an error of 0.10%.

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\* Milk and Milk Products, p. 38.

† Wiley's Agricultural Analysis, p. 278; Analyst, 21, 1896, p. 182.

‡ Zeits. Anal. Chem., 40, 1901, p. 1.



**Volumetric Fehling Process.**—From a burette containing the clear milk sugar solution above prepared, run a measured volume into the boiling Fehling liquor containing 5 cc. each of copper and alkali solution till sufficient has been introduced to completely reduce the copper, conducting the operation in the manner described in detail on page 615.

As 0.067 gram of milk sugar will reduce 10 cc. of Fehling solution (see p. 616), it follows that the number of cubic centimeters of sugar containing solution required for making the test (using preferably the average of several determinations) will contain 0.067 gram of milk sugar, from which the percentage is readily computed. Thus if 16 cc. of the milk sugar solution are necessary to reduce the copper, then 16 cc. contain 0.067 gram milk sugar.

250 cc. of solution contain 25 grams milk,

1 cc. " " " 0.1 " "

16 cc. " " " 1.6 " "

and 1.6 grams milk contain 0.067 gram milk sugar. Therefore the

sample contains  $\frac{.067 \times 100}{1.6} = 4.19\%$ .

**Gravimetric Fehling Processes.**—*O'Sullivan-Defren Method.*—Twenty-five cc. of the above milk sugar solution are added to the hot mixture of 15 cc. each of Fehling copper and alkali solutions and 50 cc. water, prepared as directed on page 615, and the test carried out in accordance with the details given on page 618. The weight of the cupric oxide,  $\text{CuO}$ , as formed, may be roughly calculated to anhydrous milk sugar by multiplying by 0.6024.

For more accurate results, however, the Defren table, page 619, should be used.

*Soxhlet's Method.\**—Twenty-five cc. of milk are diluted with 400 cc. of water in a half-liter graduated flask and 10 cc. of Fehling's copper solution are added. Then 8.8 cc. of half-normal sodium hydroxide are run in, or a sufficient quantity to nearly but not quite neutralize, the solution being still slightly acid. The flask is filled to the mark, shaken, and the contents filtered, using a dry filter.

One hundred cc. of the filtrate are added to 50 cc. of the mixed Fehling solution, which is boiled briskly in a beaker (using 25 cc. each of the copper and alkali solution). After boiling for six minutes, filter rapidly through a Gooch crucible provided with a layer of asbestos as described on page 618, and wash with boiling water till free from alkali. The asbestos

\* U. S. Dept. of Agric., Bur. of Chem., Bull. 46, p. 41; Bul. 107 (rev.), p. 119.



film with the adhering cuprous oxide is washed into a beaker by hot dilute nitric acid, and after complete solution of the copper is assured, it is again filtered and washed with hot water till a clean solution containing all the copper is obtained. Add 10 cc. of dilute sulphuric acid (containing 200 cc. of sulphuric acid, specific gravity 1.84 per liter) and evaporate on the steam-bath till the copper has largely crystallized, then carefully continue the heating over a hot plate till the nitric acid is driven out, as evidenced by the white fumes of sulphuric. Add 8 or 10 drops nitric acid (specific gravity 1.42) and rinse into a *very clean* tared platinum dish of about 100 cc. capacity, in which the copper is deposited by electrolysis. See page 634.

The weight of milk sugar is determined from that of copper found, from the table on page 139.

If the apparatus for the determination of the copper by the electrolytic method is not at hand, the cuprous oxide may be weighed directly in the Gooch crucible. In order to facilitate drying, it should be washed successively with 10 cc. of alcohol, and 10 cc. of ether, after which it is dried thirty minutes in a water-oven at 100° C., cooled, and weighed. The weight of copper is obtained from the weight of the cuprous oxide by the use of the factor 0.8883.

*Munson and Walker Method.*—The milk sugar solution is prepared as in Soxhlet's method. For details as to the copper reduction process see page 622.

**Relation between Specific Gravity, Fat, and Total Solids of Milk.**—The close relationship existing between these factors has long been known, and many formulæ have been devised, whereby, if two of them are known, the third may be computed with considerable approach to accuracy. The specific gravity and the fat are very readily determined by any dairyman, by the aid of a lactometer and the Babcock apparatus. The total solids are ascertained with more difficulty, since the use of more involved and costly apparatus is necessary, besides considerable technical skill. It is therefore common for producers to calculate the total solids from the fat and specific gravity, using one of the many tables prepared for the purpose, based on some one of the best accepted formulæ. The total solids can thus be calculated to within two or three tenths of a per cent.

The two most commonly used formulæ for this purpose are those of *Hehner and Richmond* in England, and *Babcock* in the United States. *Hehner and Richmond's* formula is

$$T = 0.25S + 1.2F + 0.14,$$



SOXHLET-WEIN TABLE FOR THE DETERMINATION OF LACTOSE.

Milli-grams of Cop-per.	Milli-grams of Lac-tose.	Milli-grams of Cop-per.	Milli-grams of Lac-tose.	Milli-grams of Cop-per.	Milli-grams of Lac-tose.	Milli-grams of Cop-per.	Milli-grams of Lac-tose.	Milli-grams of Cop-per.	Milli-grams of Lac-tose.
100	71.6	161	117.1	221	162.7	281	209.1	341	256.5
101	72.4	162	117.9	222	163.4	282	209.9	342	257.4
102	73.1	163	118.6	223	164.2	283	210.7	343	258.2
103	73.8	164	119.4	224	164.9	284	211.5	344	259.0
104	74.6	165	120.2	225	165.7	285	212.3	345	259.8
105	75.3	166	120.9	226	166.4	286	213.1	346	260.6
106	76.1	167	121.7	227	167.2	287	213.9	347	261.4
107	76.8	168	122.4	228	167.9	288	214.7	348	262.3
108	77.6	169	123.2	229	168.6	289	215.5	349	263.1
109	78.3	170	123.9	230	169.4	290	216.3	350	263.9
110	79.0	171	124.7	231	170.1	291	217.1	351	264.7
111	79.8	172	125.5	232	170.9	292	217.9	352	265.5
112	80.5	173	126.2	233	171.6	293	218.7	353	266.3
113	81.3	174	127.0	234	172.4	294	219.5	354	267.2
114	82.0	175	127.8	235	173.1	295	220.3	355	268.0
115	82.7	176	128.5	236	173.9	296	221.1	356	268.8
116	83.5	177	129.3	237	174.6	297	221.9	357	269.6
117	84.2	178	130.1	238	175.4	298	222.7	358	270.4
118	85.0	179	130.8	239	176.2	299	223.5	359	271.2
119	85.7	180	131.6	240	176.9	300	224.4	360	272.1
120	86.4	181	132.4	241	177.7	301	225.2	361	272.9
121	87.2	182	133.1	242	178.5	302	225.9	362	273.7
122	87.9	183	133.9	243	179.3	303	226.7	363	274.5
123	88.7	184	134.7	244	180.1	304	227.5	364	275.3
124	89.4	185	135.4	245	180.8	305	228.3	365	276.2
125	90.1	186	136.2	246	181.6	306	229.1	366	277.1
126	90.9	187	137.0	247	182.4	307	229.8	367	277.9
127	91.6	188	137.7	248	183.2	308	230.6	368	278.8
128	92.4	189	138.5	249	184.0	309	231.4	369	279.6
129	93.1	190	139.3	250	184.8	310	232.2	370	280.5
130	93.8	191	140.0	251	185.5	311	232.9	371	281.4
131	94.6	192	140.8	252	186.3	312	233.7	372	282.2
132	95.3	193	141.6	253	187.1	313	234.5	373	283.1
133	96.1	194	142.3	254	187.9	314	235.3	374	283.9
134	96.9	195	143.1	255	188.7	315	236.1	375	284.8
135	97.6	196	143.9	256	189.4	316	236.8	376	285.7
136	98.3	197	144.6	257	190.2	317	237.6	377	286.5
137	99.1	198	145.4	258	191.0	318	238.4	378	287.4
138	99.8	199	146.2	259	191.8	319	239.2	379	288.2
139	100.5	200	146.9	260	192.5	320	240.0	380	289.1
140	101.3	201	147.7	261	193.3	321	240.7	381	289.9
141	102.0	202	148.5	262	194.1	322	241.5	382	290.8
142	102.8	203	149.2	263	194.9	323	242.3	383	291.7
143	103.5	204	150.0	264	195.7	324	243.1	384	292.5
144	104.3	205	150.7	265	196.4	325	243.9	385	293.4
145	105.1	206	151.5	266	197.2	326	244.6	386	294.2
146	105.8	207	152.2	267	198.0	327	245.4	387	295.1
147	106.6	208	153.0	268	198.3	328	246.2	388	296.0
148	107.3	209	153.7	269	199.5	329	247.0	389	296.8
149	108.1	210	154.5	270	200.3	330	247.7	390	297.7
150	108.8	211	155.2	271	201.1	331	248.5	391	298.5
151	109.6	212	156.0	272	201.9	332	249.2	392	299.4
152	110.3	213	156.7	273	202.7	333	250.0	393	300.3
153	111.1	214	157.5	274	203.5	334	250.8	394	301.1
154	111.9	215	158.2	275	204.3	335	251.6	395	302.0
155	112.6	216	159.0	276	205.1	336	252.5	396	302.8
156	113.4	217	159.7	277	205.9	337	253.3	397	303.7
157	114.1	218	160.4	278	206.7	338	254.1	398	304.6
158	114.9	219	161.2	279	207.5	339	254.9	399	305.4
159	115.6	220	161.9	280	208.3	340	255.7	400	306.3
160	116.4								



where  $T$  is the per cent of total solids,  $S$  the lactometer reading, and  $F$  the fat. An ingenious instrument known as Richmond's milk-scale (Fig. 48) is useful in making the calculation, instead of employing either the formula or a table. This is constructed on the principle of the slide rule, and by its use the specific gravity may be corrected to the proper temperature, and the solids calculated from the fat and specific gravity.

Babcock's formula for solids not fat is as follows:

$$\text{Solids not fat} = \left( \frac{100S - FS}{100 - 1.0753FS} - 1 \right) (100 - F) 2.5,$$

$S$  being the specific gravity, and  $F$  the percentage of fat. On this formula he has prepared a table\* by means of which one may calculate solids not fat agreeing quite closely with results obtained by gravimetric analysis.† The table on page 141 has been recomputed and enlarged from that of Babcock, so as to express results in total solids rather than solids not fat.

**Calculation of Proteins.**—Van Slyke's ‡ formula for calculating proteins ( $P$ ) from the fat ( $F$ ) is:

$$P = (F - 3) \times 0.4 + 2.8.$$

Olsen § has devised the following formula for calculating proteins from total solids ( $TS$ ):

$$P = TS - \frac{TS}{1.34}.$$

Approximately  $0.8 \times \text{proteins} = \text{casein}$ .

The proteins being thus calculated, the sugar may be computed by difference. These calculations, while only approximate, give quite satisfactory results for normal, healthy milk, especially from herds.

**Determination of Acidity.**—While milk is still fresh, i.e., before it has begun to undergo lactic fermentation, it will show an acid reaction, which is sometimes expressed in terms of lactic acid. In view of the fact that

\* U. S. Dept. of Agric., Div. of Chem., Bul. 47, p. 123; Bul. 107 (rev.) p. 225.

† For approximate work Babcock has suggested the following simplified formulæ: Solids not fat  $= 0.25G + 0.2F$  and total solids  $= 0.25G + 1.2F$ ,  $G$  being the lactometer reading and  $F$  the fat.

‡ Jour. Am. Chem. Soc. 30, 1908, p. 1182.

§ Jour. Ind. and Eng. Chem., 1, 1909, p. 253.

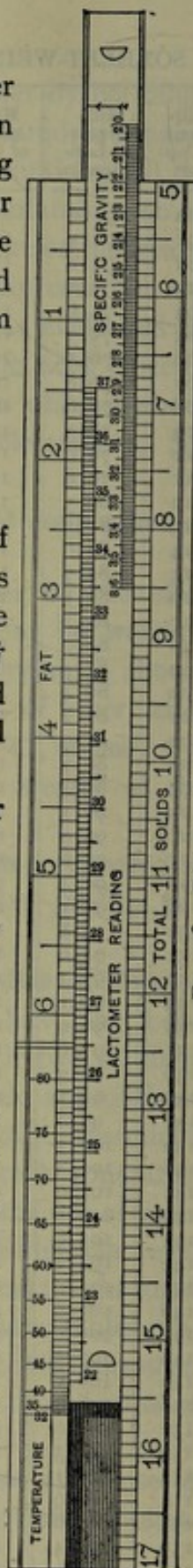


FIG. 48.—Richmond's Milk-scale.



TABLE SHOWING PER CENT OF TOTAL SOLIDS IN MILK CORRESPONDING TO QUEVENNE LACTOMETER READINGS\* AND PER CENT OF FAT.†

Per Cent of Fat.	Lactometer Reading at 15.5° C.														
	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
0.0	5.50	5.75	6.00	6.25	6.50	6.75	7.00	7.25	7.50	7.75	8.00	8.25	8.50	8.75	9.00
0.1	5.62	5.87	6.12	6.37	6.62	6.87	7.12	7.37	7.62	7.87	8.12	8.37	8.62	8.87	9.12
0.2	5.74	5.99	6.24	6.49	6.74	6.99	7.24	7.49	7.74	7.99	8.24	8.49	8.74	8.99	9.24
0.3	5.86	6.11	6.36	6.61	6.86	7.11	7.36	7.61	7.86	8.11	8.36	8.61	8.86	9.11	9.36
0.4	5.98	6.23	6.48	6.73	6.98	7.23	7.48	7.73	7.98	8.23	8.48	8.73	8.98	9.23	9.48
0.5	6.10	6.35	6.60	6.85	7.10	7.35	7.60	7.85	8.10	8.35	8.60	8.85	9.10	9.35	9.60
0.6	6.22	6.47	6.72	6.97	7.22	7.47	7.72	7.97	8.22	8.47	8.72	8.97	9.22	9.47	9.72
0.7	6.34	6.59	6.84	7.09	7.34	7.59	7.84	8.09	8.34	8.59	8.84	9.09	9.34	9.59	9.84
0.8	6.46	6.71	6.96	7.21	7.46	7.71	7.96	8.21	8.46	8.71	8.96	9.21	9.46	9.71	9.96
0.9	6.58	6.83	7.08	7.33	7.58	7.83	8.08	8.33	8.58	8.83	9.08	9.33	9.58	9.83	10.08
1.0	6.70	6.95	7.20	7.45	7.70	7.95	8.20	8.45	8.70	8.95	9.20	9.45	9.70	9.95	10.20
1.1	6.82	7.07	7.32	7.57	7.82	8.07	8.32	8.57	8.82	9.07	9.32	9.57	9.82	10.07	10.32
1.2	6.94	7.19	7.44	7.69	7.94	8.19	8.44	8.69	8.94	9.19	9.44	9.69	9.94	10.19	10.44
1.3	7.06	7.31	7.56	7.81	8.06	8.31	8.56	8.81	9.06	9.31	9.56	9.81	10.06	10.31	10.56
1.4	7.18	7.43	7.68	7.93	8.18	8.43	8.68	8.93	9.18	9.43	9.68	9.93	10.18	10.43	10.68
1.5	7.30	7.55	7.80	8.05	8.30	8.55	8.80	9.05	9.30	9.55	9.80	10.05	10.30	10.55	10.80
1.6	7.42	7.67	7.92	8.17	8.42	8.67	8.92	9.17	9.42	9.67	9.92	10.17	10.42	10.67	10.92
1.7	7.54	7.79	8.04	8.29	8.54	8.79	9.04	9.29	9.54	9.79	10.04	10.29	10.54	10.79	11.04
1.8	7.66	7.91	8.16	8.41	8.66	8.91	9.16	9.41	9.66	9.91	10.16	10.41	10.66	10.91	11.17
1.9	7.78	8.03	8.28	8.53	8.78	9.03	9.28	9.53	9.78	10.03	10.28	10.53	10.78	11.04	11.29
2.0	7.90	8.15	8.40	8.65	8.90	9.15	9.40	9.65	9.90	10.15	10.40	10.66	10.91	11.16	11.41
2.1	8.02	8.27	8.52	8.77	9.02	9.27	9.52	9.77	10.02	10.27	10.52	10.78	11.03	11.28	11.53
2.2	8.14	8.39	8.64	8.89	9.14	9.39	9.64	9.89	10.14	10.39	10.64	10.90	11.15	11.40	11.65
2.3	8.26	8.51	8.76	9.01	9.26	9.51	9.76	10.01	10.26	10.51	10.76	11.02	11.27	11.52	11.77
2.4	8.38	8.63	8.88	9.13	9.38	9.63	9.88	10.13	10.38	10.63	10.88	11.14	11.39	11.64	11.89
2.5	8.50	8.75	9.00	9.25	9.50	9.75	10.00	10.25	10.50	10.75	11.00	11.26	11.51	11.76	12.01
2.6	8.62	8.87	9.12	9.37	9.62	9.87	10.12	10.37	10.62	10.87	11.12	11.38	11.63	11.88	12.13
2.7	8.74	8.99	9.24	9.49	9.74	9.99	10.24	10.49	10.74	10.99	11.24	11.50	11.75	12.00	12.25
2.8	8.86	9.11	9.36	9.61	9.86	10.11	10.36	10.61	10.86	11.11	11.37	11.62	11.87	12.12	12.37
2.9	8.98	9.23	9.48	9.73	9.98	10.23	10.48	10.73	10.98	11.23	11.49	11.74	11.99	12.24	12.49
3.0	9.10	9.35	9.60	9.85	10.10	10.35	10.60	10.85	11.10	11.36	11.61	11.86	12.11	12.36	12.61
3.1	9.22	9.47	9.72	9.97	10.22	10.47	10.72	10.97	11.23	11.48	11.73	11.98	12.23	12.48	12.74
3.2	9.34	9.59	9.84	10.09	10.34	10.59	10.84	11.09	11.35	11.60	11.85	12.10	12.35	12.61	12.86
3.3	9.46	9.71	9.96	10.21	10.46	10.71	10.96	11.22	11.47	11.72	11.97	12.22	12.48	12.73	12.98
3.4	9.58	9.83	10.08	10.33	10.58	10.83	11.09	11.34	11.59	11.84	12.09	12.34	12.60	12.85	13.10
3.5	9.70	9.95	10.20	10.45	10.70	10.95	11.21	11.46	11.71	11.96	12.21	12.46	12.72	12.97	13.22
3.6	9.82	10.07	10.32	10.57	10.82	11.08	11.33	11.58	11.83	12.08	12.33	12.58	12.84	13.09	13.34
3.7	9.94	10.20	10.44	10.70	10.94	11.20	11.45	11.70	11.95	12.20	12.45	12.70	12.96	13.21	13.46
3.8	10.06	10.31	10.56	10.81	11.06	11.32	11.57	11.82	12.07	12.32	12.57	12.82	13.08	13.33	13.58
3.9	10.18	10.43	10.68	10.93	11.18	11.44	11.69	11.94	12.19	12.44	12.69	12.94	13.20	13.45	13.70
4.0	10.30	10.55	10.80	11.05	11.30	11.56	11.81	12.06	12.31	12.56	12.81	13.06	13.32	13.57	13.83
4.1	10.42	10.67	10.92	11.17	11.42	11.68	11.93	12.18	12.43	12.68	12.93	13.18	13.44	13.69	13.95
4.2	10.54	10.79	11.04	11.29	11.54	11.80	12.05	12.30	12.55	12.80	13.05	13.31	13.56	13.82	14.07
4.3	10.66	10.91	11.16	11.41	11.66	11.92	12.17	12.42	12.67	12.92	13.18	13.43	13.68	13.94	14.19
4.4	10.78	11.03	11.28	11.53	11.78	12.04	12.29	12.54	12.79	13.04	13.30	13.55	13.80	14.06	14.31
4.5	10.90	11.15	11.40	11.65	11.90	12.16	12.41	12.66	12.91	13.16	13.42	13.67	13.92	14.18	14.43
4.6	11.02	11.27	11.52	11.78	12.03	12.28	12.53	12.78	13.03	13.28	13.54	13.79	14.04	14.30	14.55
4.7	11.14	11.40	11.65	11.90	12.15	12.40	12.65	12.90	13.15	13.40	13.66	13.91	14.16	14.42	14.67
4.8	11.27	11.52	11.77	12.02	12.27	12.52	12.77	13.02	13.27	13.52	13.78	14.03	14.28	14.54	14.79
4.9	11.39	11.64	11.89	12.14	12.39	12.64	12.89	13.14	13.39	13.64	13.90	14.15	14.40	14.66	14.91
5.0	11.51	11.76	12.01	12.26	12.51	12.76	13.01	13.26	13.51	13.76	14.02	14.27	14.52	14.78	15.03
5.1	11.63	11.88	12.13	12.38	12.63	12.88	13.13	13.38	13.63	13.89	14.14	14.39	14.64	14.90	15.15
5.2	11.75	12.00	12.25	12.50	12.75	13.00	13.25	13.50	13.75	14.01	14.26	14.51	14.76	15.02	15.27
5.3	11.87	12.12	12.37	12.62	12.87	13.12	13.37	13.62	13.87	14.13	14.38	14.63	14.88	15.14	15.39
5.4	11.99	12.24	12.49	12.74	12.99	13.24	13.49	13.74	14.00	14.25	14.50	14.76	15.01	15.26	15.51
5.5	12.11	12.36	12.61	12.86	13.11	13.36	13.61	13.86	14.12	14.37	14.62	14.88	15.13	15.38	15.63
5.6	12.23	12.48	12.73	12.98	13.23	13.48	13.73	13.99	14.24	14.49	14.75	15.00	15.25	15.50	15.75
5.7	12.35	12.60	12.85	13.10	13.35	13.60	13.85	14.11	14.36	14.61	14.87	15.12	15.37	15.62	15.87
5.8	12.47	12.72	12.97	13.22	13.47	13.72	13.97	14.22	14.48	14.74	14.99	15.24	15.49	15.74	15.99
5.9	12.59	12.84	13.09	13.34	13.59	13.84	14.10	14.35	14.60	14.86	15.11	15.36	15.61	15.86	16.12
6.0	12.71	12.96	13.21	13.46	13.71	13.96	14.22	14.47	14.72	14.98	15.23	15.48	15.73	15.98	16.24

\*The lactometer reading is expressed in whole numbers for convenience. The true specific gravity corresponding to a given lactometer reading is obtained by writing 1.0 before the lactometer reading. Thus, 1.026 is the specific gravity corresponding to lactometer reading 26, etc.

†An. Rep. Mass. State Board of Health, 1901, p. 445. (Analyst's Reprint, p. 25.)



the acidity of "sweet" milk is due partly to the presence of acid phosphates and partly to dissolved carbonic acid in the milk, and not to lactic acid, which is probably absent, a better plan is to express the acidity in terms of the number of cubic centimeters of tenth-normal alkali necessary to neutralize a given quantity of the milk, either 25 or 50 cc., using phenolphthalein as an indicator. See also page 1033.

If it is desired to calculate the acidity in terms of lactic acid, multiply the number of cubic centimeters of tenth-normal alkali used by 0.897, and divide by the number of cubic centimeters of milk titrated, the result being the percentage of lactic acid.

#### MODIFIED MILK.

A comparison of the composition of cow's milk and human milk, as in the following table by Dr. Emmett Holt,\* shows very marked differences.

	Woman's Milk, Average.	Cow's Milk, Average.
Fat.....	4.00	3.50
Sugar.....	7.00	4.30
Proteins.....	1.50	4.00
Ash.....	0.20	0.70
Water.....	87.30	87.50

The per cent of fat in the two kinds of milk is nearly the same. There is, however, too little sugar and an excess of proteins and ash in the milk of the cow, assuming human milk as the ideal infant food, so that in basing a diet for infants on the basis of human milk considerable modification is necessary. Moreover, aside from the actual variation in the amount of ingredients, there are certain inherent differences in the character of the same ingredient, as found in the milk of the cow and in human milk. The proteins of cow's milk, are for instance, found to be much more difficult of digestion than those of woman's milk, and the same is probably true of the fat. Aside from the mere statement of a few of these differences, it is obviously beyond the scope of this work to discuss this phase of the subject in detail, reference being made, however, to such books as Dr. T. M. Rotch's "Pediatrics," and "Infancy and Childhood" by Dr. Emmett Holt, for full particulars. So great has been the demand by physicians for "modified milk" for infant feeding, that laboratories for this exclusive purpose have been established

\* "Infancy and Childhood."



in many of the larger cities, in which not only is milk prepared in accordance with certain fixed formulæ supposed to be adapted to average infants of varying age, but milk of any desired composition is prepared, in accordance with special prescriptions of physicians to apply to individual cases.

*Methods and Ingredients.*—The proteins and the ash in cow's milk are much higher than in human milk, and both are brought to the proper degree of reduction by diluting the milk with water. Milk sugar is increased by the addition of lactose, and the fat is increased or diminished by addition of cream or by skimming.

The dilution of cow's milk with a measured amount of water shows the following results on the proteins and ash:

	Cow's Milk.	Diluted Once.	Diluted Twice.	Diluted Three Times.	Diluted Four Times.
	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
Proteins. ....	4.00	2.00	1.33	1.00	0.80
Ash. ....	0.70	0.35	0.23	0.18	0.14

The ingredients commonly employed for modifying milk are (1) cream, containing 16% of fat, (2) centrifugally skimmed milk, otherwise known as "separator milk" from which the fat has been removed, (3) milk sugar, or a standard solution of milk sugar of, say, 20% strength, and (4) lime water. Unusual care should be taken in the selection of the milk supply to insure cleanness, purity, and freshness, as well as in the care of utensils, etc., used in the laboratory, which should in all cases be scrupulously clean. Samples prepared in accordance with a given formula or formulæ are pasteurized in separate bottles, or, if desired, sterilized, and after stoppering with cotton are kept on ice.

*Formulæ.*—It is obviously impossible to establish formulæ universally applicable even to healthy infants, but the following may be regarded as typical formulæ, representing the composition of modified milk to suit the needs of an average growing infant during its first year:

Period.	Fat.	Proteins.	Sugar.
	Per Cent	Per Cent	Per Cent
Third to fourteenth day. ....	2	0.6	6
Second to sixth week. ....	2.5	0.8	6
Sixth to eleventh week. ....	3	1.0	6
Eleventh week to fifth month..	3.5	1.5	7
Fifth to ninth month. ....	4	2	7
Ninth to twelfth month. ....	3.5	2.5	3.5



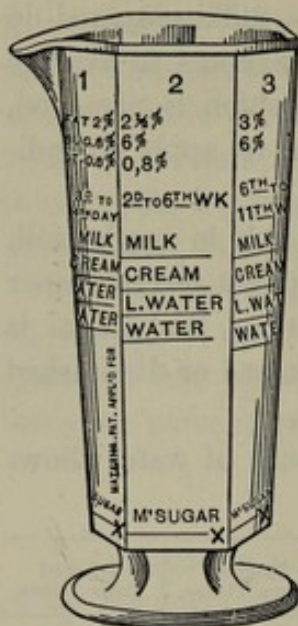


FIG. 49.—The "Materna"  
Graduate for Modifying  
Milk.

Milk according to the above formulæ can be very simply prepared by the aid of a specially made graduate known as the "Materna" and shown in Fig. 49.

*Sodium Citrate* has long been used in modifying milk in cases where the casein forms large lumps which pass through the body undigested. England\* attributes the beneficial action to the formation of sodium chloride with the hydrochloric acid of the stomach which influences the digestion of the protein. Van Slyke and Bosworth† more recently have observed that sodium citrate reacts with calcium caseinate, forming sodium caseinate or sodium-calcium caseinate. With 0.4 gram per 100 cc. no curdling takes place, with smaller amounts the curd is more or less soft, depending on the amount.

#### ADULTERATION OF MILK.

**Systems of Milk Inspection.**—A typical method of general food inspection has already been outlined (see pp. 6 and 8), which may easily be modified to include the inspection of milk in connection with other foods, or to provide for a system of milk inspection exclusively. In the examination of such a perishable food as milk, it has not been found practicable for the analyst to reserve for the benefit of the defendant a sealed sample, as in the case of other foods, but experience has shown it had best be made the duty of the collector or inspector to give a sealed sample of milk to the dealer, when the latter requests it at the time of taking the sample. For this purpose the collector is provided with small bottles and sealing paraphernalia, in addition to the tagged sample bottles or cans in which he collects the milk. The collector should use the same precautions for obtaining a perfectly fair representative sample as does the chemist in making the analysis, i.e., he should carefully pour the milk from the original container into an empty can or vessel and back again, before taking his sample.

Each sample is properly numbered by the collector in presence of the dealer, and the data as to the taking of the sample entered at once under

\* Jour. Amer. Med. Assn., 47, 1906, p. 1241.

† N. Y. State Agr. Exp. Sta. Tech. Bul., 34, 1914.



the proper number in the collector's book. If a sealed sample is given, it should bear the same number as the sample reserved for analysis, and a receipt should invariably be required from the dealer, as evidence that his request for a sealed sample has been complied with.

**Milk Standards Fixed by Law.**—In localities where a systematic form of milk inspection prevails, there is usually in force a statute fixing the legal standard for the total solids, and in many cases for the fat or for the solids exclusive of fat. In some states the statute is so drawn that any deviation from the legal standard constitutes an adulteration in the eye of the law, and hence the offender, who has such milk in his possession with intent to sell, is liable to the same fine as if he actually added water or a foreign substance to the milk.

In other states a distinction is made by the statute between milk that is simply below the legal standard of total solids, and milk containing actually added ingredients (water or otherwise), a much lighter fine being imposed for the former than for the latter offense. Where such a distinction prevails, it often becomes incumbent upon the analyst to show to the satisfaction of the court, in case of milk low in solids, whether or not the milk has been fraudulently watered after being drawn from the cow, it being well understood that cows may give milk below the standard.

The U. S. standards for some years in force fixed the minimum limits of 8.5% for solids not fat and 3.25% for fat, but more recently it has seemed impracticable to fix minimum limits that will apply to all sections and the state and municipal standards have been deemed sufficient. These latter are by no means uniform. The minimum limits for total solids range from 11 to 13% and for fat from 2.5 to 3.7 %.

Pure milk that is low in solids may owe its deficiency either to poor feeding, or to an inherent tendency on the part of the cow to give milk always of poor quality. Thus the Holstein cow, more than any other breed, is open to the charge of sometimes giving milk below the standard.\* That the Holstein cow is a favorite with the producer is by no means

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\* This statement should not be taken as condemning the Holstein, for it is true that cows of this breed often give milk far above the standard. A large number of samples of milk of known purity from Holsteins analyzed by the writer have been found to be of excellent quality. It is a curious fact that among the samples of known purity analyzed by the Massachusetts Board of Health, both the lowest and highest total solids on record came from a Holstein cow; the lowest recorded total solids in a "known purity" milk being 9.96 per cent. (seventh annual report of Massachusetts State Board of Health, Lunacy, and Charity, p. 160), and the highest being 17.06 per cent. (twenty-second annual report of the Massachusetts State Board of Health, p. 405).



strange, from the fact that no other breed can with moderate feeding be made to give so large a quantity of milk.

Wherever there is a statute fixing the standard for milk, it commonly provides also that the addition of any foreign substance whatsoever constitutes an adulteration.

**FORMS OF ADULTERATION.**—Milk is ordinarily adulterated (1) by watering, (2) by skimming, (3) by both watering and skimming, and (4) by the addition of one or more foreign ingredients.

**Watering and Skimming.**—The fact that milk is found below the standard of total solids, while more often due to an excess of water, may also be due to a deficiency in fat. In one case the milk is commonly termed watered, and in the other skimmed, using the terms broadly and not necessarily meaning actual and fraudulent tampering with the milk. In a third case, and almost invariably fraudulently, both watering and skimming may be found to have been practiced on the same sample. The analyst judges which of these causes have produced a milk low in solids, by a careful study of the relation between the percentages of total solids, fat, and solids not fat.

If both the total solids and solids not fat are abnormally low, and the proportion of fat to solids not fat about the same as, or higher than, in a normal milk, it is generally safe to assume that the sample has been watered; if both the total solids and the fat are well below the standard, and the solids not fat nearly normal, then the milk has undoubtedly been skimmed; if, in the third place, the total solids and the solids not fat are proportionally reduced below the standard, while the ratio of fat to solids not fat is abnormally small, it is safe to adjudge the milk to be low by reason of both skimming and watering.

**Milk of Known Purity.**—It is difficult to place the minimum figure for total solids, below which a milk sample may safely be pronounced by the analyst as fraudulently watered after having been drawn from the cow. Nearly nine hundred samples of milk of known purity from various breeds of cow, milked in the presence of an inspector, have been analyzed in the Department of Food and Drug Inspection of the Massachusetts State Board of Health, extending over a period of fifteen years, and among these are many samples from Holstein cows. It is extremely rare that any of these known purity samples have been found with total solids as low as 11%, though there are instances where total solids have run as low as 10%.



It is safe to assume that in the few cases on record showing less than 10.75% of total solids, either there was something decidedly abnormal about the health of the cow, or, through some accident, the cow was only partially milked, it being a well-known fact that the last fraction of the milking includes the larger percentage of fat. (See page 113.)

It is therefore nearly always safe to condemn a milk standing below 10.75 as fraudulently watered, if at the same time it has a proportionately high per cent of fat.

The average total solids of 800 samples of milk of known purity analyzed by the Massachusetts Board up to and including the year 1890 amounted to about 13 $\frac{1}{4}$ %.

It is rare indeed to find a herd of ten or more well-fed cows of mixed breeds in which the average milk of the herd falls below 12 $\frac{1}{2}$ % of solids.

The milk of forty-seven Holstein cows, examined in 1885, was found to contain an average of 12.51% of total solids, while the milk of eleven Jerseys examined in the same year averaged 14.02% of solids. These examples represent the two extremes commonly met with.

*Variation in Standard.*—In Massachusetts the law fixes a different standard for total solids in milk during the summer, or pasture-fed season, from that in force during the winter, or stall-fed period. From April to September inclusive the legal standard is 12% of total solids, of which 9% are solids not fat, and from October to March inclusive it is 13%, of which 9.3% are solids not fat. Bearing on the question of difference in normal quality of milk during the two periods, averages were taken of the milks collected by the corps of inspectors of the Massachusetts Board of Health during a month in each period, December and June being selected as most typical, and during these months all the samples were analyzed both for total solids and fat. The samples were taken from stores, milkmen, and producers, and represented as nearly as possible the milk as actually sold to the consumers. In making the averages, all samples of skimmed milk, as well as all samples standing above 17% of total solids, or under 10.75%, were deducted. The results are summarized as follows:



**QUALITY OF MILK SOLD IN MASSACHUSETTS CITIES AND TOWNS IN  
WINTER AND SUMMER.**

December.								
	Number of Samples.	Total Solids.			Fat.			Solids not Fat. Average Per Cent.
		Highest Per Cent.	Lowest Per Cent.	Average Per Cent.	Highest Per Cent.	Lowest Per Cent.	Average Per Cent.	
Cities. ....	403	16.86	10.88	13.21	8.50	2.40	4.37	8.74
Towns. ....	99	15.48	12.02	13.44	6.65	3.50	4.48	8.96
Summary....	502	16.86	10.88	13.32	8.50	2.40	4.42	8.85

June.								
	Number of Samples.	Total Solids.			Fat.			Solids not Fat. Average Per Cent.
		Highest Per Cent.	Lowest Per Cent.	Average Per Cent.	Highest Per Cent.	Lowest Per Cent.	Average Per Cent.	
Cities. ....	311	16.90	10.75	12.67	8.80	2.10	4.03	8.54
Towns. ....	76	15.71	10.99	12.63	7.10	3.00	4.09	8.54
Summary....	387	16.90	10.75	12.65	8.80	2.10	4.06	8.54

It is interesting to note that the average for total solids of the 889 samples examined for both months stands at just 13%, of which 4.24% is fat and 8.76 is solids not fat.

**Rapid Approximate Methods of Determining the Quality of Milk.—**

*The Lactometer.*—A rough idea of the quality of milk can be gained by the use of the lactometer (page 118), but, in view of the fact that a low specific gravity may be the result either of a watered milk or of a milk high in fat, good judgment is necessary in connection with its use. A milk of good standard quality should have a specific gravity between the limits of 1.027 and 1.033. A watered milk would run below the former and a skimmed milk above the latter figure, though a milk unusually rich in fat would also run low. It should easily be apparent from the taste and appearance of the milk, whether a low specific gravity is due to watering or unusual richness in fat. The fact should also be recognized, that a milk sample may be far below the standard, and still show a specific gravity within the limits of pure milk, by skillfully subjecting the milk to both skimming and watering.

*The Lactoscope.*—Feser's lactoscope (Fig. 50) gives an approximation to the amount of fat in milk, and its use, especially in connection with the lactometer, is of some value. This instrument consists of a graduated glass barrel, *a*, into the bottom of which is accurately fitted the stopper, bearing



a white glass cylinder, having black lines thereon. Four cc. of milk are introduced into the barrel by means of a pipette, *c*, and water is added with thorough mixing till the translucence of the mixture is sufficient to allow the black lines to be perceptible through it. The height of the level of milk and water in the barrel *a* is then read off, the number indicating roughly the percentage of fat in the sample.

As in the case of the lactometer, the purity of a milk sample cannot be positively established by the lactoscope alone. For instance, a watered milk abnormally high in fat would often be found to read within the limits of pure milk, when as a matter of fact its total solids would be below standard. By a careful comparison of the readings of both the lactoscope and lactometer, however, it is rare that a skimmed or watered sample could escape detection.

Thus, if the specific gravity by the lactometer is well within the limits of pure milk, and the fat, as shown by the lactoscope, is above  $3\frac{1}{4}$  per cent., the sample may be safely passed as pure, or as conforming to the standard.

A normal lactometer reading in connection with an abnormally *low* lactoscope reading shows both watering and skimming, and with an abnormally *high* lactoscope reading shows a milk high in fat, or a cream. With the lactoscope reading below three, and a low lactometer reading, watering is indicated. A lactometer reading above thirty-three, and a low lactoscope reading, indicate skimming.

*Heeren's Pioscope.*—This instrument consists of a hard-rubber disk, having in the center a shallow receptacle, the circular rim of which is raised above the level of the disk. Into this receptacle are introduced a few drops of the milk to be tested, and a circular cover-glass containing a number of variously tinted segments is placed over the receptacle, which spreads the milk out into a thin layer, and causes it to assume a tint against the black background that can be matched with one of the colors on the glass, the various tints indicating milks of various grades from the very poorest to rich cream. This test is at best a very rough one.

**Examination of the Milk Serum.**—*Detection of Added Water.*—This may often be detected by determining the specific gravity or the degree of refraction of the milk serum, since it has been found that under fixed conditions the composition of the milk serum, or clear "whey," is more constant than that of the milk itself. Hence any considerable amount of watering is manifest from the physical constants of the serum.

In using this method the analyst should carefully work out his own



standards for comparison, by personal experiment on milk of known composition to which varying amounts of water have been added using the same conditions for obtaining the serum in all cases.

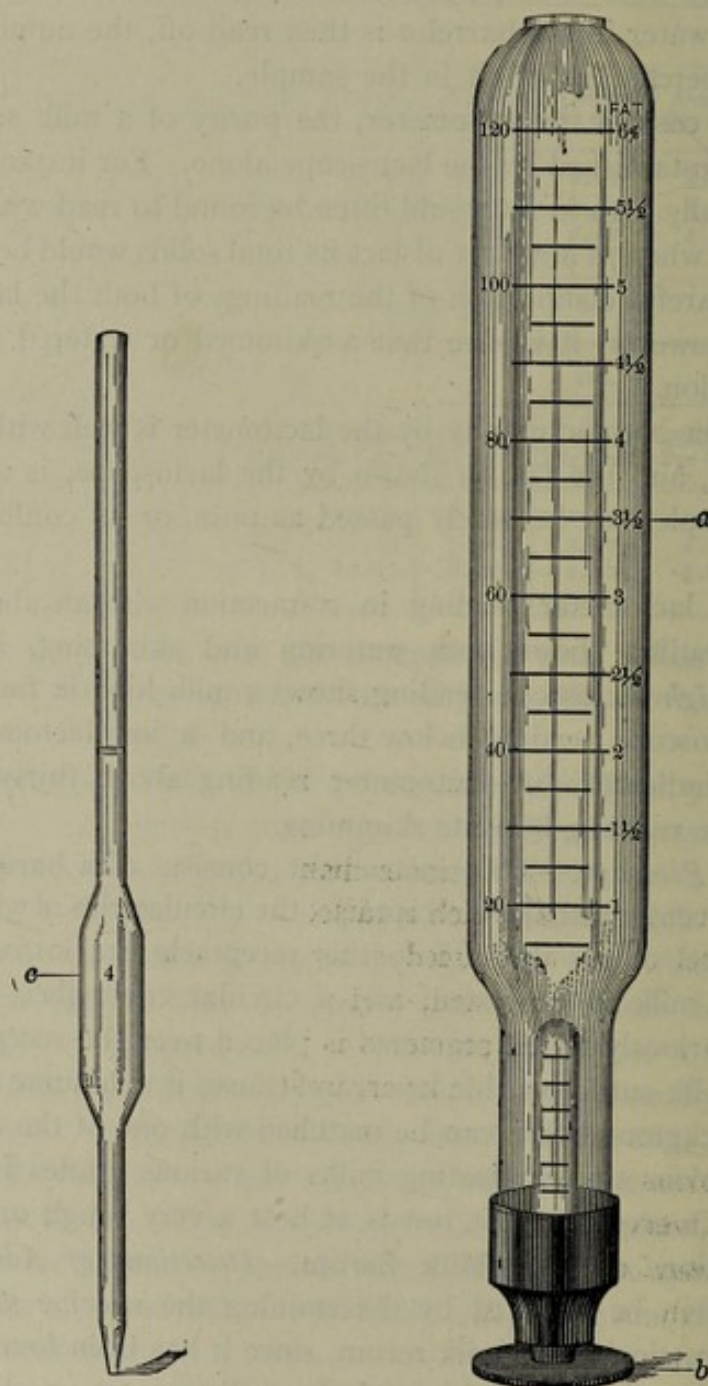


FIG. 50.—Feser's Lactoscope.

*Preparation of the Serum.*—In addition to natural souring, the following methods of preparing the serum have been described:



1. *Acetic Acid Method*.\*—To 100 cc. of the milk at about 20° C., add 2 cc. of 25% acetic acid (sp. gr. 1.035), mix well, and heat on a water-bath at 70° C. for 20 minutes. Cool 10 minutes in ice-water and filter.

2. *Calcium Chloride Method*.†—Mix thoroughly 90 cc. of the milk and 0.75 cc. of calcium chloride solution (sp. gr. 1.1375; refraction diluted 1 : 10, 26). Heat in a boiling water-bath under a reflux condenser for 15 minutes, cool to 20° C., mix without shaking, and filter.

3. *Asaprol Method*.‡—The reagent consists of 30 grams of asaprol and 55.89 grams of crystallized citric acid in 1 liter of water. If the refraction is not 36.3 at 20° C., add citric acid or water as required. Mix equal volumes of the milk and the reagent, shake well, and filter.

4. *Copper Sulphate Method*.§—Dissolve 72.5 grams of crystallized copper sulphate in water and dilute to 1 liter. The refraction should be adjusted if necessary so as to be 36° at 20° C. To 4 volumes of the milk add 1 volume of the copper solution, shake well, and filter.

Of the above Lythgoe's copper sulphate method has the advantage of simplicity, accuracy, and narrow range of refraction for pure milk.

*Specific Gravity*.—The specific gravity of the clear filtrate, obtained by the method described above, is taken at 15° C., with the Westphal balance.

*Immersion Refractometer Reading*.—The instrument used is the Zeiss immersion or dipping refractometer described on pages 97 to 107. The serum, prepared as directed in a preceding paragraph, is examined in one of the small beakers accompanying the apparatus at a temperature of 20° C.

*Composition and Serum Constants of Milk of Known Purity*.—In the table on page 152 are given Lythgoe's results || on 33 samples from individual cows and 4 samples from herds, all of known purity. He concludes after several years' experience with samples of known purity that the presence of added water is shown by a refraction of less than 36°, furthermore that when the protein exceeds the fat the sample is skimmed milk.

*Nitrates*.—Pure milk, free from contamination with stable filth, contains no nitrates; well water, however, often contains a sufficient amount to enable the detection of a 10% admixture in milk.

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\* Woodman, Jour. Amer. Chem. Soc., 21, 1899, p. 503.

† Ackerman, Zeits. Unters. Nahr. Genussm, 13, 1907, p. 186.

‡ Baier and Neumann, Ibid., p. 369.

§ Lythgoe, Mass. State Bd. Health Rep., 1908, p. 38.

|| Ibid., Rep. 1910, p. 44.



ANALYSES OF MILK OF KNOWN PURITY (LYTHGOE).

Breed.	Months in Mil.	Weight of Milk (Lbs.).	Sp. Gr. 15° (Quevenne Scale.)	Total Solids.	Fat.	Lactose.	Protein.	Ash.	Solids not Fat.	COPPER SERUM.				NATURAL SOUR SERUM.		
										Refrac- tion, 20°.	Sp. Gr. 20° 4°.	Solids.	Lactose.	Refrac- tion, 20°.	Sp. Gr. 20° 4°.	Ash.
Grade Durham.....	2	15	35	14.58	5.10	5.00	3.35	0.81	9.48	30.7	28.0	6.28	4.43	44.4	20.2	0.768
Holstein.....	2	15	34	13.65	4.50	4.95	3.33	0.72	9.15	38.4	27.1	6.05	4.54	43.0	27.3	0.776
Grade Durham.....	2	16	36	13.52	4.00	5.20	3.54	0.72	9.06	38.9	27.2	6.09	4.38	43.0		
Grade.....	1	20	33	13.36	4.30	5.00	3.25	0.81	9.06	38.3	27.0	5.90	4.63	42.0		
Grade.....	4	18	34	13.30	4.00	5.00	3.54	0.76	9.00	38.7	26.9	6.07	4.56	43.1		
Holstein.....	5	10	32	13.29	4.40	4.70	3.14	0.70	8.89	38.8	27.5	6.10	4.34	42.2		
Grade Durham.....	2	15	34	13.27	4.00	5.40	3.30	0.78	9.00	39.2	28.2	6.20	4.50	43.3		
Grade Ayrshire.....	1	12	33	13.26	4.20	5.50	2.84	0.66	9.06	39.7	28.1	6.30	4.78	43.9		
Grade Swiss.....	2	16	33	13.20	4.35	4.75	3.29	0.76	8.85	37.6	26.1	5.86	4.48	40.8		
Grade Durham.....	2	15	33	13.06	3.80	5.75	3.37	0.69	9.20	38.0	27.1	6.05	4.82	42.4		
Grade Durham.....	2	16	34	13.02	4.10	5.30	3.01	0.80	9.02	39.3	27.7	6.19	4.47	43.1		
Ayrshire.....	9	10	31	12.91	3.90	4.35	3.51	0.76	9.01	37.3	25.9	5.73	3.94	41.0		
Grade Ayrshire.....	2	21	31	12.85	4.30	5.20	3.15	0.68	8.55	37.1	25.9	5.66	4.40	49.9		
Grade Holstein.....	2	17	32	12.78	4.00	5.05	2.89	0.71	8.78	38.0	27.4	6.07	4.40	42.1		
Grade Holstein.....	2	18	32	12.78	4.00	4.90	2.89	0.69	8.78	38.5	27.1	6.05	4.40	42.2		
Holstein.....	6	10	31	12.66	3.90	4.35	2.97	0.76	8.76	37.6	26.8	5.85	4.21	41.8		
Grade Holstein.....	2	20	32	12.64	3.80	5.15	3.01	0.62	8.84	38.7	27.3	6.11	4.71	41.9		
Grade Durham.....	2	18	35	12.58	3.70	5.00	3.12	0.74	8.88	38.4	26.9	5.93	.....	41.9		
Grade Durham.....	2	18	34	12.50	3.30	5.25	2.97	0.68	9.04	38.8	27.4	6.06	4.14	42.7		
Grade Durham.....	4	16	31	12.29	3.80	4.85	2.65	0.74	9.00	38.6	27.1	6.03	4.14	42.7		
Holstein.....	1	10	33	12.27	3.40	5.05	2.87	0.72	8.49	37.7	26.6	5.87	4.38	41.8		
Grade Holstein.....	8	18	32	12.12	3.50	4.40	3.32	0.84	8.62	37.6	26.4	5.82	4.11	41.2		
Grade Ayrshire.....	3	16	32	12.08	3.70	4.65	2.80	0.78	8.38	37.5	25.8	5.75	4.25	41.4		
Grade Holstein.....	4	20	32	12.03	3.45	5.00	2.89	0.72	8.58	38.6	27.3	6.09	4.33	40.6		
Grade Holstein.....	1	20	34	12.00	3.10	5.05	2.90	0.71	8.90	38.4	27.1	6.07	4.61	42.1		
Grade Holstein.....	4	16	30	11.77	3.70	4.50	2.67	0.71	8.07	37.2	26.1	5.79	4.30	41.5		
Holstein.....	4	18	33	11.40	3.20	5.00	2.48	0.72	8.20	37.5	26.1	5.79	4.30	41.5		
Holstein.....	7	15	31	11.27	3.15	4.30	3.09	0.78	8.12	36.6	25.3	5.47	4.22	41.0		
Holstein.....	10	18	31	11.21	3.35	4.30	2.81	0.75	7.86	36.3	25.0	5.37	3.88	39.7		
Holstein.....	3	20	31	11.48	3.40	4.50	2.78	0.80	8.08	36.8	25.0	5.37	3.88	38.7		
Grade Holstein.....	4	16	30	10.66	2.85	4.40	2.66	0.65	7.81	36.4	25.4	5.51	4.02	40.6		
Holstein.....	1	28	30	10.20	2.65	4.50	2.40	0.65	7.55	36.5	25.6	5.45	3.76	38.6		
Mixed milk*.....	.....	.....	33	13.40	4.20	5.10	3.13	0.70	9.20	38.5	26.9	6.05	4.09	42.3		
Mixed milk†.....	.....	.....	33	13.08	4.10	4.65	3.23	0.76	8.08	37.7	26.2	5.84	4.34	41.3		
Mixed milk†.....	.....	.....	33	12.73	3.80	4.70	3.33	0.71	8.03	38.0	26.1	5.94	4.22	41.4		
Mixed milk§.....	.....	.....	32	12.53	3.70	4.75	3.35	0.73	8.83	37.7	25.9	5.77	4.28	41.5		

\* Grade Ayrshire, Durham, Shorthorn and Holstein cows.

† Grade Jersey, Ayrshire and Holstein cows.

‡ Grade Holstein and Grade Jersey cows.  
§ Holstein, Grade Holstein and Grade Jersey cows.



The diphenylamin test, first employed by Soxhlet to detect nitrates in milk, has since been variously modified.\*

Place in a small porcelain crucible one cc. of a solution of 0.1 gram of diphenylamin in 1000 cc. of concentrated sulphuric acid and allow a few drops of the milk serum to flow over the surface. A blue color appearing within 10 minutes indicates the presence of nitrates. On longer standing, a brown color forms, whether or not nitrates are present. According to Willeke, Schellbach, and Jilke † milk to which hydrogen peroxide has been added also gives the blue color.

The delicacy of the test is increased by adding to the reagent a small amount of powdered sodium chloride shortly before using.

**Determination of Freezing Point.**—Beckmann, ‡ who proposed this means of detecting added water, reached the conclusion that the freezing point of pure milk ranged from  $-0.58^{\circ}$  to  $-0.54^{\circ}$  C., and that water influences the result in proportion to the amount added. While most of the later investigators find that  $-0.58^{\circ}$  is none too high for the minimum limit, Grüner § reports a maximum for single cows of  $-0.535^{\circ}$ , Pins || of  $-0.529^{\circ}$ , Stutterheim ¶ of  $-0.52^{\circ}$  and König \*\* of  $-0.515^{\circ}$ . Mixed herd milk appears seldom to fall outside of the limits  $-0.57^{\circ}$  and  $-0.53^{\circ}$ . Most authors agree that the per cent of fat, as well as the age, breed, period of lactation, and feed of the cow have little or no influence. Stutterheim, however, found that poor feeding gave freezing points in the case of eight cows from  $-0.52^{\circ}$  to  $-0.536^{\circ}$ . Souring and the addition of certain preservatives without question lower the freezing point (Bon-nema, †† Keister ‡‡). Gooren \*\* finds that homogenizing, pasteurizing, and sterilizing also lower it.

While the freezing point is undoubtedly a valuable constant and will detect with reasonable certainty as high as 10% of water, whether it serves for finer distinctions and is as reliable a means of diagnosis as either the

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\* Möslinger, Ber. 7 Versam. bayer. chem. Berlin, 1889; Richmond, Analyst, 18, 1893, p. 272; Hefelmann, Zeits. öffentl. Chem., 7, 1901, p. 200; Reisz, Pharm. Ztg., 49, 1904, p. 608; see also Tillmans, Zeits. Unters. Nahr. Genussm., 20, 1910, p. 676.

† Zeitz. Unters. Nahr. Genussm., 24, 1912, p. 227.

‡ Milch Ztg., 23, 1894, p. 702.

§ Ann. Ist. Agric., 6, 1901-1903, p. 27.

|| Inaug. Dis., Leipzig, 1910.

¶ Pharm. Weekbl., 54, 1917, p. 458.

\*\* Gooren, Centbl. Bakt., 35, II, 1912, p. 625.

†† Pharm. Weekbl., 43, 1906, No. 18.

‡‡ Jour. Ind. Eng. Chem., 9, 1917, p. 862.



solids-not-fat or the refraction, can be settled only by numerous determinations on authentic samples produced under a variety of conditions. The apparatus, although not so expensive as the immersion refractometer, requires more skill in manipulation. The possible presence of considerable lactic acid, preservatives, and common salt, the latter added to offset the effects of watering, should always be taken into account.

The apparatus and general process of determination are described on page 49.

**Other Milk Constants.**—*The Viscosity* of milk has been determined by various chemists. Kooper\* claims to be able to detect 5% of added water by this constant.

*The Specific Heat* of milk and milk products has been determined by Hammer and Johnson† because of its practical value in pasteurizing and refrigerating, also in the manufacture of butter and ice cream.

*The Electrical Conductivity* is stated by Favilli‡ to be unsatisfactory for determining added water in milk.

*Capillary and Adsorption Phenomena* have been studied by Kreidl and Lenk.§ Cow's milk on bibulous paper forms three concentric zones—casein, fat, and water. On dilution to a certain point no casein zone is formed.

*Oxidation Index.*—This constant, proposed by Comanducci,|| represents the number of cubic centimeters of N/10 potassium permanganate required in the presence of sulphuric acid to oxidize 1 cc. of milk. It is designed to distinguish cow's from goat's and sheep's milk.

**SYSTEMATIC EXAMINATION OF MILK FOR ADULTERATION.**—If a large number of samples of milk have to be examined daily for adulteration, it may be an advantage to submit all to a preliminary test with the lactoscope and lactometer, excluding from further analysis, as above the standard, such samples as pass certain prescribed limits which experience has proved these tests to be capable of showing to an experienced observer, and submitting the remainder to a chemical analysis. In using such an instrument as the lactoscope for this purpose, the individual element is a most important consideration, and the use of this instrument

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\* Milchw. Zentbl., 43, 1914, p. 169.

† Iowa Agric. Exp. Sta., Res. Bul. 14, 1913, p. 451.

‡ Riv. sci. latte, 1, 1911, p. 33.

§ Pflüger's Arch. Ges. Physiol., 141, 1911, p. 541.

|| Gaz. chim. Ital., 36 II, 1906, p. 813.



in the milk laboratory should be limited only to a skillful operator, accustomed to interpret its results.

The method used by the Mass. Board of Health has been to submit all samples to the regular test for solids, and such samples as fall below the legal standard for solids, are further examined for fat.

**Total Solids, Ash, and Fat.**—It is presupposed that the analyst is equipped with a sufficient number of platinum dishes for the number of milk samples daily analyzed. It is a convenience to have these dishes numbered, and instead of weighing each dish, to have a system of numbered counterweights (Fig. 51, A) corresponding to the dishes. The counterweights recommended by Leach for this purpose are easily made from half-inch lead pipe, cut to the appropriate length and flattened. Each weight is then carefully adjusted to its appropriate dish, by trimming off the weight with a knife, or by adding bits of lead scraps, if necessary, by simply prying open in the center, inserting the required amount of scrap, and then closing by a blow of the hammer, the weight being plainly numbered before final adjustment. A rack is provided by the side of the balance-case (Fig. 51) with slits for holding the weights in their appropriate places. Such a set of counterweights is not difficult to make, requires very little care to keep in adjustment, and is an immense labor-saving device.

*Details of Manipulation.*—The method of examining large numbers of milk samples, long in use in the laboratory of the Massachusetts State Board of Health, has proved to be rapid, easy, and accurate. It is here given in some detail.

From 12 to 20 samples of milk are conveniently weighed out at a sitting, the unopened sample cans or bottles being contained in a tray at the left of the operator on a low stand, another low stand and tray being at this right hand for the cans, after removing the weighed portions, and a third tray on the table at the right of the balance for the platinum dishes with the weighed samples. The analyst enters the number of the platinum dish in his note-book, or on a card,\* in line with the number of the milk sample, verifies the correctness of the counterweight, and weighs out exactly 5 grams of the milk with the aid of a pipette, after first having thoroughly mixed the sample. This operation is repeated with all the samples, the platinum dishes containing the weighed amounts

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\* Specially ruled library cards, as shown on page 157, are useful for this purpose.



of each being placed in succession on the tray, which is finally carried to the water-bath and the dishes transferred thereto. The time required for weighing out 12 samples of milk in this manner is about fifteen minutes.

The water-bath is inclosed in a hood, and the sliding front is so arranged that it can be shut down and locked, so that if the analyst has to leave

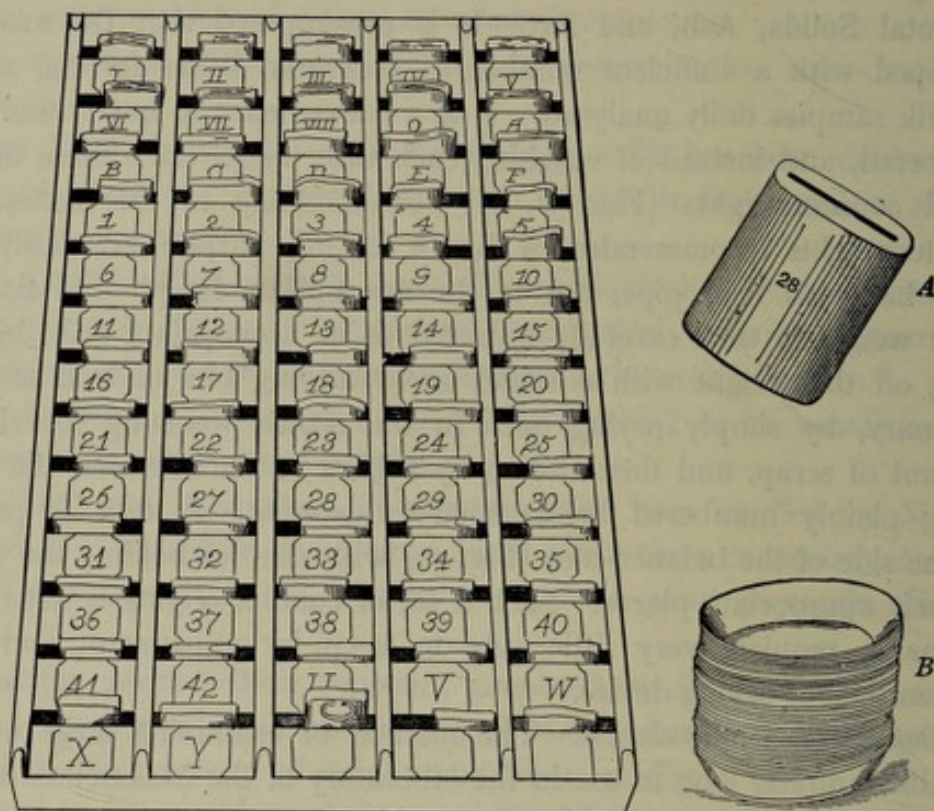


FIG. 51.—Set of Counterweights for Numbered Platinum Dishes, in a Convenient Rack.

- A. One of the Counterweights.  
B. Platinum Dishes.

the laboratory during the three hours required for the evaporation, he can swear in court that the samples could not be tampered with during his absence (see page 20).

When ready to make the second weighings for the total solids, each dish is taken from contact with the steam, and, while still hot, is wiped dry with a soft towel, till twelve of the dishes are placed on the tray, which is then taken to the balance. Experience has shown that with ordinary rapidity in weighing, twelve of the residues may be thus dealt with at a time without the need of a desiccator, the gathering of moisture during that time being inappreciable, excepting in very damp weather, when a less number of dishes should be removed at a time from the bath. In making the second weighing, and employing the counterweight as



Specimen Card for Analyst's Records of Milk Analyses. To be filed in a cabinet.



before, the exact net weight of the residue is at once ascertained and entered in the appropriate column in the note-book. Multiplied by 20 it gives at once the percentage of total solids.

It is a great saving of time to weigh out exactly 5 grams as above described. The knack of quickly measuring out the exact amount is easily acquired with practice, the 5-gram weight is the only one required for the operation with the counterweight of the dish, and the laborious figuring of percentage due to using a fraction above or below the 5 grams of milk is avoided.

Such samples as are found to stand below the standard of total solids are further examined for fat by the Babcock process (p. 123), entering the number of the fat bottle in the note-book in the appropriate column, and subsequently the percentage of fat.

Ordinarily the specific gravity is not determined, excepting in some cases of badly watered milk, when, for purposes of a check, it is customary to take the specific gravity, and calculate the solids from the gravity and the fat by Babcock's formula (p. 140), or the Richmond sliding scale, and compare the result with the figure directly determined.

The ash is rarely weighed except in special cases.

The dishes containing the dry residues are easily cleaned by first burning to an ash and cooling, after which they are treated successively with strong nitric acid, which is poured from one to another, the dishes being rinsed thoroughly with water and finally heated to redness.

A convenient device for ashing a large number of residues for purposes of cleaning the platinum dishes and for final heating is the incinerator shown in Fig. 52, made of Russia iron. The digestion stand for the Kjeldahl method (Fig. 27*a*) may also be used.

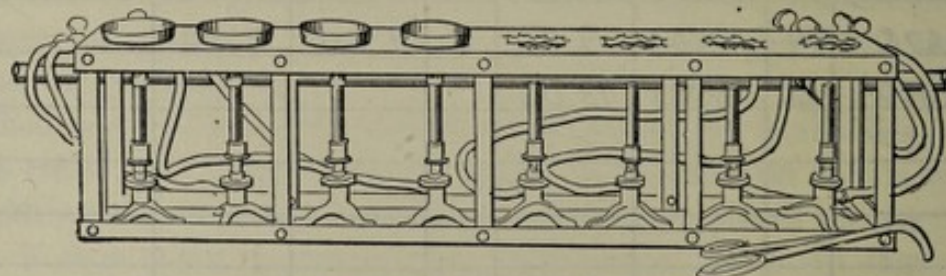


FIG. 52. A Sheet-metal Incinerator, Specially Used for Ashing Milk Residue.

**ADDED FOREIGN INGREDIENTS.**—Passing over such mythical adulterants as chalk and such rarely used substances as calves' brains, starch, glycerin, sugar, etc., often discussed in manuals on milk, but



with few authentic instances of their actual occurrence, the commonly found adulterants may be divided into two classes: coloring matters and preservatives.

The coloring matters almost exclusively used are annatto, azo-colors, and caramel. The preservatives commonly met with are formaldehyde, boric acid, borax, and sodium bicarbonate. Rarely salicylic and benzoic acids are found.

**COLORING MATTERS.**—While it is more often true that an artificially colored milk is also found to be watered, the coloring being added to cover up evidence of the watering, it is not uncommon to find added coloring matter in milk above the standard.\*

About 95% of the milks found colored in Massachusetts showed on analysis the fraudulent addition of water.

Statistics of the Massachusetts State Board of Health show that out of 48,000 samples of milk collected throughout the state and analyzed during nine years (from 1894 to 1902 inclusive) 342 samples or 0.7% were found to contain foreign coloring matter. Of these samples, about 67% contained annatto, approximately 30% were found with an azo-dye, and about 3% with caramel.

Until comparatively recently annatto was employed almost exclusively for this purpose. Caramel is least desirable of all the above colors from the point of view of the milk-dealer, in that it is difficult to imitate with it the natural color of milk, by reason of the fact that the caramel color has too much of the brown and too little of the yellow in its composition. Annatto, on the other hand, when judiciously used and with the right dilution, gives a very rich, creamy appearance to the milk, even when watered, which accounts for its popularity as a milk adulterant. Of late, however, the use of one or more of the azo-dyes has been on the increase, and so far as a close imitation of the cream color is concerned, these colors are quite as efficient as annatto.

**Appearance of Artificially Colored Milk.**—The natural yellow color of milk confines itself largely to the cream. An artificial color, on the contrary, is dissipated through the whole body of the milk, so that when the cream has risen in a milk thus colored, the underlying layers, instead of showing the familiar bluish tint of skimmed milk, are still distinctly tinged below the layer of the fat, especially if any considerable quantity of the color has been used. This distinctive appearance is in itself often

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\* In one instance an azo-dye was found by the writer in a milk that contained over 17% of total solids.



sufficient to direct the attention of the analyst to an artificially colored milk, in the course of handling a large number of samples.

**Nature of Annatto.**—Annatto, arnatto, or annotto is a reddish-yellow coloring matter, derived from the pulp inclosing the seeds of the *Bixa orellana*, a shrub indigenous to South America and the West Indies.

A solution of the coloring matter in weak alkali is the form usually employed in milk.

**Nature of "Anilin Orange."**—Of the coal-tar colors employed for coloring milk, the azo-dyes are best adapted for this purpose and are most used. A few samples of these commercial "milk improvers" have fallen into the hands of the Department of Food and Drug Inspection of the Massachusetts Board of Health, and have proved, on examination, to be mixtures of two or more members of the diazo-compounds of anilin. A mixture of what is known to the trade as "Orange G" and "Fast Yellow" gives a color which is practically identical with one of these preparations, secured from a milk-dealer and formerly used by him.

For purposes of prosecution or otherwise, it is obviously best in our present knowledge of the subject to adopt a generic name such as "a coal-tar dye" or "anilin orange"\* to designate this class of coloring matters in milk, rather than to particularize.

**Systematic Examination of Milk for Color.**—The general scheme employed by the writer for the examination of milk samples suspected of being colored is as follows:† About 150 cc. of the milk are curdled by the aid of heat and acetic acid, preferably in a porcelain casserole over a Bunsen flame. By the aid of a stirring-rod, the curd can nearly always be gathered into one mass, which is much the easiest method of separation, the whey being simply poured off. If, however, the curd is too finely divided in the whey, the separation is effected by straining through a sieve or colander. All of the annatto, or of the coal-tar dye present in the milk treated would be found in the curd, and part of the caramel. The curd, pressed free from adhering liquid, is picked apart, if necessary, and shaken with ether in a corked flask, in which it is allowed to soak for several hours, or until the fat has been extracted, and with it the annatto. If the milk is uncolored, or has been colored with annatto, on pouring off the ether the curd should be left perfectly white. If, on

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\* The term "anilin orange" has been so commonly applied during Leach's experience to any color or mixture of colors of this class in complaints in the Massachusetts courts, as to have acquired a special meaning perfectly well understood.

† Jour. Am. Chem. Soc., 22, 1900, p. 207.



the other hand, anilin orange or caramel has been used, after pouring off the ether the curd will be colored more or less deeply, depending on the amount of color employed. In other words, of the three colors, annatto, caramel, and anilin orange, the annatto only is extracted by ether. If caramel has been used, the curd will have a brown color at this stage; if anilin orange, the color of the curd will be a more or less bright orange.

**Tests for Annatto.**—The ether extract, containing the fat and the annatto, if present, is evaporated on the water-bath, the residue is made alkaline with sodium hydroxide, and poured upon a small, wet filter, which will hold back the fat, and, as the filtrate passes through, will allow the annatto, if present, to permeate the pores of the filter. On washing off the fat gently under the water-tap, all the annatto of the milk used for the test will be found to have been concentrated on the filter, giving it an orange color, tolerably permanent and varying in depth with the amount of annatto present. As a confirmatory test for annatto, stannous chloride may afterward be applied to the colored filter, producing the characteristic pink color.

**Tests for Caramel.**—The fat-freed curd, if colored after the ether has been poured off, is examined further for caramel or anilin orange, by placing a portion of the curd in a test-tube, and shaking vigorously with concentrated hydrochloric acid. If the color is caramel, the acid solution of the colored curd will gradually turn a deep blue on shaking, as would also the white fat-free curd of an uncolored milk, the blue coloration being formed in a very few minutes, if the fat has been thoroughly extracted from the curd; indeed, it seems to be absolutely essential for the prompt formation of the blue color in the acid solution that the curd be free from fat. Gentle heat will hasten the reaction. It should be noted that it is only when the blue coloration of the acid occurs in connection with a colored curd that caramel is to be suspected, and if much caramel be present, the coloration of the acid solution will be a brownish blue. If the above treatment indicates caramel, it would be well to confirm its presence, by testing a separate portion of the milk in the following manner.\*

About a gill of the milk is curdled by adding to it as much strong alcohol. The whey is filtered off, and a small quantity of subacetate of lead is added to it. The precipitate thus produced is collected upon a small filter, which is then dried in a place free from hydrogen sulphide. A pure milk thus treated yields upon the filter-paper a residue which is

\* See Nineteenth Annual Report of the Mass. State Board of Health (1887), p. 183.



either wholly white, or at most of a pale straw color, while in the presence of caramel, the residue is a more or less dark-brown color, according to the amount of caramel used.

**Tests for Coal-tar Dye.**—If the milk has been colored with an azo-dye, the colored curd, on applying the strong hydrochloric acid in the test-tube, will immediately turn pink. If a large amount of the anilin dye has been used in the milk, the curd will sometimes show the pink coloration when hydrochloric acid is applied directly to it, before treatment with ether, but the color reaction with the fat-free curd is very delicate and unmistakable.\*

*Lythgoe*† has shown that the amount of anilin orange ordinarily present in a milk for the purposes of coloring can be detected by adding directly to say 10 cc. of the sample an equal quantity of strong hydrochloric acid and mixing, whereupon the pink coloration is produced, if the dye is present in more than minute traces. The test is more delicate if carried out in a white porcelain dish. It had best be used as a preliminary test only, and confirmed by a subsequent test on the fat-free curd as above.

#### SUMMARY OF SCHEME FOR COLOR ANALYSIS.

Curdle 150 cc. milk in casserole with heat and acetic acid. Gather curd in one mass. Pour off whey, or strain, if curd is finely divided. Macerate curd with ether in corked flask. Pour off ether.

##### Ether Extract.

Evaporate off ether, treat residue with NaOH and pour on wetted filter. After the solution has passed through, wash off fat and dry filter, which if colored orange, indicates presence of annatto. (Confirm by  $\text{SnCl}_2$ .)

##### Extracted Curd.

(1) *If Colorless.*—Indicates presence of no foreign color other than in ether extract.

(2) *If Orange or Brownish.*—Indicates presence of anilin orange or caramel. Shake curd in test-tube with concentrated hydrochloric acid.

If solution *gradually* turns blue, indicative of caramel. (Confirm by testing for caramel in whey of original milk.)

If orange curd *immediately* turns pink, indicative of anilin orange.

**PRESERVATIVES.**—In most states and municipalities where pure food laws are in force preservatives in milk are regarded as adulterants

\* Occasional samples of milk colored with a coal-tar dye of a different class from those already described have recently been found in Massachusetts. In these cases the color of the separated fat-free curd does not change when treated with hydrochloric acid. The color of the curd is, however, very marked, being deep orange, bordering on the pink.

† Jour. Am. Chem. Soc., 22, 1900, p. 813.



Their use, however, seems to be on the decrease. Of 6,186 samples of milk examined by the Massachusetts State Board of Health during one year (1899) 71 samples, or 1.2%, were found to contain a preservative. Of these 55 were found with formaldehyde, 13 containing boric acid, borax, or a mixture of the two, and 3 contained carbonate of soda.

Comparative tests were made of the keeping qualities of these common preservatives, the milk being kept during the experiment at the temperature of the room, which at that season of the year (February) was about 20° C.\* The preservatives were added about five hours after milking. The samples were titrated for acidity each morning, the acidity being expressed by the number of cubic centimeters of decinormal sodium hydroxide necessary to neutralize 5 cc. of the milk.

The proportions of preservatives used in this experiment, as shown in the table on page 164, were intended to cover a wide range, from the weakest that could aid in preserving the milk up to a strength limited only by being perceptible to the taste. The results obtained appear in the table.

**Formaldehyde**, the most commonly used preservative for milk, is sold to the trade under various names, such as "Preservaline," "Freezine," "Ice-line," etc., all being dilute aqueous solutions of formaldehyde, containing from 2 to 6 per cent of the gas, being nearly always diluted from the 40% solution known as formalin. These preparations are usually accompanied by directions, which specify the amount to be used, varying from a tablespoonful of the solution in 5 to 10 gallons of the milk. It is commonly used in the strength of 1 part of the gas in 20,000, and rarely less than 1 part in 50,000. The antiseptic power of formaldehyde increases in a marked degree as the strength of the preservative is increased. Milk treated with 1 part in 10,000, for instance, according to the table was found to keep sweet 5½ days. In the strength of 1 part to 5000, the milk did not curdle for 10½ days, while 1 part of formaldehyde to 2500 parts of milk kept the milk from curdling for 55 days, the acidity up to that time being nearly normal.

Formaldehyde is thus shown to be decidedly the most efficient of all milk preservatives, besides being inexpensive and convenient to use.

Whether the growth of other bacteria than those that produce lactic fermentation is inhibited by formaldehyde in milk is not definitely settled. The claim has been made that pathogenic varieties are destroyed by its use.

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\* Thirty-first Annual Report Mass. State Board of Health, 1899, p. 611.



## ACTION OF VARIOUS PRESERVATIVES ON MILK.

Preservative.	First Day.		Second Day.		Third Day.		Fourth Day.		Fifth Day.	
	Acidity.	Con- dition.	Acidity.	Con- dition.	Acidity.	Con- dition.	Acidity.	Con- dition.	Acidity.	Con- dition.
Blank.	0.78	Sweet	0.96	Sweet	3.95	Sour	—	—	—	—
Sodium carbonate:	—	Sweet	0.75	Sweet	4.05	Sour	—	—	—	—
I : 1000.	—	"	0.75	"	4.05	"	—	—	—	—
I : 500.	—	"	0.75	"	4.05	"	—	—	—	—
I : 250.	—	"	0.75	"	4.05	"	—	—	—	—
Boric acid 75%, borax 25%:	—	Sweet	0.96	Sweet	4.04	Sour	—	—	—	—
I : 2000.	—	"	1.05	"	1.64	Sweet	—	—	—	—
I : 1500.	—	"	1.19	"	1.44	"	—	—	—	—
I : 1000.	—	"	1.50	"	1.52	"	—	—	—	—
I : 500.	—	"	—	"	—	"	—	—	—	—
Boric acid 25%, borax 75%:	—	Sweet	1.00	Sweet	2.77	Sweet	—	—	—	—
I : 2000.	—	"	1.00	"	1.05	"	—	—	—	—
I : 1500.	—	"	1.04	"	1.27	"	—	—	—	—
I : 1000.	—	"	1.12	"	1.40	"	—	—	—	—
I : 500.	—	"	—	"	—	"	—	—	—	—
Formaldehyde:	0.78	Sweet	0.80	Sweet	1.09	Sweet	—	—	—	—
I : 50000.	—	"	0.79	"	0.81	"	—	—	—	—
I : 20000.	—	"	0.78	"	0.80	"	—	—	—	—
I : 10000.	—	"	0.78	"	0.79	"	—	—	—	—
I : 5000.	—	"	—	"	—	"	—	—	—	—
Blank.	1.19	Sweet	4.10	Sour	0.94	Sweet	1.10	Sweet	1.32	Sour
Sodium carbonate:	0.92	"	0.93	Sweet	0.96	Sweet	1.10	Sweet	1.32	Sour
I : 1000.	—	"	—	"	—	"	—	"	—	"
I : 500.	—	"	—	"	—	"	—	"	—	"
I : 250.	—	"	—	"	—	"	—	"	—	"
Boric acid 75%, borax 25%:	—	"	—	"	—	"	—	"	—	"
I : 2000.	—	"	—	"	—	"	—	"	—	"
I : 1500.	—	"	—	"	—	"	—	"	—	"
I : 1000.	—	"	—	"	—	"	—	"	—	"
I : 500.	—	"	—	"	—	"	—	"	—	"
Boric acid 25%, borax 75%:	—	"	—	"	—	"	—	"	—	"
I : 2000.	—	"	—	"	—	"	—	"	—	"
I : 1500.	—	"	—	"	—	"	—	"	—	"
I : 1000.	—	"	—	"	—	"	—	"	—	"
I : 500.	—	"	—	"	—	"	—	"	—	"
Formaldehyde:	—	"	—	"	—	"	—	"	—	"
I : 50000.	—	"	—	"	—	"	—	"	—	"
I : 20000.	—	"	—	"	—	"	—	"	—	"
I : 10000.	—	"	—	"	—	"	—	"	—	"
I : 5000.	—	"	—	"	—	"	—	"	—	"



Notwithstanding the claims of manufacturers as to the harmlessness of formaldehyde, its use can not be too strongly condemned.\*

**Detection of Formaldehyde.**—*Leach Test.*†—Hydrochloric acid (specific gravity 1.2) containing 2 cc. of 10% ferric chloride per liter is used as a reagent. Add 10 cc. of the acid reagent to an equal volume of milk in a porcelain casserole, and heat slowly over the free flame nearly to boiling, holding the casserole by the handle, and giving it a rotary motion while heating to break up the curd. The presence of formaldehyde is indicated by a violet coloration, varying in depth with the amount present. In the absence of formaldehyde, the solution slowly turns brown. By this test 1 part of formaldehyde in 250,000 parts of milk is readily detected before the milk sours. After souring, the limit of delicacy proves to be about 1 part in 50,000.

Various aldehydes, when introduced into milk, give color reactions under the above treatment, but formaldehyde alone gives the violet coloration, which is perfectly distinguishable and unmistakable.

*Hehner Test.*‡—To 5 to 10 cc. of milk in a wide test-tube add about half the volume of concentrated commercial sulphuric acid,§ pouring the acid carefully down the side of the tube, so that it forms a layer at the bottom without mixing with the milk. A violet zone at the junction of the two liquids indicates formaldehyde. This test may be combined with the Babcock test for fat, noting whether a violet color forms on addition of the commercial sulphuric acid to the milk in the test bottle.

*Tests with Distilled Milk.*—To confirm the above test, distil 100 cc. of the milk sample acidified with citric or sulphuric acid, and test the first 20 cc. of the distillate as described in Chapter XVIII.

**The Determination of Formaldehyde in Milk** is unsatisfactory since it gradually disappears, as shown by Williams and Sherman,|| owing

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\* Milk-dealers are led to believe, by artful dealers in preservative preparations, that the chemist cannot detect them. The manufacturer of a widely used preservative, a weak solution of formaldehyde, issued an attractive pamphlet in which he made the following remarkable claims:

"It is not an adulterant. It immediately evaporates, so that no trace of it can be found as soon as it has rendered all the bacteria inert. No chemical analysis can prove its presence in milk, quantitatively or otherwise."

† Annual Report Mass. State Board of Health, 1897, p. 558; also 1899, p. 699.

‡ Analyst, 20, 1895, p. 155.

§ The coloration produced seems to depend on the presence of iron salts in the acid, hence the use of commercial acid is recommended. If only pure acid is available, a little ferric chloride should be added.

|| Jour. Am. Chem. Soc., 27, 1905, p. 497.



to the formation of condensation products with the proteins. According to Smith,\* the first 20 cc. of the distillate contain nearly one-third of the total formaldehyde then present. In some cases it may be useful to determine the amount present in this distillate by the potassium cyanide method (p. 883).

**Boric Acid**, either in the form of the free acid or of the sodium salt borax, has been much used in milk. While its addition to butter is legalized in England, food authorities in all countries are generally agreed that its use in milk is highly objectionable.

**Detection of Boric Acid.**—This is best accomplished by the turmeric-paper test applied either directly to the milk or to the ash (p. 885). In the former case 10 cc. of milk are thoroughly mixed with 6 drops of concentrated hydrochloric acid, after which the turmeric paper, previously marked for identification with a lead pencil, is moistened with the mixture and dried. Bertrand and Agulhon† find by their spectroscopic method 0.5–1.11 mg. of boron as hydroxide per liter of milk to which nothing has been added. This amount is not evident by ordinary tests.

**Determination of Boric Acid.**—The Gooch method (p. 887) or the Thompson method (p. 886) may be used.

Richardson and Walton‡ propose the following rapid method, which is stated to be more accurate than the Thompson method, since it obviates the loss of boric acid by volatilization with the fat. While other authors, including the author and reviser, have not found this loss considerable if the ignition is properly conducted, the proposed method recommends itself because of its simplicity. To 50 cc. of milk, or 10 grams of cream diluted with 40 cc. of water, add 5 cc. of 5% copper sulphate solution, stir, heat to boiling for a few seconds, filter, and wash the precipitate containing the proteins and fat. Cool the filtrate and determine the boric acid by titration, using 2 cc. of 1% neutral phenolphthalein solution as indicator.

**Carbonate and Bicarbonate of Soda.**—These substances are occasionally used in milk, though, as the table on p. 164 shows, they possess little or no value as milk preservatives. They do, however, serve to neutralize the acidity of slightly soured milk and to postpone the time of actual curdling.

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\* Jour. Am. Chem. Soc., 25, 1903, pp. 1032, 1037.

† Compt. rend., 156, 1913, p. 2027.

‡ Analyst, 38, 1913, p. 40.



**Detection of Carbonate and Bicarbonate of Soda.**—The addition of carbonates is manifest by the effervescence caused by treating the milk-ash with acid. Effervescence in the milk-ash is quite perceptible, when as much as 0.05% of sodium carbonate is present.

Schmidt's method of detecting sodium carbonate or bicarbonate, when present to the extent of 0.1% or more, is as follows: Ten cc. of milk are mixed with an equal volume of alcohol, and a few drops of a 1% solution of rosolic acid are added. If carbonate is present, a rose-red color will be produced, while pure milk shows a brownish-yellow coloration. The suspected sample thus treated should be compared with a similarly treated sample of pure milk at the same time.

**Salicylic and Benzoic Acids**, in view of the much more efficient antiseptics at hand, are now rarely used as milk preservatives, though the analyst should be on the outlook for them. Salicylic acid is a poor milk preservative, in view of the fact that it affects the taste of the milk, when present in sufficient quantity, to be of service.

**Detection of Salicylic Acid.**—(1) To 50 cc. of the milk add 1 cc. of acid nitrate of mercury reagent (p. 134), shake and filter. The filtrate, which should be perfectly clear, is then shaken with ether in a separatory funnel, the ether extract evaporated to dryness, and a drop of ferric chloride reagent applied. If salicylic acid be present, a violet color will be produced. In carrying out the test it should be noted that a small portion only of the salicylic acid is in the filtered whey, the larger part being left in the curd. The color test is, however, so delicate as to show its presence, when an appreciable amount is used.

(2) Proceed exactly as directed for benzoic acid (below). On applying the ferric chloride to the final solution, after evaporation of the ammonia, a violet color shows the presence of salicylic acid.

**Detection of Benzoic Acid.**—Shake 5 cc. of hydrochloric acid with 50 cc. of the milk in a flask. Then add 150 cc. of ether, cork the flask and shake well. Break up the emulsion which forms by the aid of a centrifuge, or, in the absence of a centrifuge, extract the curdled milk by gently shaking with successive portions of ether, avoiding the formation of an emulsion. A volume of ether largely in excess over that of the curdled milk has been found to be less apt to emulsionize. Transfer the ether extract to a separatory funnel, and separate the benzoic acid from the fat by shaking out with dilute ammonia, which takes out the former as ammonium benzoate. Evaporate the ammonia solution in



a dish over the water-bath until neutral to test paper and add a few drops of neutral ferric chloride reagent (page 891).

*Revis Method.\**—Dilute 100 cc. of milk or 50 cc. of cream to 200 cc., add 5 cc. of 10% sodium carbonate solution, place on a boiling water bath, and after 2 to 3 minutes add 10 cc. of 25% calcium chloride solution and continue the heating until the casein is coagulated. Cool, filter, add hydrochloric acid to the filtrate until neutral to litmus, then 10 cc. of Fehling copper sulphate solution and 10 cc. of potassium hydroxide solution (31.81 grams per liter). Filter, acidify the filtrate with hydrochloric acid, extract with 50 cc. of ether, and wash the ether three times with water. Without removing from the separatory funnel add to the ether 10 cc. of water, 1 drop of phenolphthalein solution, and titrate with saturated barium hydroxide solution until a pink color persists after vigorously shaking. Remove the aqueous layer, filter, and evaporate to about 5 cc. Filter again, add 1% acetic acid until colorless, then 2 drops additional and test with 1 drop of freshly prepared 10% neutral ferric chloride solution as described on page 891.

*Robin Method.†*—Add 50 cc. of milk slowly with stirring to a mixture of 10 cc. of 5% sulphuric acid and 20 cc. of 95% alcohol, filter after 4 to 5 minutes, extract with ether, and proceed as described on page 891.

**Determination of Benzoic Acid in Milk.**—See pages 893 to 896.

*Liverseege and Evers Method.‡*—Distil a mixture of 100 cc. of milk and 10 cc. of concentrated sulphuric acid in a current of steam until 600 cc. have condensed. Acidulate the distillate with 5 cc. of concentrated hydrochloric acid, extract with 100 cc. and two portions of 35 cc. of ether, allow the extract to evaporate at room temperature in a tared dish, dry, weigh, deduct 5 milligrams (or a quantity found by blank experiment) from the total weight, and calculate the percentage of benzoic acid by a factor which should be determined by each analyst for the apparatus employed. In the apparatus used by the originators of the method about 45% of the total amount was recovered.

**Hydrogen Peroxide** is used in "perhydrase" or "Buddized" milk.

**Detection of Hydrogen Peroxide.**—*Arnold and Mentzel Vanadic Acid Method.§*—To 10 cc. of the milk add 10 drops of a solution of 1 gram of vanadic acid in 100 grams of dilute sulphuric acid. The presence of hydro-

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\* Analyst, 37, 1912, p. 346.

† Ann. chim. anal. appl., 14, 1909, pp. 2, 53.

‡ Jour. Soc. Chem. Ind., 32, 1913, p. 319.

§ Chem. Ztg., 26, 1902, p. 589.



gen peroxide is shown by the appearance of a red color. Utz \* states that the reaction is obtained whether or not the milk has been heated previous to adding the peroxide.

*Peroxidase Methods.*—Several of the methods for detecting peroxidase (pp. 173 and 174), notably the paraphenylenediamine (Dupouy), the benzidine (Wilkinson and Peters), and the iodide-starch (Roi and Köhler) methods, can be reversed for the detection of hydrogen peroxide. The tests are conducted as described except that the hydrogen peroxide is omitted and, since the peroxidase may have been destroyed by hydrogen peroxide or by heating, it is usually necessary to add raw milk of known purity. La Wall† in performing the Wilkinson and Peters test first coagulates a mixture of 10 cc. of raw milk and 2 cc. of 4% alcoholic benzidine with 2 to 3 drops of glacial acetic acid, then adds a few cubic centimeters of the suspected sample. A blue zone is formed when from 1.5 to 30 parts of hydrogen peroxide per 10,000 are present.

*Hehner-Feder Formaldehyde Method.*‡—This is the Hehner method for formaldehyde reversed and slightly modified. Mix 5 cc. of the milk with 5 cc. of concentrated hydrochloric acid and a drop of dilute formaldehyde solution, then heat at 60° C. for 3 to 4 minutes and shake once. If a violet color develops hydrogen peroxide is indicated. Wilkinson and Peters § have shown that the reaction is most decisive when about 0.005% of hydrogen peroxide and 0.004 to 0.013% of formaldehyde are present; with other proportions it may fail. Ferric salts, nitrites, and possibly nitrates also give a violet color.

**Routine Inspection of Milk for Preservatives.**—It was Leach's custom in Massachusetts to examine all the samples of milk collected during the months of June, July, August, and September for the commonly used preservatives, in addition to the regular analysis for total solids and fat. The number of samples thus examined amounted to upwards of 500 per month, varying from 10 to 60 per day. The results of such an examination during four years are shown on p. 170. ||

Such a system by no means involves a large amount of time or labor, and is really essential before passing judgment upon the purity of the milk, since, unlike added color, there is nothing in the physical appear-

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\* Milchw. Zentbl., 1, 1905, p. 175.

† Am. Jour. Pharm., 82, 1908, p. 57.

‡ Zeitz. Unters. Nahr. Genussm., 15, 1908, p. 234.

§ Ibid., 16, 1908, p. 515.

|| Mass. State Bd. Health, Rep., 1902, p. 474.



ance of the milk to suggest the presence of preservatives, nor are they rendered apparent by the taste, if skillfully used.

#### PRESERVATIVES IN MILK.

Year.	Samples Examined.	Number Containing Form-aldehyde.	Per Cent Containing Form-aldehyde.	Number Containing Boric Acid.	Per Cent Containing Boric Acid.	Number Containing Carbonate.	Total Containing Preserva-tive.
1898.....	1046	26	2.5	11	1.0	4	41
1899.....	2105	55	2.6	13	0.6	3	71
1900.....	2018	61	3.0	6	0.3	—	67
1901.....	2154	42	1.9	12	0.5	—	54
1902.....	1934	29	1.5	14	0.7	—	43
Totals.....	9257	213	2.3	56	0.6	7	376

The methods employed are carried out as follows: \*

(1) *Formaldehyde*.—After having been examined for total solids and fat, the milk samples are arranged in order in their original containers, and about 10 cc. of each sample are poured into a casserole and tested in succession by means of the hydrochloric acid and ferric chloride test (p. 165). A large stock bottle, which may be fitted with a siphon if desired, is kept on hand containing the hydrochloric acid reagent. Less than one minute is required in making the formaldehyde test for each sample.

(2) *Carbonate and Boric Acid*.—These tests have been so simplified as to be, as it were, a side issue in the process of cleaning the platinum dishes used for the determination of total solids. The various residues from the total solids are burnt to an ash in the original numbered dishes in succession, these dishes, after incineration, being arranged side by side on a flat tray. By means of a pipette, one or two drops of dilute hydrochloric acid are introduced into each dish in succession, noting at the time any effervescence that may ensue, which is in itself an indication of sodium carbonate. After every milk ash has been acidulated, a few cubic centimeters of water are added to each dish by means of a wash-bottle, the dissolving of the ash being hastened by giving a rotary motion to the tray containing the dishes. A strip of turmeric-paper is then allowed to soak for a minute or so in each dish, after which it is withdrawn from

\* Mass. State Bd. Health, Rep., 1901, p. 447.



contact with the solution and allowed to adhere to the side of the dish above the liquid, where it remains until dry. If the paper when dry is of a deep cherry-red color, turning a dark olive when treated with dilute alkali, the presence of boric acid is assured. These methods are, of course, preliminary tests for quickly singling out the preserved samples. Such confirmatory tests as are desired may in all cases be employed.

**VARIOUS ADULTERANTS.—Cane Sugar** is said to be used to increase the total solids of milk, but if present to any marked degree, it could hardly fail of detection by reason of the sweet taste imparted to the milk. Cane sugar in milk may be detected \* by boiling 5 to 10 cc. of the sample with about 0.1 gram of resorcin and a few drops of hydrochloric acid for a few minutes. In the presence of cane sugar, a rose-red color is produced.

According to Richmond, cane sugar may be estimated by first ascertaining the total polarization of the sample as in the estimation of milk sugar (p. 134). The milk sugar is then determined by Fehling's solution (pp. 136 to 138) either volumetrically or gravimetrically. The difference between the anhydrous milk sugar found by the latter, or Fehling method, and that calculated by dividing the polarization by 1.217 will give the percentage of cane sugar present.

Cotton's method † of detecting cane sugar, when present to the extent of 0.1% consists in mixing in a test-tube 10 cc. of the suspected milk with 0.5 gram of powdered ammonium molybdate, and adding to the mixture 10 cc. of dilute hydrochloric acid (1 to 10). Ten cc. of milk of known purity, or 10 cc. of a 6% solution of milk sugar are similarly treated by way of comparison. Both tubes are placed in a water-bath and the temperature gradually raised to 80° C. If cane sugar is present, an intense blue coloration is produced, while the genuine milk or the solution of milk sugar remains unchanged at the temperature of 80°. If the temperature is raised to the boiling-point, however, the pure milk or milk sugar solution may also turn blue.

**Detection of Starch in Milk.**—A small quantity of milk is heated in a test-tube to boiling, cooled, and a drop of iodine in potassium iodide added. A blue coloration indicates starch.

**Condensed Skimmed Milk as an Adulterant.**—The use of condensed unsweetened skimmed milk to raise the solids of a skimmed or watered

\* Woodman and Norton, *Air, Water, and Food*, New York, 1914, p. 151.

† *Abs. Analyst*, 23, 1898, p. 37.



milk above the standard has been noted in Massachusetts. This sophistication is rendered apparent by the abnormally high solids not fat of the sample, which in some instances have exceeded 11%. A solid not fat in excess of 10% is suspicious of this form of adulteration. By fixing a legal standard for both fat and solids not fat, such tampering with milk may readily be checked.

**Analysis of Sour Milk.**—It occasionally becomes necessary for the analyst to deal with samples of sour milk, especially in the summer-time, when the milk has been brought from a long distance. While the process of lactic fermentation results in the formation of traces of volatile acids, unless the sample has become so badly curdled as to render an even homogeneous mixture of the various parts impossible, a fair determination of the solids and fat can readily be made. Experience has proved that, excepting in instances of milk so badly soured as to have become actually putrid, the analysis of sour milk, if carefully made, should not differ materially from that of the same milk before souring.

Care must be taken to secure an even emulsion of the curd and whey. This may sometimes be accomplished by repeatedly pouring the sample back and forth from one container to another. Again, it is sometimes necessary to use an egg-beater of the spiral wire pattern, which preferably should easily fit the can or milk-container. Unless a fine, even emulsion can be secured, it is impossible to make a satisfactory analysis of sour milk. With such an emulsion results can be relied on.

In measuring portions of the thoroughly mixed sample of sour milk for analysis, a pipette should be used having a large opening.

#### HOMOGENIZED MILK.

This product is prepared from ordinary milk by heating and then passing through the "homogenizer" whereby the fat globules are broken up into smaller globules and the creaming power reduced practically to nil. In the homogenizer the milk is forced, under a pressure varying up to 4000 pounds or more per square inch, into fine jets or sheets which impinge either against each other or against an agate surface thus disrupting the globules. These in normal milk often exceed  $10\mu$  and are mostly 5 to  $6\mu$  while in well-homogenized milk they are mostly only 1 to  $2\mu$ .\*

The machine is also used to emulsify oleo, cottonseed, and other oils and low melting-point fats with skim milk thus furnishing a wholesome food

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\* Baldwin, Am. Jour. Pub. Health, 6, 1916, p. 862.



for calves, hogs, and even human beings, although the temptation to market the products dishonestly has not always been resisted. Homogenized mixtures have also been used in cream, condensed milk, and ice cream.

**Analysis of Homogenized Milk.**—It has been demonstrated by Richmond\* that the Adams paper coil method gives low percentages of fat with homogenized milk while the Röse-Gottlieb, Werner-Schmidt, and Gerber methods are satisfactory. Other constituents are determined as in ordinary milk.

Distinctions from untreated milk are based on the size of the fat globules as above noted, also on physical constants, particularly the viscosity.

#### PASTEURIZED MILK.

The analyst may be called on to determine whether or not milk has been pasteurized to conform with municipal or state regulations.

**Detection of Peroxidase.**—The following tests show whether the milk has been pasteurized at 80° C., or higher but, as found by Lythgoe,† are of no value when 63°, which is now deemed sufficient, is employed.

*Dupouy Paraphenylenediamin Method.*‡—Shake 5 cc. of the milk in a test tube with 1 drop of 0.2% hydrogen peroxide solution (containing 1 cc. of concentrated sulphuric acid per liter) and 2 drops of 2% paraphenylenediamin. If the milk becomes blue immediately it has not been heated to 78° C.; if it becomes gray-blue immediately or within half a minute it probably has been heated to 79–80°; while if it remains white or becomes a faint violet-red it has been heated above 80°.

*Wilkinson and Peters Benzidine Method.*§—To 10 cc. of the milk add 2 cc. of a 4% alcoholic solution of benzidine and 2–3 drops of glacial acetic acid, or an amount just sufficient to coagulate the milk, and shake. Add cautiously to the mixture 2 cc. of 3% hydrogen peroxide solution, allowing the reagent to run down the sides of the test tube. With raw milk or milk heated below 78° C. an intense blue color appears at once; with milk heated at 80° or higher no color appears.

*Other Tests* are the original Arnold guaiac test|| and its modifications and the Roi and Köhler iodide-starch test.¶

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\* Analyst, 31, 1906, p. 218.

† Jour. Ind. Eng. Chem., 5, 1913, p. 922.

‡ Dupouy, Rep. pharm. III, 9, 1897, p. 206; Storch, Copenhagen Exp. Sta. Rep., 1898.

§ Jour. Dep. Agric., Victoria, 6, 1908, p. 251.

| Jahr. Königl. Tierärz. Hochsch., 1880–1882, p. 161.

¶ Milch. Ztg., 31, 1902, pp. 17, 113.



**Detection of Aldehyde Reductase.**—*Schardinger Method*. \*—To 20 cc. of the milk add 1 cc. of a reagent consisting of 5 cc. of a saturated alcoholic solution of methylene blue, 5 cc. of 40% formaldehyde, and 190 cc. of water. Place in a water-bath kept at 45–50° C., and note the time required for decolorization. Lythgoe found that decolorization with raw milk took place in 5 minutes, while with milk pasteurized at 63° for 35 minutes, kept not longer than 2 days, it did not take place in 20 minutes.

#### FERMENTED MILK.

**Yogurt** is a Bulgarian product, prepared with a starter known as *Maya*, which, because of the longevity of the natives who subsist to a large degree on it, has come to be regarded as a kind of elixir of life. The souring of the milk is caused by *Bacillus Bulgaricus* which, like domestic yeast and butter starters, is perpetuated by primitive methods, although other bacteria take part in the changes. Cultures of the bacillus in tablet and liquid form are now on the American market with which the beverage commonly known as buttermilk is prepared either by the dairyman or the housewife from milk or skim milk.

The characteristic constituent is lactic acid, both the dextro and levorotary forms, produced at the expense of a portion of the lactose.

Other acid products, similar to yogurt, are **Leben** of Egypt, **Giöddu** of Sicily (**Cieddu** of Sardinia), **Dadhi** of India, and **Tätté** of Scandinavia.

**Kumiss** is indigenous to Central Asia and the Steppes region of Russia where it is prepared from mare's, camel's, and ass's milk. The alcoholic fermentation is caused by a peculiar yeast that acts directly on the lactose, although bacteria also play a part. With us kumiss is a preparation of cow's milk used chiefly as a therapeutic food, the alcohol being commonly generated by ordinary yeast acting on added glucose or sucrose. In addition to alcohol some lactic acid is also formed from the lactose, the proteins are more or less peptonized or otherwise acted on, butyric acid is liberated, and esters are formed. Dr. L. L. Van Slyke has kindly communicated the following as an average analysis of kumiss made from cow's milk:

Total Solids.	Lactose.	Alcohol.	Acidity.	Total Nitrogen.	Casein Nitrogen.
11.00	5.00	1.00	0.30	0.65	0.55

Alcoholic beverages similar to kumiss are **Kefir** prepared in the Caucasus from cow's, sheep's, or goat's milk, using so-called "Kefir grains"

\* Zeits. Unters. Nahr. Genussm., 5, 1902, p. 1113.



which bear much the same relation to the product as yeast cakes do to bread, and **Mazun** made in Armenia from sheep's, goat's, or buffalo's milk. Ginzberg\* has studied the chemical changes which take place in the preparation of both kumiss and kefir, as well as their imitations.

**Analysis of Fermented Milks.**—*The sampling* requires special care owing to the more or less curdled or granular condition. Lumps of curd may be rubbed through a sieve while lumps of fat, such as occur in butter-milk, may be strained out, weighed, and separately analyzed. In special cases the whole sample may be neutralized with ammonia, taking account of volumes.

*Total Solids, Total Protein, Casein, Albumin, Other Nitrogenous Constituents, Lactose, and Ash* are determined by the methods described under milk with such minor modifications as the nature of the substance may require. For example the casein, already partially or completely precipitated, requires only a small addition of acetic acid, if any. Again since lactose is present in only small amount, a correspondingly larger quantity of this milk may be polarized.

*Fat* is best extracted by the Röse-Gottlieb method after neutralizing the free acid. Obviously ether extraction of the acid material whether or not evaporated to dryness would yield fat contaminated with lactic acid. Centrifugal methods should be employed only when checked against the standard method.

*Total Acids* are titrated directly using phenolphthalein as indicator. *Volatile Acids* and *Alcohol* are distilled together and the former titrated; the slightly alkaline liquid is then redistilled and the alcohol determined.

#### CONDENSED MILK.

Canned condensed milk has become a very important article of food, its use having increased greatly during recent years. The universally accepted meaning of the term "condensed milk" in the United States is milk both condensed and preserved with cane sugar, being what is commonly known in England as "preserved milk." The unsweetened variety is termed "evaporated milk" and sold as such.

**Condensed Milk**, or more properly sweetened condensed milk, is prepared by adding cane sugar to whole milk, usually with previous pasteurization, and evaporating in a special form of vacuum pan to a thick consistency. A considerable quantity is sold to large consumers in bulk,

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\* Biochem. Zeits., 30, 1910, pp. 1, 25.



in which form it keeps indefinitely by reason of the large percentage of sugar, but for domestic use it is commonly packed in hermetically sealed cans.

*Composition.*—Various standards committees agree in placing 28% of milk solids and 8% of fat as the minimum limits. As Hunziker has noted manufacturers are not likely to allow their products to drop below 28% of milk solids as that percentage is essential for holding the sugar in suspension without which the product would not be readily marketable. Not infrequently, however, the percentage of fat falls below 8% indicating that skim milk or an abnormally poor product was used. As at no time during the process the heating is carried on at a high temperature, the evaporation may be continued until the percentage of milk solids is raised to considerably over 30% without danger of curdling.

Upward of 350 samples, representing 110 brands, were analyzed in full by the Massachusetts State Board of Health in the course of eight years. As some of the samples were obviously prepared from partially or wholly skimmed milk, maximum, minimum, and average figures have no significance in judging the composition of the genuine product, but the selected analyses of a few typical brands given in the following table are instructive:

COMPOSITION OF SWEETENED CONDENSED MILK.

Points to be Noted.	Total Solids, Per Cent.	Water Per Cent.	Milk Solids, Per Cent.	Cane Sugar, Per Cent.	Lactose, Per Cent.	Proteins, Per Cent.	Fat, Per Cent.	Ash, Per Cent.	Fat in Original Milk, Per Cent.
High in fat, much added sugar.....	79.17	20.83	31.32	47.85	9.57	7.95	12.00	1.80	4.60
High fat, low milk sugar...	68.70	32.30	30.27	38.43	6.38	10.70	11.46	1.73	5.63
Low fat, high milk sugar, low proteins.....	69.30	30.70	31.83	37.47	16.75	6.34	7.20	1.54	2.77
Normal constituents throughout.....	74.29	25.71	32.37	41.92	11.97	8.46	10.65	1.29	4.56
Condensed from skimmed milk.....	69.30	30.70	29.15	40.15	11.89	12.15	3.06	2.05	1.11
Condensed from centrifugally skimmed milk.....	69.06	30.94	25.88	43.18	11.55	11.78	0.09	2.46	Trace

**Evaporated Milk**, or unsweetened condensed milk, formerly erroneously branded evaporated cream, differs from the sweetened variety in that it does not contain added sugar and therefore must be marketed in sterilized form if not required for immediate use.

Formerly 28% of milk solids containing at least 27.5% of fat was required but careful investigations by Patrick, Hunziker, and others showed that it was impracticable to comply with this standard in all regions and at all seasons, without the milk curdling. At present 25.5% of solids and



7.8% of fat (the latter percentage being of the evaporated milk and not of the milk solids) are recognized as the minimum limits regardless of conditions.

Mohan \* states that swells, flat sours, and sweet curdling are due to understerilization, while other forms of curdling are due to the action of heat on milk with high solids and acidity, and the hard granules sometimes found at the bottom of cans, chiefly to calcium phosphate precipitated by over evaporation.

*Composition.*—The following typical analyses made at the Massachusetts State Board of Health are selected from about 30 representing 8 brands:

COMPOSITION OF UNSWEETENED CONDENSED MILK.

Points to be Noted.	Total Solids, Per Cent.	Water, Per Cent.	Lactose, Per Cent.	Proteins, Per Cent.	Fat, Per Cent.	Ash, Per Cent.	Fat in Original Milk, Per Cent.	No. of Times Condensed.
High in fat.....	36.00	64.00	10.65	11.63	12.00	1.72	4.61	2.6
Low in proteins.....	31.25	68.75	13.40	7.02	9.60	1.23	4.18	2.3
Normal constituents throughout.	28.16	69.24	9.85	8.66	8.10	1.55	3.68	2.2
Condensed from skimmed milk..	35.17	64.83	13.90	15.37	4.20	1.70	1.28	3.3

A summary of analyses of 12 brands found on sale in the State of Maine during the year 1916 follow:†

	Water.	Fat.	Lactose.	Protein.	Ash.
Maximum.....	75.93	8.84	11.45	7.17	1.65
Minimum.....	72.02	7.62	9.13	5.71	1.34
Average.....	73.35	8.13	10.31	6.69	1.52

McGill ‡ gives in the table on page 178 the averages of results obtained during the years 1910 and 1915 on 16 brands collected in Canada:

**Adulteration.**—Aside from such foreign substances as may be present in the original milk without the knowledge of the manufacturer, such as preservatives and colors, the only common form of adulteration is the use of skim milk, although homogenized foreign fats are sometimes used to make up for the deficiency. Watering, as it entails greater labor in evaporation, would not be practiced by the manufacturer. If the milk furnished him is watered the defect is corrected by evaporation.

\* Jour. Soc. Chem. Ind., 34, 1915, p. 109.

† Maine Agr. Exp. Sta., Off. Ins., p. 76.

‡ Lab. Int. Rev. Dept. Canada, Buls. 208 and 305.



1910.			1915.		
Number of Samples.	Solids.	Fat.	Number of Samples.	Solids.	Fat.
3	25.29	5.92	6	22.63	6.74
8	29.02	7.52	30	26.52	7.51
2	23.86	6.74	3	25.72	7.19
1	30.20	8.12	1	22.35	6.48
1	22.04	5.64			
.....	.....	.....	2	25.53	6.62
11	27.47	7.27	12	27.14	7.67
1	24.64	6.00			
6	26.97	6.70	31	26.11	7.21
.....	.....	.....	2	25.39	6.39
.....	.....	.....	6	26.08	7.00
.....	.....	.....	2	27.25	7.45
.....	.....	.....	1	21.92	6.21
12	26.64	6.94	60	26.54	7.44
.....	.....	.....	19	25.55	6.87
.....	.....	.....	3	23.47	0.26

## ANALYSIS OF CONDENSED MILK.

**Preparation of the Sample.**—Mix the sample thoroughly, best by transferring the entire contents of the can to a large evaporating dish and working it thoroughly with a pestle till homogeneous throughout. Weigh 40 grams of the mixed sample, preferably in a tared weighing-tray for sugar analysis, transfer by washing to a graduated 100-cc. sugar flask, and make up to the mark with water.

Another method \* is to weigh the can and contents together, remove the contents to a liter flask with tepid water, dry the can, and subtract its weight from that previously obtained. As the weight of the contents varies this method involves more calculation.

**Determination of Total Solids.**—*Gravimetric Method.*—Dilute an aliquot part of the mixed solution further with an equal amount of water and pipette 5 cc. of the diluted mixture, corresponding to 1 gram of the sample, into a tared platinum dish, such as is used for ordinary milk, and rinse the pipette into the dish by means of a wash-bottle. Evaporate, dry at the temperature of boiling water and weigh as in the case of milk (p. 119).

The character of the residue should be noted. It should not, excepting in the case of a skimmed milk, be caked down hard and glossy on the

\* Conn. Agric. Exp. Sta. Rep. 1904, p. 133.



bottom of the dish, but, if the operation is properly carried out, should have a well-separated fat layer at the top, and the residue should resemble in appearance that from ordinary milk. This result is accomplished by the extreme dilution of the sample.

*Calculation Methods for Evaporated Milk.*—Hunziker's formula \* is as follows:

$$T = \left[ \left( \frac{145.5}{145.5 - B} \right) \times 1,000 - 1,000 \right] \times \frac{1}{4} + 1.2 \times f,$$

in which  $B$  is the Baumé reading at 60° F. and  $f$  the per cent of fat. The hydrometer reading is calculated to 60° F. by adding 0.0313 for every degree over that temperature.

Evenson's modification † of the Babcock formula follows:

$$T = \frac{L - 0.5}{4} + 1.2 \times f,$$

in which  $T$  is the total solids,  $L$  the Quevenne reading at 15.5° C. after holding the sample at 37–40° C. 45 minutes, and  $f$  the per cent of fat.

**Determination of Fat in Sweetened Condensed Milk.**—It has long been known that fat can not be accurately determined in sweetened condensed milk either by extraction after evaporation, as in the asbestos or paper coil methods, or by the ordinary centrifugal methods. In the former case the sugar encloses particles of fat and prevents their contact with the ether while in the latter case it chars with the acid and gives a black fat column. In exact work the *Röse-Gottlieb Method* (p. 193) should be used but for many purposes the two following modifications of the Babcock method are sufficiently accurate.

*Leach Method.*‡—In a Babcock milk bottle with a mark on the bulb showing a volume of 17.6 cc., place 25 cc. of the diluted sample, add 4 cc. of copper sulphate solution of the strength used for Fehling solution, and allow to stand some minutes without shaking. Add water nearly to the neck, shake thoroughly, and whirl without heating in a centrifuge until the precipitated proteins, carrying with them the fat, have entirely settled. Remove the clear liquid, add water nearly to the neck, break up the lumps

\* Ind. Agr. Exp. Sta. Rept., 1913, p. 43.

† Jour. Ind. Eng. Chem., 9, 1917, p. 499.

‡ Mass. State Bd. Health Rept., 1896, p. 630; Jour. Amer. Chem. Soc., 22, 1900, p. 589. Slight changes in the manipulation which Winton has found desirable are given in the above description.



with a wire, shake, and again whirl. After removing the clear liquid repeat once again the addition of water, shaking, whirling, and decantation. Finally add water up to 17.6 cc., mix thoroughly, and proceed as in the usual Babcock method. To obtain the percentage of fat, multiply the reading by 18 and divide by the grams of condensed milk in the aliquot taken.

*Farrington Method.\**—Weigh 40 to 60 grams of the sample into a 200-cc. flask, dissolve in 100 cc. of water, make up to the mark, and shake. Pipette 17.6 cc. into a milk test-bottle, add 3 cc. of sulphuric acid of the strength used for the test, and shake thoroughly. Whirl for 6 minutes at 1000 revolutions per minute in a steam-driven turbine centrifuge in which the chamber reaches a temperature of about 93° C., pour off cautiously the clear solution, add 10 cc. of water, shake, then add 3 cc. of acid, whirl, and decant a second time. Shake the curd with 10 cc. of water and proceed as in the ordinary Babcock test, calculating to the weight of sample taken.

**Determination of Fat in Evaporated Milk.**—The finely curdled particles of casein formed during sterilization enclose fat which is removed with difficulty by ether extraction. Hunziker and Spitzer† found that, after removing the greater part of the casein from the Adams coils by dilute acetic acid, extraction for 8 hours gave the full amount of fat. The asbestos and sand methods are preferable to the Adams method and thorough distribution by dilution facilitates the extraction. Extensive investigations by Patrick and others indicate that the Röse-Gottlieb method gives the best results.

The curd flocks are dissolved with difficulty in the Babcock acid, hence clear readings and the full amount of fat are not always obtained by the usual process. The Hunziker and Spitzer Method, by the use of a quarter quantity of the sample and hot 1 : 1 acid in filling the test-bottles, largely obviates these defects. Utt‡ finds additional heating essential and Bigelow and Fitzgerald,§ in their modification, use 9 grams of the sample, read to the bottom of the meniscus, and add 15% to the result.

As such factors as temperature, thoroughness of mixing, period of action, and method of reading influence the results, the analyst should test his procedure against a standard method or with standard samples derived from milk of known composition evaporated to a definite concentration.

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\* Amer. Chem. Jour., 24, 1900, p. 267.

† Ind. Agric. Exp. Sta. Bul. 134, 1909, p. 591.

‡ Jour. Ind. Eng. Chem., 5, 1913, p. 168.

§ Res. Lab. Nat. Canners' Assn. Bul. 5, 1915, p. 8.



**Determination of Protein.**—Proceed with an aliquot of the diluted sample as described under milk (p. 132), calculating the protein from the total nitrogen or determining it directly by the Ritthausen method.

**Determination of Lactose.**—*Volumetric Method.*—Pipette 25 cc. of the 40% solution into a 500 cc. flask and proceed as directed under milk (p. 137).

In calculating the percentage of lactose use the following formula:

$$L = \frac{100 \times 0.067}{S \times 0.02},$$

in which  $L$  is the per cent of lactose and  $S$  the number of cubic centimeters of 40% solution required to reduce 10 cc. of Fehling solution. Calculation may be avoided by the use of the following table:

PER CENT MILK SUGAR CORRESPONDING TO NUMBER OF CUBIC CENTIMETERS USED.

Strength of solution 2 grams in 100 cc.

Cu. Cm.	Per Cent.	Cu. Cm.	Per Cent.	Cu. Cm.	Per Cent.	Cu. Cm.	Per Cent.
18.0	18.61	25.0	13.40	32.0	10.47	39.0	8.59
18.5	18.10	25.5	13.14	32.5	10.31	39.5	8.49
19.0	17.63	26.0	12.89	33.0	10.15	40.0	8.37
19.5	17.18	26.5	12.64	33.5	10.00	40.5	8.27
20.0	16.75	27.0	12.41	34.0	9.85	41.0	8.17
20.5	16.34	27.5	12.18	34.5	9.71	41.5	8.07
21.0	15.95	28.0	11.97	35.0	9.57	42.0	7.98
21.5	15.58	28.5	11.75	35.5	9.43	42.5	7.88
22.0	15.22	29.0	11.55	36.0	9.30	43.0	7.78
22.5	14.89	29.5	11.35	36.5	9.17	43.5	7.70
23.0	14.56	30.0	11.16	37.0	9.05	44.0	7.61
23.5	14.25	30.5	10.89	37.5	8.93	44.5	7.53
24.0	13.95	31.0	10.80	38.0	8.81		
24.5	13.67	31.5	10.63	38.5	8.70		

**Gravimetric Methods.**—Lactose may be determined in the 40% solution of the condensed milk by the O'Sullivan-Defren method (p. 137), the Soxhlet method (p. 137), or the Munson and Walker method (p. 138), the solution being treated exactly as if it were milk.

It has long been known that the percentages of lactose by copper reduction in sweetened condensed milk are somewhat high owing to the presence of sucrose. This appears to be due to the sucrose itself rather than to reducing sugars present in it as impurities. Winton found, in an



attempt to determine the corrections for different proportions of sucrose, that while agreeing results could be obtained on solutions of pure lactose, when a definite amount of sucrose was added the figures were not merely higher but were discordant even when the heating and other conditions were apparently the same. The experience of Knight and Formanek would seem to indicate that greater accuracy can be secured by directly determining the sucrose and obtaining the lactose by difference.

**Cane Sugar.**—This is ordinarily obtained by difference, deducting the milk solids (the sum of the milk sugar, proteins, fat, and ash) from the total solids first obtained.

When only the sucrose is desired this may be determined by polarization. The Knight and Formanek method depends on double dilution (see Wiley method, p. 136) to correct for the bulk of the precipitate and on a modified Clerget formula to eliminate the rotatory influence of the lactose. Rakshit\* destroys the lactose entirely by means of a measured quantity of Fehling solution calculated from a volumetric determination.

*Knight and Formanek Method.*†—Make the entire contents of the usual 12 to 15-ounce can up to 500 cc. Place 50 and 100-cc. aliquots in 200-cc. flasks and clarify with 1.7 cc. of 5% phosphotungstic acid solution and 2.1 cc. of 25% normal lead acetate solution for each 10 grams of the sample in the aliquot, shaking well after adding each reagent. Make up to the mark, shake again and filter. To the filtrates, measuring about 100 cc., add potassium oxalate crystals in 0.1-gram portions with constant shaking until a curdy precipitate forms which quickly settles leaving a clear liquor. Usually 0.5 gram is sufficient; a large excess should be avoided. Filter again using hardened filters with 3 to 5 grams of fuller's earth in the apex and test the first 10 cc. with potassium oxalate. Polarize at exactly 20° C. preferably in a Bates instrument set for maximum sensitivity and using a bichromate cell. Multiply the reading of the dilute solution by 4 and subtract from the product the reading of the stronger solution. The difference is the direct polarization corrected for the volume of the precipitate.

Pipette 50-cc. portions of the filtrates into 100-cc. flasks and invert with 5 cc. of concentrated hydrochloric acid by standing over night at 20 to 25° C. Add a few drops of phenolphthalein solution and strong sodium hydroxide solution until a pink color appears, then a few drops of N/10 hydrochloric acid until the color disappears. Make up to the

\* Jour. Ind. Eng. Chem., 6, 1914, p. 307.

† Ibid., 8, 1916, p. 28.



mark, cool at room temperature and polarize as before using 400 mm. tubes for instruments other than Bates. Subtract the reading of the strong solution from four times that of the weak and multiply the difference by 2 (except when 400-mm. tubes were used) thus obtaining the invert polarization ( $P'$ ) corresponding to the direct polarization ( $P$ ). The per cent of sucrose ( $S$ ) is obtained by the following formula:

$$S = \frac{26000(P - P')}{W(141.7 - T/2)},$$

in which  $W$  is the weight of condensed milk in the can and  $T$  is the degrees Centigrade of the invert readings.

**Detection of Foreign Fats and Oils.**—The invention of the homogenizer has led to the preparation of emulsions of oleo, cotton seed and other oils, as well as of butter fat, with skim milk and milk in imitation of whole milk and cream and the use of such homogenized products in condensed milk and ice cream. This substitution is detected by the separation and examination of the fat as follows:

*Paul's Method.*\*—Dilute 100 grams of the material with 300 cc. of water, heat to boiling and add slowly, while boiling, 25 cc. of Fehling's copper sulphate solution.

Filter through a filter paper on a Büchner funnel, wash three times with hot water and allow to suck dry. Remove filter and precipitate from the funnel, break into small pieces, dry over night at room temperature and grind with about 25 grams of anhydrous copper sulphate.

Place a layer of anhydrous copper sulphate in the bottom of the inner tube of a Johnson extractor (p. 54), then add the powdered mixture and place a loose plug of cotton on the top. Connect the extractor with a flask, pour 50 cc. of ether through the mixture and proceed as usual with the extraction.

Dry the fat as quickly as possible and weigh. Determine the refractive index, volatile fatty acids and such other constants as seem desirable (Chapter XIII).

**Calculation of Fat in Original Milk.**—The "fat in original milk," as given in the tables on pages 176 and 177, was calculated by assuming a percentage of solids not fat of 9.3 in the original milk, this being the standard fixed by the Massachusetts law. Calculate first the fat and the milk solids to the basis of the cane-sugar-free sample. This is done by divid-

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\* U. S. Dept. of Agric., Bur. of Chem., Circ. 90, p. 10.



ing the per cent of each as found in the sample by 100 less the percentage of cane sugar, and multiplying the result by 100. Ascertain the difference between the milk solids and the fat thus obtained in the cane-sugar-free sample, and divide this percentage of milk solids not fat by 9.3. The result is the "number of times condensed" (if cane sugar were not present as a diluent).

The per cent of fat in the cane-sugar-free sample, divided by the number of times condensed, as above obtained, gives the percentage of fat in the original milk.

The above calculation from the solids not fat of the factor designated as "the number of times condensed," necessitates determinations of fat, ash, proteins, and milk sugar, in fact, a complete analysis of the sample.

A simpler method of calculating the "number of times condensed," involving determinations of fat and ash only in the sample, consists in dividing the per cent of ash found in the condensed milk by 0.7, this figure being the assumed ash of normal, standard milk. Then, by dividing the fat in the sample by the "number of times condensed" as last calculated, the result is the fat in the original milk. If this is found to be well below 3%, there is reason to suspect that skimmed milk was used in its preparation.

The "fat in the original milk" as thus calculated is, of course, an arbitrary factor and is useful only in deciding whether or not skimmed milk has been used in preparing the sample. By assuming the above very reasonable figures for the solids not fat, or for the ash of natural milk (according to which method is used for calculation), it is readily seen that the highest result is obtained for the "fat in the original milk" and hence the benefit of the doubt as to the use of skimmed milk is given to the manufacturer.

#### MILK POWDER.

There are numerous brands of desiccated milk or milk powder on the market, sold in bulk and by the can, and largely used by bakers and manufacturers of milk chocolate. Many of these, purporting to contain all the ingredients of milk excepting water, have been found to be pulverized dried skimmed milk.

In the table on page 185 are given the maximum and minimum figures obtained by Fleming\* in 2 samples of cream powder and 1 of whey powder,

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\* Jour. Ind. Eng. Chem., 4, 1912, p. 543.



and by Stewart\* in 7 samples of whole milk powder, 1 of half skim milk powder, and 8 of skim milk powder.

	Cream Powder.*	Whole Milk Powder.†	Half Skim Milk Powder.†	Skim Milk Powder.†	Whey Powder.*
Water.....	0.81-0.76	5.52- 1.46	4.58	8.00- 4.76	1.40
Fat.....	67.64-53.08	30.00-23.55	10.00	1.86- 0.34	0.60
Lactose.....	26.04-15.92	41.23-34.48	41.54	54.07-48.41	77.20
Total protein....	16.89-12.21	27.69-23.92	30.90	35.72-31.96	12.50
Soluble albumin..	.....	6.06- 1.60	2.23	8.03- 1.25	10.20
Ash.....	3.78- 2.67	6.33- 5.33	6.98	8.43- 7.26	9.10

Analyses by Fleming.

† Analyses by Stewart.

#### ANALYSIS OF MILK POWDER.

The methods designed for milk apply in general to milk powder, observing certain precautions:

**Determination of Fat.**—The Röse-Gottlieb and Werner-Schmidt methods are applicable. Neither extraction of the dry material with absolute ether nor the Babcock method yields the full amount of fat. McLellan † obtained satisfactory results extracting with concentrated (not absolute) ether (sp.gr. 0.72), extending the extraction through 2 days, and soaking over night before each extraction. Fleming also uses concentrated ether but finds 16 hours' extraction without soaking sufficient.

*Redmond Modification of the Babcock Method.*‡—Weigh 2.5 grams of the powder into a clean dry Babcock milk bottle, using a small funnel to aid in transferring. Add 31 cc. of dilute sulphuric acid (395 cc. of conc.  $H_2SO_4$  diluted to 1 liter) and heat in a dish of gently boiling water, with frequent shaking, until all the powder is dissolved and the mixture is dark brown which usually requires 7 to 10 minutes. Remove from the water, add 12 cc. of sulphuric acid (sp.gr. 1.82-1.83), mix thoroughly, and proceed as in the usual Babcock method. Place in water at 55 to 80° C., read the fat to 0.05 on the scale and multiply the reading by 7.2.

**Determination of Lactose.**—Follow the usual methods, grinding any lactose crystals that may be present to a powder, and dissolving in warm water.

\* Eighth Int. Cong. App. Chem., 18, 1912, p. 329.

† Analyst, 31, 1908, p. 353.

‡ Jour. Ind. Eng. Chem., 4, 1912, p. 544.



## CREAM.

**Composition.**—Cream varies in composition according to the method by which it is obtained, i.e., whether (1) by allowing it to separate from the milk set in shallow pans, whence it is removed by hand-skimming, (2) by setting in deep vessels surrounded by cold water (as for example in the "Cooley" creamer) the skimmed milk being commonly drawn off from below, or (3) by the centrifugal separator. Most of the heavy cream found in the market at the present time is the product of the third or separator process. Analyses of different kinds of cream follow:

COMPOSITION OF CREAM.

Character of Cream.	Number of Analyses.	Authority.		Water.	Proteins.	Fat.	Sugar.	Ash.	Total Solids.	Solids not Fat.
By natural separation. ....	46	König	Mean	68.82	3.76	22.66	4.23	0.53	31.18	8.42
By centrifugal separator,										
"Heavy" cream. ....	18	Leach	Maximum	54.80	....	46.40	....	....	53.24	8.50
			Minimum	46.76	....	38.10	....	....	45.20	4.20
			Mean	51.68	....	42.02	....	....	48.32	6.30
"Light" cream. ....	18	Leach	Maximum	83.29	....	21.60	....	....	29.50	9.30
			Minimum	70.50	....	8.60	....	....	16.71	7.22
			Mean	77.89	....	13.86	....	....	22.11	8.25

**U. S. Standards.\***—*Standard Cream* is cream containing not less than 18% of milk fat. *Standard Evaporated Cream* is cream from which a considerable portion of water has been evaporated.

**Adulteration of Cream.**—In some localities fat standards are fixed for cream both "heavy" and "light," those falling below such standards being deemed adulterated.

**Foreign Fats.**—Oleo oil, possibly other fats, "homogenized" or emulsified with milk or skim milk, is now being substituted for true cream. A product known as "Syntho" belongs in this class but is sold by its manufacturers under its true name.

**Preservatives.**—The same preservatives are employed in cream as in milk.

**Gelatin.**—The author has detected this substance in cream sold in Massachusetts. It serves as a thickener and is sometimes sold in powder form mixed with boric acid.

\* U. S. Dept. of Agric., Off. of Sec., Circ. 19.



**Sucrate of Lime in Milk and Cream.**—Pasteurizing reduces the consistency of cream so that its apparent richness and its value for certain culinary preparations is impaired. Babcock and Russell\* have shown that sucrate of lime ("viscogen") may be used to thicken such cream, but insist that the treated product be sold under a distinctive name, such as "visco-cream" or "pasteurized visco-cream."

To prepare "viscogen" dissolve  $2\frac{1}{2}$  parts by weight of cane-sugar in 5 parts of water, add, after straining, 1 part of quicklime slaked in 3 parts

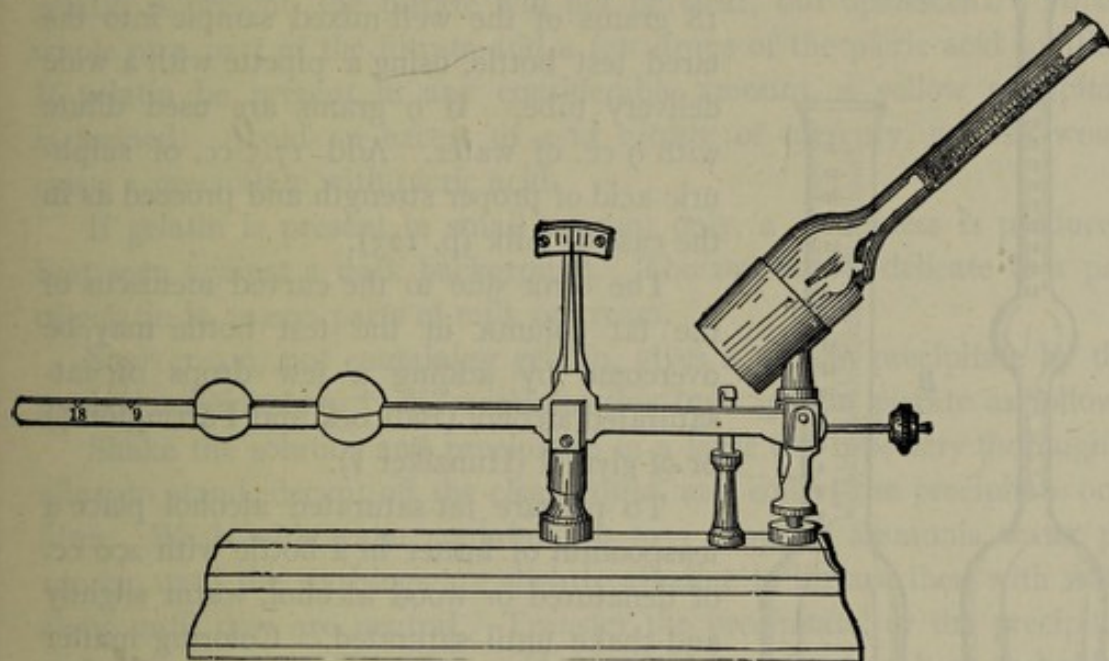


FIG. 53.—A Babcock Cream-test Scale.

of water; shake, allow to settle, siphon off the supernatant liquid, and bottle. For thickening cream use two-thirds of the amount required to neutralize its acidity. It will also thicken milk and condensed milk.

#### ANALYSIS OF CREAM.

Total solids, ash, sugar, proteins, and fat (gravimetric) are determined by the methods used in milk analysis (pp. 119-138).

**Determination of Fat.**—*Babcock Process.*—Owing to the viscosity of cream and its variation in density strictly accurate results can be secured only by weighing the sample. Fig. 53 shows a cream scale provided with a sliding poise for balancing the bottle and a second for weighing the cream. If a large number of tests are to be made, a balance for weighing

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\* Wisconsin Exp. Station, Bul. 54.



several samples on each pan or the Wisconsin hydrostatic cream balance will be found convenient.\* The latter, devised by Babcock and Farrington,† is constructed on the principle of the lactometer. It is provided with a pan on the top of the stem for holding the test bottle and weights.

Two forms of test bottles are shown in Fig. 54. Others with graduations up to 50% are also obtainable.

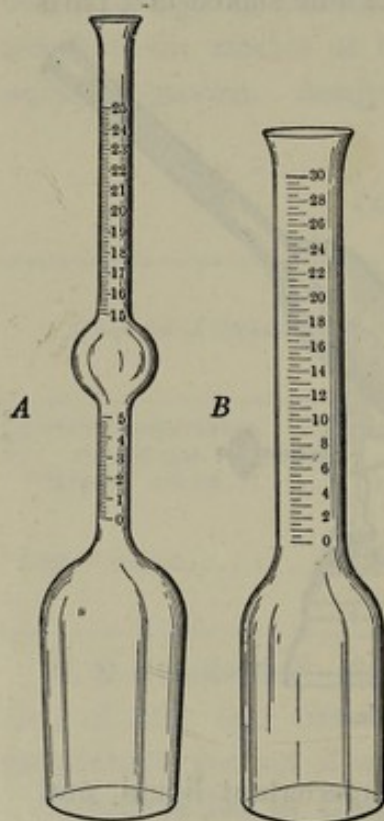


FIG. 54.—Varieties of Babcock Test Bottle for Cream.  
A, Bartlett Bottle; B, Winton Bottle.

The process is as follows: Weigh 9 or 18 grams of the well-mixed sample into the tared test bottle, using a pipette with a wide delivery tube. If 9 grams are used dilute with 9 cc. of water. Add 17.5 cc. of sulphuric acid of proper strength and proceed as in the case of milk (p. 123).

The error due to the curved meniscus of the fat column in the test bottle may be overcome by adding a few drops of fat-saturated alcohol (Babcock and Farrington ‡) or of glymol (Hunziker §).

To prepare fat-saturated alcohol place a teaspoonful of butter in a bottle with 200 cc. of denatured or wood alcohol, warm slightly and shake until saturated. Coloring matter may be added to further facilitate the reading. Glymol may be colored with alkanet root.

**Detection of Foreign Fats.**—Determine the refractive index and the volatile fatty acids of the fat obtained by the Babcock method.

**Detection of Preservatives.**—See pp. 162-169.

In testing for formaldehyde, using ferric chloride and hydrochloric acid, the sample should be diluted with an equal volume of water, heated with the reagents in a casserole but finally poured into a test tube to observe the color.

\* Farrington and Woll, *Testing Milk and its Products*, 20th Ed., pp. 81-83.

† Wisconsin Exp. Station, Bul. 195.

‡ *Loc. cit.*

§ Ind. Agric. Exp. Sta. Bul. 145, p. 193.



**Detection of Gelatin.**—*Stokes Method.*\*—The reagents are as follows:

(1) Acid nitrate of mercury, prepared by dissolving metallic mercury in twice its weight of concentrated nitric acid (sp.gr. 1.42) and diluting with twenty-five times its bulk of water, and (2) a saturated aqueous solution of picric acid.

To about 10 cc. of the cream add the same amount of the acid nitrate of mercury solution and 20 cc. of cold water. Shake the mixture vigorously and allow to rest for five minutes, after which filter. If much gelatin is present, the filtrate will not be clear, but opalescent. To the whole or a part of the filtrate add a few drops of the picric acid solution. If gelatin be present in any considerable amount, a yellow precipitate is formed. Avoid an excess of acid nitrate of mercury, as this would cause a precipitate with picric acid.

If gelatin is present in small amount only, a cloudiness is produced, best seen against a dark background. The reaction is delicate to 1 part of gelatin in 10,000 parts of milk or cream.

Sour cream, not containing gelatin, gives a protein precipitate by this method. Seidenberg † differentiates this from gelatin picrate as follows:

Shake the solution and precipitate in a large test-tube very thoroughly, allow to stand, decant off the clear liquid, and collect the precipitate on a filter. Wash with water containing 2 to 3 drops of ammonia water per 100 cc. until the washings are slightly alkaline to litmus, then with water alone until they are neutral. Transfer the precipitate, or the precipitate and filter, to a small beaker, add 10 to 20 cc. of water, heat to boiling, and filter hot into a test-tube. The filtrate will contain the gelatin picrate but not the protein. Cool and test for gelatin by adding an equal volume of the picric acid solution.

**Detection of Sucrate of Lime.**—This is indicated by the presence of sucrose, in connection with an abnormally high alkalinity of ash and excessive calcium oxide. The tests are as follows:

**Lythgoe's Modification of Baier and Neuman's Test for Detecting Sucrose.**‡—To 25 cc. of milk or cream, add 10 cc. of a 5% solution of uranium acetate, shake well, allow to stand for 5 minutes, and filter. To 10 cc. of the clear filtrate (in the case of cream use the total filtrate, which will be less than 10 cc.) add a mixture of 2 cc. saturated ammonium molybdate and 8 cc. dilute hydrochloric acid (1 part 25% acid

\* Analyst, 22, 1897, p. 220.

† Jour. Ind. Eng. Chem., 5, 1913, p. 927.

‡ Zeits. Unters. Nahr. Genussm., 16, 1908, p. 51.



and 7 parts water), and heat in a water-bath at  $80^{\circ}$  C. for 5 minutes. If the sample contains sugar, the solution will be of a Prussian blue color, which should be compared in a colorimeter with standard Prussian blue solution, prepared by adding a few drops of potassium ferrocyanide to a solution of 1 cc. of 1% ferric chloride in 20 cc. of water.

Occasional samples of pure milk will give a pale blue color, but this can be entirely removed by filtration, the filtrate being green, while the color due to sugar will pass through the filter, giving the usual blue solution. This color, due to a reduction of molybdic acid, is also produced by levulose and dextrose. Solutions of 1 gram of lactose, levulose, dextrose, and sucrose in 35 cc. of water heated with molybdenum reagent for 5 minutes reacted as follows: lactose no color, levulose a heavy blue, sucrose a weaker blue, and dextrose the weakest blue, the intensity of the last three being as 10 : 3 : 1.

Stannous chloride, ferrous sulphate, and hydrogen sulphide give this blue color in the cold, but it disappears on heating except in case the stannous or ferrous salt is present to the extent of at least 1% (calculated as the metal) which amount would coagulate the cream and impart a very disagreeable taste.

As a confirmatory test for sugar, the resorcine test may be applied to the serum prepared with uranium as described above. This test is given by sucrose and levulose, but not by dextrose or lactose.

**Determination of Alkalinity of Ash and Calcium Oxide.**—Weigh 25 grams of cream into a platinum dish, place in an oven at about  $125$  to  $150^{\circ}$  C. over night, and burn to an ash in a muffle at a low-red heat. Dissolve the ash in 20 cc. N/10 sulphuric acid, boil to expel the carbon dioxide, and titrate back with N/10 sodium hydroxide, using phenolphthalein as the indicator. Express results as cc. N/10 acid required to neutralize the ash of 100 grams of cream.

Make the final solution of the above determination acid with acetic acid, heat to boiling, add 1 gram of sodium acetate, and to the clear solution add an excess of ammonium oxalate, boil for a few minutes, filter, and wash with water. Dissolve the calcium oxalate in hot dilute sulphuric acid, and titrate hot with N/10 potassium permanganate. The number of cubic centimeters of N/10 permanganate, multiplied by 0.0112 ( $4 \times 0.0028$ ), gives the percentage of CaO in the sample.

Lythgoe and Marsh\* have calculated the maximum percentages of

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\* Jour. Ind. Eng. Chem., 7, 1910, p. 327.



calcium oxide  $C$  corresponding to the percentages of fat  $F$  within the limits of 15 and 54%. The following formula is based on their figures:

$$C = 0.181 - 0.00246(F - 15).$$

#### ICE CREAM.

For many years a wide variety of iced foods have been made and sold under the general name of ice cream, many of which are largely composed of ingredients other than milk or cream. In the study and classification of foods of such a miscellaneous nature as ice cream, in its popularly accepted meaning, it is not always easy to satisfactorily define and fix standards, either as regards the constituents or the minimum limit for butter fat.

This perplexity, which exists in standardizing all mixtures, is increased in the present instance by a name which suggests merely frozen cream when milk, condensed milk, skim milk, emulsions of butter fat, oleo oil, or cotton-seed oil may be present. Had frozen creamy mixtures come to be known under a generic non-suggestive name they might now pass unchallenged, like cake and pudding, regardless of their food constituents, provided nothing unwholesome was used in their preparation. It has, indeed, been argued that the name ice cream is strictly analogous to names such as cake and pudding, the word cream referring merely to the creamy nature of the product. As precedents for such a usage the names creamed potatoes, chocolate creams, cream of wheat, and *crème de menthe* are cited. While this is an extreme view it serves to illustrate the difficulties of establishing a system of nomenclature that will prevent deception and at the same time not discourage the use of wholesome substitutes, or be more exacting for one class of products than another.

**U. S. Standards.\***—*Ice cream* is a frozen product made from cream and sugar with or without a natural flavoring, and contains not less than 14% of milk fat.

*Fruit ice cream* is a frozen product made from cream, sugar, and sound, clean, mature fruits, and contains not less than 12% of milk fat.

*Nut ice cream* is a frozen product made from cream, sugar, and sound, non-rancid nuts, and contains not less than 12% of milk fat.

These standards have given rise to much controversy and have never become universally recognized. Certain states have established standards of their own.

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\* U. S. Dept. of Agric., Office of Secretary, Circ. 19.



**Classification.**—Washburn \* recognizes two divisions: (1) *Plain ice cream*, which is an uncooked mixture of cream, sugar, and flavoring material, usually with gelatin or some other binder but rarely or never with eggs and (2) *French or Neapolitan ice cream*, which contains eggs and is virtually a frozen custard.

*Water ices* although containing no milk or cream should be mentioned in this connection as they are made by practically the same freezing process and are often packed in bricks in layers alternating with ice cream of different flavors.

Mortensen † extends the classification so as to cover a wide variety of frozen desserts.

**Influence of Ingredients and Process.**—Washburn ‡ after an extensive investigation has reached the following conclusions:

“The flavor is influenced by the fat content of the cream; by its freedom from contamination of all sorts; by a low cream acidity; by the addition of minute quantities of common salt; and by the ripening or aging of the ice cream. A good body is the result of the presence of plenty of fat, but not too much; of the aging and thorough cooling of the cream; and, sometimes, of the use of fillers. A fine texture is promoted by the richness of the cream; by the proper conduct of the freezing process; by the aging of the cream; and, if the goods are not to be used promptly, by the use of a gelatinoid binder. Swell (or overrun) is caused by the incorporation of air into the cream and is affected by the viscosity of the cream; by the rate of freezing; and particularly by the length of time elapsing while the cream is dropping from 34 to 29° F.; and by the speed of the agitating mechanism. The richness or the leanness of the cream within working limits has little effect thereon; neither does the use of gelatin, gum tragacanth or other binders. . . .

“A clean cream is of course essential. Neither a very rich nor a too thin cream should be used, about 22% fat seeming ideal. A day's keeping improves cream, and if it is kept cold so much the better, since the fat globules harden and a better body is obtained. Acidity is to be decried, although unless excessive it is not fatal to success. A pasteurized cream, if allowed to age, may be used to advantage. The homogenation of cream greatly increases its viscosity and tends to better the body, texture and general creaminess of the final product. There is no essential difference

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\* Vt. Agric. Exp. Sta., Bul. 155, 1910, p. 6.

† Iowa Agric. Exp. Sta., Bul. 123, p. 353.

‡ *Loc. cit.*



between centrifugal and gravity creams as used in ice cream making; condensed milk may be used to advantage to better the body and smoothness of the goods.

"A filler is used to give body; a binder to prevent coarse crystallization when held for one day or longer. Starch, flour, eggs, and rennet are used as fillers with greater or less satisfaction, generally less. Gelatin, gum tragacanth, and ice cream powders are used as binders often with good satisfaction; but their use, though legal in Vermont, is forbidden in several states. There appears to be arguments on both sides of the question as to the advisability of the use of binders in commercial cream. The adverse arguments are that inferiority and age are thus concealed, the swell unduly augmented, the use of low grade materials encouraged, insanitary holding conditions favored, and adequate food control rendered difficult. Those advanced in favor of their use are that they prevent granulation and consequent deterioration, discourage the return and re-usage of unsold goods, and assist trade regulations."

**Homogenized Products.**—Unsalted butter emulsified with milk or skim milk is now extensively substituted for true cream in the manufacture of so-called ice cream. Oleo oil and cotton seed oil are also used in such emulsions; neither of these oils is considered permissible in the product sold as ice cream.

**Ice Cream Cones.**—These are cornucopias made of a kind of dry crust used to serve ice cream without a spoon, the cones as well as the ice cream being eaten from the hand. In addition to flour, sugar, and eggs or gelatin, which are proper constituents, they frequently contain saccharin, artificial color and borax, the latter being used to prevent sticking to the mold during baking.

#### ANALYSIS OF ICE CREAM.

**Determination of Fat.**—*Röse-Gottlieb Method.*\*—Prepare a 40% solution, as for condensed milk (p. 178). Of this solution measure 10 cc. into a Röhrig tube † (Fig. 55), or a glass cylinder 2 cm. in diameter and 40 cm. high, to which a narrow siphon can be fitted; dilute with 0.5 cc. of water, add 1.25 cc. of concentrated ammonium hydroxide (2 cc. if the sample is sour) and mix thoroughly. Add 10 cc. of 95% alcohol and shake well. Then add 25 cc. of washed ethyl ether, shake *vigorously*

\* Röse, Zeits. angew. Chem., 1889, p. 100; Gottlieb, Landw. Versuchs-Stat., 40, 1892, p. 1; Patrick, U. S. Dept. Agric., Bur. of Chem., Circ. 66.

† Zeits. Unters. Nahr. Genussm., 9, 1905, p. 531.



for half a minute, add 25 cc. of petroleum ether (p. 55), preferably redistilled below  $60^{\circ}\text{C}$ ., and shake again for half a minute. Let stand twenty minutes or until the upper liquid is clear and its lower level constant.

Draw off as much as possible of the ethereal liquid—usually 0.5 to 0.8 cc. is left—through a diminutive filter into a weighed flask. Extract the liquid remaining in the tube in the same manner as before except that only 15 cc. each of the ethers are used, draw off through the same paper into the flask and wash with a few cc. of the mixed ethers (1 : 1). Evaporate the drawn off and filtered liquid slowly and dry in a boiling-water oven, one hour at a time, to constant weight. The ether used must be tested for residue upon evaporation and a correction introduced if necessary.

The dried and weighed fats should be dissolved in a little petroleum ether; if a residue be found (due to a trace of the aqueous liquid which may have passed the filter) it must be washed in the flask, dried, and its weight deducted from that of the crude fat.

This method is also applicable to condensed milk, cream, milk, skim milk, buttermilk, and whey. With substances of low fat content the second extraction may be omitted, the weight of the fat being increased to correspond to the entire volume of ethereal liquid measured in the tube.



FIG. 55.—Röhrig Tube for Röse-Gottlieb Method.

*White Method.\**—Weigh into a Babcock milk bottle 6 grams of the melted and well-mixed sample, dilute to 18 cc., and add 8 cc. of sulphuric acid in two portions, allowing 2 minutes to elapse before adding the second portion and mixing carefully after each addition. The mixture should be light brown and not black as in the case of milk. If a black color is obtained—due to the violent action on the sugar because of a low percentage of casein—repeat the test using a smaller amount of acid. Whirl in a steam centrifuge for 3 minutes at a high speed then fill to the neck with distilled water at  $65^{\circ}\text{C}$ . If black particles are mixed with the fat shake vigorously for a few seconds, whirl again for 3 minutes, add water so as to bring the fat within the scale, whirl the third time for 2 minutes, and read at  $63^{\circ}\text{C}$ . Multiply the reading by 3 to obtain the per cent of fat.

\* Penn. Agric. Exp. Sta. Rep., 1910, p. 243.



*Halverson*\* also uses only sulphuric acid, added in portions, as a reagent but the details of the process are different from those of the foregoing. The test is made in a special bottle for drawing off the acid liquid after the second whirling.

*Lichtenberg Method.*†—Weigh into a Babcock milk bottle 9 grams of the melted and well-mixed sample. Add 20 cc. of glacial acetic acid (sp.gr. 1.049), mix well, then add 10 cc. of sulphuric acid (sp.gr. 1.83) and mix again. Proceed as in the regular Babcock test reading the fat column from one extreme to the other at 55° C. The reading multiplied by 2 gives the per cent of fat.

Utt‡ follows essentially the same method with minor changes in the proportion of the reagents and details of manipulation.

**Detection of Foreign Fats and Oils.**—Separate and examine the fat as described on page 183.

**Detection of Thickeners.**—*Patrick's Method.*§—Add 25 cc. of water to 50 cc. of the sample, and boil till any thickener present is dissolved. Add 2 cc. of a 10% solution of acetic acid, heat to boiling, add 3 heaping teaspoonfuls of kieselguhr, and after shaking pass at once through a plaited filter. To 3 cc. of the clear filtrate add 12 cc. of 95% alcohol and mix thoroughly, thus precipitating the milk proteins not already removed, and also the gums and some of the gelatin, if much is present. Add 3 cc. of a mixture of 95 cc. of 95% alcohol and 5 cc. of concentrated hydrochloric acid. This acidified alcohol dissolves completely the milk proteins, and, if a clear solution then remains, no gums or vegetable jellies have been used as thickeners. Turbidity does not, however, necessarily indicate presence of a thickener, as it may be caused by a large amount of eggs, or by the souring of the ice cream. Dilute the mixture, if turbid, by adding 3 cc. of water. Any precipitate due to gelatin or eggs will be dissolved at this dilution, but not that due to vegetable gums. If gum tragacanth be present, the precipitate will be stringy and cohesive, especially after shaking, while agar-agar or other vegetable thickeners will cause a fine flocculent precipitate.

Souring of the ice cream sometimes produces a turbidity or precipitate under the above conditions, which is not always dissolved after diluting with water. Formation of such a precipitate (due to sourness) may, however, apparently be prevented by the previous addition of formaldehyde to the sample.

\* Jour. Ind. Eng. Chem., 5, 1913, p. 403. † Ibid., 7, 1915, p. 773.

‡ Ibid., 5, 1913, p. 786.

§ U. S. Dept. of Agric., Bur. of Chem., Bul. 116, p. 26.



*Howard's Test for Gums.*—Precipitate 10 cc. of the melted sample with acetone, and wash with 2 or 3 portions of dilute alcohol, using the centrifuge. Boil the washed residue with 6 to 8 cc. of water and 1 cc. of 10% sodium hydroxide solution for half a minute. Cool, let stand a few minutes, filter, and heat the filtrate to boiling. Add one and one-half volumes of warm alcohol and shake. If agar-agar or gum tragacanth be present, a flocculent precipitate will immediately separate. Disregard a mere turbidity. To prove the absence of any considerable quantity of milk proteins in the precipitate, dissolve in cold water and saturate the solution with ammonium sulphate.

*Gelatin.*—Use the method of Stokes (p. 189) on 10 to 15 cc. of the sample, disregarding a faint cloudiness at the end.

*Starch* is detected by the usual iodine test.

**Detection of Preservatives.**—Formaldehyde and boric acid are tested for as in milk.

**Detection of Colors.**—See Chapter XVII. The colors used are not merely yellows and oranges such as are added to milk, but include also reds, greens, and even blues, coal-tar dyes being most commonly employed.

#### BUTTER.

The value of butter as a food depends almost entirely on its fat content, although minute quantities of protein and milk sugar are also included in its composition.

Hence butter is more logically treated in detail under the heading of fats, Chapter XIII.

#### CHEESE.

**Nature and Composition.**—Cheese consists principally of the coagulum removed in a mass from milk, which has been curdled by the natural souring or by the action of rennet. The separated mass, after being compressed, is allowed to undergo certain changes, which constitute the ripening or curing, due to the action of micro-organisms and enzymes. Cheese is also made from cream, skim milk, and whey.

Besides nitrogenous bodies, fat, ash (including salt), and water, which are its chief constituents, ordinary cheese contains small quantities of lactic acid and lactose. The fat in certain varieties also undergoes some changes such as the liberation of butyric acid and the lactic acid (at least in Swiss cheese) splits up into proprionic and acetic acids.

During the ripening process, which requires [from a few weeks to several months, the characteristic flavor is developed chiefly by the changes



in the proteins, and the digestibility of the cheese is improved. The nature of these changes is little understood, but a variety of complex nitrogenous products are formed, which L. L. Van Slyke divides as follows: paracasein, unsaturated paracasein lactate, paranuclein, caseoses (albumoses), peptones, amides, and ammonia.

Whey cheese, a product of importance in Scandinavian countries, contains lactose as its chief constituent.

**Varieties.**—Well-known cheeses of commerce are often named from districts, towns, or localities where they originated or are still made. They may be classified as cream, whole-milk, or skimmed-milk cheese, according to the quality of the product from which they are made, or again as hard, medium, or soft, according to whether (1) they are pressed, or (2) allowed to drain for days and sometimes weeks without pressure to a firm consistency, or (3) are made in the space of a few hours, being quickly drained on a sieve by hand pressure.

*Cheddar Cheese*, which is the common cheese of the United States (though originally made some 250 years ago in England and still made there), is a type of the hard cheese. *Stilton*, an English cheese, *Emmenthal* and *Gruyère*, Swiss cheeses; *Edam*, a Dutch cheese, and *Pineapple*, an American cheese, belong to the hard class, while *Camembert*, *Brie* and *Neufchatel*, French cheeses, also *Limburger* and *Brick* cheeses are representatives of the soft class. *Roquefort*, made originally from ewe's milk in the French town of that name, and ripened in caves in the mountains, and *Gorgonzola*, an Italian cheese resembling Roquefort, are characterized by the streaks of green mold. In Norway a sweet cheese is made from goat's milk and whey.

The following table, compiled by Woll,\* shows the average composition of various cheeses of commerce, both foreign and domestic:

	Water.	Casein.	Fat.	Sugar.	Ash.
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Cheddar.....	34.38	26.38	32.71	2.95	3.58
Cheshire.....	32.59	32.51	26.06	4.53	4.31
Stilton.....	30.35	28.85	35.39	1.59	3.83
Brie.....	50.35	17.18	25.12	1.94	5.41
Neufchatel.....	44.47	14.60	33.70	4.24	2.99
Roquefort.....	31.20	27.63	33.16	2.00	6.01
Edam.....	36.28	24.06	30.26	4.60	4.90
Swiss.....	35.80	24.44	37.40	.....	2.36
Full cream, mean of 143 analyses.....	38.60	25.35	30.25	2.03	4.07

\* Dairy Calendar, p. 223.



Van Slyke has furnished the following analysis of the nitrogen compounds in a sample of normal American Cheddar cheese six months old and cured at 60° F.:

Per Cent N in Cheese.	Per Cent Water-soluble N.	Per Cent N as Paranucelin.	Per Cent N as Caseoses.	Per Cent N as Peptones.	Per Cent N as Amides.	Per Cent N as Ammonia.
3.80	1.46	0.14	0.22	0.18	0.79	0.13

**U. S. Standards.\***—*Cheese* is the sound, solid, and ripened product made from milk or cream by coagulating the casein thereof with rennet or lactic acid, with or without the addition of ripening ferments and seasoning, and contains, in the water-free substance, not less than 50% of milk fat. By act of Congress, approved June 6, 1896, cheese may also contain added coloring matter.

*Skim-milk Cheese* is defined the same as cheese except that it is made from skim milk, and no minimum percentage of fat in the water-free substance is specified.

**Adulteration.**—Cheese is commonly adulterated in two ways: first, by the partial or total substitution for the milk fat of a foreign fat, as oleomargarine or lard, and, second, by using skimmed milk as a material for its manufacture.

In many localities a standard percentage for fat in cheese is fixed by law, as in the case of the U. S. standard noted above, all samples falling below that standard, unless sold as skim-milk cheese, being deemed adulterated.

Some states have specific standards for varying grades of cheese, classified as to their fat content. Thus under the Pennsylvania law † cheese is divided into five grades, as follows:

Full-cream cheese must contain not less than 32% butter fat.

Three-fourths cream cheese must contain not less than 24% butter fat.

One-half cream cheese must contain not less than 16% butter fat.

One-fourth cream cheese must contain not less than 8% butter fat.

All cheese having less than 8% fat must be branded "Skimmed Cheese."

The term "full cream" as applied to cheese made from whole milk, although quite generally used and legalized by certain state laws, is misleading in view of the fact that true cream cheese is on the market.

\* U. S. Dept. of Agric., Off. of Sec., Circ. 19.

† Penn. Laws, 1901, Act. 95, p. 128.



The following analyses by Leach illustrate the difference in composition between whole-milk and skim-milk cheese:

Varieties of Cheese.	Water.	Fat.	Protein.*	Ash.	Fuel Value per Pound.
Cream (soft).....	37.63	47.40	13.70	1.27	2255
Whole milk (hard).....	21.89	38.00	37.71	2.40	2305
Whole milk (soft).....	55.95	24.00	16.49	3.56	1320
Skimmed milk (soft).....	62.17	15.20	21.36	1.27	1039
Centrifugally skimmed milk (soft).....	72.80	2.00	23.52	1.68	522

\* By difference.

"Filled cheese" is the common name for a product in which a foreign fat, such as oleo oil or lard, has been used. Formerly filled cheese was made in the United States for the export trade but owing to drastic laws it is now practically unknown.

#### ANALYSIS OF CHEESE.

**Sampling.**—If the cheese is spherical or drum-shaped, cut a narrow, wedge-shaped segment, reaching from the circumference to the center, if brick shaped, cut a thin slice through the middle; the rind may be included in the sample or rejected according to the purpose of the analysis. The sample is prepared for analysis by chopping, grating, or kneading until uniform, taking care to weigh before and after the treatment if loss of moisture is sustained.

A less accurate, but for many purposes quite satisfactory, method of sampling is to remove cores from different parts of the cheese with a trier using the rind to stop up the holes. By this procedure the cheese is not materially damaged.

**Determination of Water.**—Dry 5 grams of the sample in a flat-bottomed metal dish 5 cm. in diameter in a boiling-water oven until the weight is practically constant. If ash is subsequently to be determined in the same portion a platinum or porcelain dish must be used.

Winton\* has found that reasonably constant weight is obtained in drying American full cream and skim-milk cheese only after 12 to 18 hours while in drying Roquefort losses, evidently other than water, continued for days. For practical purposes drying for 12 hours is sufficient.

**Determination of Fat.**—*Lythgoe's Modified Babcock Method.*—Weigh accurately about 6 grams of the sample in a tared beaker. Add 10 cc. of boiling water, and stir with a rod till the cheese softens and an even

\* U. S. Dept. of Agric., Div. of Chem., Bul. 35, 1892, p. 13.



emulsion is formed, preferably adding a few drops of strong ammonia to aid in the softening and emulsifying, and keeping the beaker in hot water till the emulsion is tolerably complete and free from lumps.

If the sample is a full-cream cheese, which is usually evident from its taste and appearance, a Babcock cream-bottle is employed. The contents of the beaker, after cooling, are transferred to the test-bottle as follows: Add to the beaker about half of the 17.6 cc. of sulphuric acid regularly used for the test, stir with the rod and pour carefully into the bottle, using the remainder of the acid in two portions for washing out the beaker. Finally proceed as in the regular Babcock test for milk. Multiply the fat reading by 18 and divide by the weight of the sample taken to obtain the per cent of fat.

*Short's Method.\**—Grind to a uniform powder 2 to 5 grams of the sample, and about twice its weight of anhydrous copper sulphate. Place a layer of anhydrous copper sulphate about 2 cm. thick on the bottom of the inner tube of a Johnson or Knorr extractor, add the ground mixture, and rinse the mortar first with a little anhydrous copper sulphate and finally with ether. Extract for 16 hours, evaporate the ether from the extraction-flask, and dry the fat in a boiling-water oven to constant weight.

*Werner-Schmidt Method.*—Boil 2 to 3 grams of the sample in the Werner-Schmidt tube (p. 126) with 5 cc. of water and 10 cc. of concentrated hydrochloric acid till, with constant shaking, all but the fat is dissolved. Cool, add 25 cc. of ether, and shake the tube well. Draw off as much as possible of the ether, after separation, in the usual manner, and extract with four or five additional portions of the solvent.

Distil off the ether from the combined extractions, and weigh the fat.

**Determination of Total Protein.**—Calculate from the nitrogen, determined by the Kjeldahl or Gunning method on 1 to 2 grams of the sample, using the factor 6.38. Van Slyke adds a piece of copper sulphate the size of a pea during the digestion.

**Separation and Determination of Nitrogen Compounds.**—*Methods of Van Slyke.†*—Twenty-five grams of the sample are mixed in a porcelain mortar with an equal volume of clear quartz sand. Transfer the mixture to a 450-cc. Erlenmeyer flask, add about 100 cc. of water at 50° C., and keep the temperature at 50° to 55° C. for half an hour with frequent shaking. Decant the liquid through an absorbent-cotton filter into a

\* U. S. Dept. of Agric., Div. of Chem., Bul. 35, pp. 15, 17, 225.

† Van Slyke, N. Y. Exp. Station, Bul. 215.



500-cc. graduated flask. Treat the residue with repeated portions of 100 cc. each of water, heating, shaking, and decanting as above till the filtrate, or water extract, at room temperature amounts to just 500 cc. exclusive of the fat floating on top, and use aliquot parts of this water extract for the various determinations.

*Water-soluble Nitrogen.*—Determine the nitrogen by the Gunning method in 50 cc. of the above water extract, corresponding to 2.5 grams of cheese.

*Nitrogen as Paranuclein.*—Add 5 cc. of a 1% solution of hydrochloric acid to 100 cc. of the above water extract (corresponding to 5 grams of cheese), and keep the temperature at 50° to 55° till the separation is complete, as shown by a clear supernatant liquid. Filter, wash the precipitate with water, and determine the nitrogen therein by the Gunning method.

*Nitrogen as Coagulable Protein.*—Neutralize the filtrate from the preceding determination with dilute potassium hydroxide, and heat at the temperature of boiling water till the coagulum,\* if any, settles completely. Filter, wash the precipitate, and determine the nitrogen therein.

*Nitrogen as Caseoses.*—Treat the filtrate from the preceding with 1 cc. of 50% sulphuric acid saturated with C. P. zinc sulphate, and warm to about 70° C. till the caseoses settle out completely. Cool, filter, wash with a saturated solution of zinc sulphate acidified with sulphuric acid, and determine the nitrogen in the precipitate.

*Nitrogen as Amino Acids and Peptones.*—Place 100 cc. of the water extract in a 250-cc. graduated flask, add 1 gram of sodium chloride and a solution containing 12% of tannin, till the addition of a drop to the clear supernatant liquid does not further precipitate. Dilute to the 250-cc. mark, shake, pour upon a dry filter, and determine the nitrogen in 50 cc. of the filtrate, which gives the amount of nitrogen in the amino-acid and ammonia compounds. Deduct from this the amount of nitrogen as ammonia separately determined, and the difference is the amino-nitrogen.

Nitrogen as peptones is obtained by subtracting the sum of the amounts of nitrogen as paranuclein, coagulable proteins, caseoses, amino-bodies, and ammonia from the total nitrogen in the water extract.

*Nitrogen as Ammonia.*—Distil 100 cc. of the filtrate from the above tannin-salt precipitation into standardized acid, and titrate in the usual manner.

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\* According to Van Slyke a precipitate at this point is rare in cheese.



**Nitrogen as Paracasein Lactate.**—Treat the residue insoluble in water in obtaining the water extract, with several portions of a 5% solution of sodium chloride, to form a 500-cc. salt extract of the same, in an analogous manner to that employed in preparing the water extract. Determine the nitrogen in an aliquot part of this salt extract.

**Determination of Lactose.**—Rub up ten grams of the sample with water at 40 to 50° C., and decant onto a filter. Repeat the operation until the filtrate measures 100 cc. Determine lactose in an aliquot by one of the methods described under milk.

Except in the case of whey cheese, lactose is present only in minute amount if at all. Commonly lactose, lactic acid, and other minor constituents are determined by difference subtracting the sum of the water, fat, total protein, and total ash from 100.

**Determination of Ash.**—Ignite at dull redness 5 grams of the cheese in a tared platinum or porcelain dish until a white ash is obtained, using preferably a muffle furnace, cool in a desiccator, and weigh.

**Determination of Sodium Chloride.**—Treat the ash with water acidified with nitric acid, filter, wash, and precipitate the chlorine in the filtrate with silver nitrate solution. Filter on a Gooch crucible, heat cautiously to melting, and weigh. Calculate the equivalent sodium chloride.

The chlorine may also be determined volumetrically, without filtering, by Volhard's method.

**Acidity.**—*Jensen and Plattner Method.*\*—Rub up 10 grams of the sample in a mortar with water at 40 to 50° C., and decant onto a filter. Repeat the rubbing and decanting until the filtrate measures 100 cc. Titrate an aliquot with N/10 sodium hydroxide, using phenolphthalein as indicator; 1 cc. = 0.009 gram lactic acid.

Although the acidity is usually calculated as lactic acid, other acids or acid-reacting substances may be present.

**Detection of Foreign Fat.**—The cheese fat, separated in the manner described below, is subjected to the various processes detailed under butter, in precisely the same way, the fat of cheese being identical with that of butter. The most ready means for judging its purity consists in determining the refraction with the butyro-refractometer, and the Reichert number.

The distinction of cow's milk from goat's milk fat is best accomplished, according to Hals and Sunde † by the ratio of the Polenske to the Reichert-

\* Zeits. Unters. Nahr. Genussm., 12, 1906, p. 193.

† Tidskr. Kem. Farm. Ter., 5, 1908, p. 8.



Meissl number which for goat's milk is about 1:5 and for cow's milk 1:8 to 1:9.

**Separation of the Fat for Examination.**—Place a quantity, say 25 grams, of the finely divided sample in a large Erlenmeyer flask, add about 100 cc. of petroleum ether, cork the flask and allow it to stand for several hours with frequent shaking. Decant the petroleum ether through a filter, evaporate off the solvent by the aid of heat, and the residue will be found to consist of nearly pure fat.

Or, wrap a sufficient portion of the finely divided sample in a muslin cloth, place this in a dish, and heat on the water-bath. The fat which runs out is afterward filtered and dried at 100°.

Sufficient cheese fat may usually be obtained for the refractometer reading from the neck of the test-bottle, after completing the Babcock test, and, usually (except in the case of skimmed-milk cheese), for the Reichert number.

**Detection of Skimmed-milk Cheese.**—In a cream cheese the fat should greatly exceed the protein; in a whole-milk cheese the per cent of fat should at least equal that of the protein, and is generally in excess. If the fat is considerably less than the protein, the cheese has undoubtedly been made from skimmed milk. The analyses on page 199 illustrate these points.

#### PROTEIN PREPARATIONS.

**Casein** is the basis of a variety of preparations, some of which are intended for the use of invalids and people of weak digestion, and others, from their compactness, for travellers and campers. Among these foods are the following:

*Nutrose.*—This is a caseinate of sodium formed by the action of the alkali upon dried casein. It is soluble in water.

*Eucasin* is a caseinate of ammonium, a soluble powder somewhat similar to nutrose.

*Plasmon.*—This is a yellowish powder, prepared by treatment with sodium bicarbonate of the curd precipitated from skimmed milk. The compound is kneaded in an atmosphere of carbon dioxide, and reduced to a soluble powder.

The following analysis of *plasmon* was made by Woods and Merrill:\*

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\* Maine Agric. Exp. Sta., Bul. 178, p. 101.



Water.	Protein.	Fat.	Carbohydrates.	Ash.	Fuel Value.
8.5	75.0	0.2	8.9	7.4	2044

*Hogg's Protein Nerve Food* is practically the same as sanotogen.

*Sanose*.—This is a powder. It contains 80% of pure casein and 20% of albumose obtained from the white of egg. The powder possesses a slight taste and an odor suggestive of milk. By briskly stirring the powder with water, an emulsion may be made much resembling milk, but on standing it soon breaks up.

*Sanatogen* is a grayish-white, tasteless powder, containing 95% of casein and 5% sodium glycono-phosphate. When treated with cold water it swells, forming on heating a milk-like emulsion.

*Vitafer* and *Cibrola* are similar to sanotogen but contain more glycono-phosphate and less casein.

*Lactalbumin* in dried and powdered form, like lactose, is obtained from whey.



## CHAPTER VIII.

### FLESH FOODS.

#### MEAT.

**General Structure and Composition.**—Meat is structurally made up of *muscle fibers*, held together by *connective tissue*, through which blood vessels, nerves, and usually fat cells are more or less abundantly distributed. Each muscle fiber has a sheath or covering known as *sarcolemma* inclosing the meat juices, which are solutions in water of proteins, non-protein nitrogenous extractives, and salts.

The insoluble portion of the living muscle (connective tissue, sarcolemma, etc.) is known as *stroma*, the soluble portion constituting about 60% by weight as obtained by high pressure, as *plasma*. After *rigor mortis* sets in certain soluble constituents coagulate and the liquid obtained by pressure is a *serum* analogous to the serum separated from blood clot.

Nitrogen compounds constitute by far the most abundant and important portion of the substance of lean meat. Carbohydrates are almost entirely lacking, the small amount of glycogen and muscle sugar together constituting rarely more than 1 per cent.

*The Nitrogenous Substances* consist of proteins and so-called extractives. The stroma proteins include the *elastin* and *collagen* of connective tissue and the proteins of the sarcolemma which are imperfectly understood. Formerly *myosin*, a globulin-like substance, was considered the chief protein of the plasma but now it is believed that *myogen*, an albumin, is a more abundant constituent. Peptones and proteoses are formed in meat after death by enzymic action. The extractives of chief interest to the analyst are *creatine*, *creatinine*, *carnosine*, *carnitine*, *methyl guanidine*, and the purine bases *xanthine*, *hypoxanthine*, *guanine*, and *adenine*.

*The Fats* are considered in Chapters III and XIII.

*Carbohydrates.*—

*Glycogen* ( $C_6H_{10}O_5$ ), sometimes called animal starch, is a white, amorphous, tasteless, and colorless substance, when pure, much resembling starch. It is soluble in water, forming an opalescent solution, and is insoluble in ether and nearly so in cold alcohol. With iodine a port-wine color is produced, which disappears on heating and reappears on cooling.



Glycogen is strongly dextro-rotatory and converted into dextrose by boiling with dilute mineral acid. It occurs in small amount in all fresh muscle, particularly that of the horse, and in larger amount in liver, but disappears to a large extent on aging.

*Muscle Sugar* is either entirely absent in the living muscle, or exists in traces only. After death it is formed presumably from the glycogen, and exists in a very minute quantity, probably as dextrose.

*Inosite* ( $C_6H_{12}O_6 + H_2O$ ) is found in traces in the muscular substance and animal organs. It is a protoplasmic substance related to the carbohydrates.

*The Acids* developed in meat consist chiefly of *lactic*. *Succinic*, *inosinic*, and *uric* acids are among the other acids present in small amount.

*The Ash* of meat consists, like that of plants, of phosphates, sulphates, and chlorides of potassium, sodium, calcium, magnesium, and iron together with traces of other inorganic constituents. In the meat itself some of the inorganic elements exist in organic combination.

The approximate proportions in which the chief constituents are present in meat is thus shown by König:

Water.....		75.0	to	77.0
	Sarcolemma (muscle fiber).....	13.0	to	18.0
	Connective tissue.....	2.0	to	5.0
	Albumin (coagulable protein).....	0.6	to	4.0
	Creatine.....	0.07	to	0.34
Nitrogenized compounds.....	Hypoxanthine.....	0.01	to	0.03
	Creatinine.....	Undetermined		
	Xanthine.....			
	Inosinic acid.....			
	Uric acid.....			
	Urea.....	0.01	to	0.03
Fat.....		0.05	to	3.5
	Lactic acid.....	0.05	to	0.07
	Butyric acid.....	Undetermined		
Other nitrogen-free compounds.....	Acetic acid.....			
	Formic acid.....			
	Inosite.....			
	Glycogen.....	0.3	to	0.5
Salts.....		0.8	to	1.8
	Composed of:			
	Potash.....	0.40	to	0.50
	Soda.....	0.02	to	0.08
	Lime.....	0.01	to	0.07
	Magnesia.....	0.02	to	0.05
	Oxide of iron.....	0.003	to	0.01
	Phosphoric acid.....	0.40	to	0.50
	Sulphuric acid.....	0.003	to	0.04
	Chlorine.....	0.01	to	0.07



**Proximate Composition of the Commoner Meats.**—The chief characteristics of the flesh of various animals are in the main very similar, whatever the nature of the animal. So true is this, indeed, that it is usually impossible from a chemical analysis to distinguish a particular kind of flesh when mixed with that of other animals in finely divided meat preparations, such as sausages, potted and deviled meats, and the like.

The average composition of the commoner cuts of beef, veal, mutton, lamb, and pork, as well as of fowl and game, is shown in the following tables, compiled from the work of Atwater and Bryant\*, the accompanying diagrams serving to locate, in the case of ordinary meats, the portion of the animal from which the meat is taken.

**Constants of Fat.**—These as found by Bigelow are given in the table on page 212.

**Characteristics of Sound Meat.**—The reaction of meat should be acid. If neutral or alkaline, decomposition is indicated, except that alkalinity may be due to the use of alkaline salts as preservatives.

Letheby † gives the following characteristics of sound, fresh meat. In color it is neither pale pink nor deep purple, the former indicating that the animal was affected with some disease, and the latter that it died a natural death, and was not slaughtered. In appearance it is marbled, due to the presence of small veins of fat distributed among the muscles. In consistency it is firm and elastic to the touch, and should hardly moisten the finger; a wet, sodden, or flabby consistency with a jelly-like fat is indicative of bad meat. As to odor, it is practically free; whatever odor there is should not be disagreeable; a sickly or cadaverous smell is indicative of diseased meat. After standing for a day or so, it should not become wet, but on the contrary should grow drier. When dried at 100° C. it should not lose more than 70 to 74 per cent in weight; unsound meat frequently loses 80% or more. It should shrink very little in cooling.

**Inspection of Meat.**—While carefully drawn laws exist almost everywhere relating to the sale of meat, and government inspectors are appointed to carry out the requirements of the laws, yet in this country there is undoubtedly some meat unfit for food on the market, owing to the small number of inspectors, and the consequent comparative safety with which unscrupulous dealers may sell meats forbidden by law and escape detection. The inspection of meats and fish under municipal ordinances is not always carried out as thoroughly as might be desired.

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\* U. S. Dept. of Agric., Off. of Exp. Stations, Bul. 28 (Revised Ed.).

† Lectures on Food, p. 210.



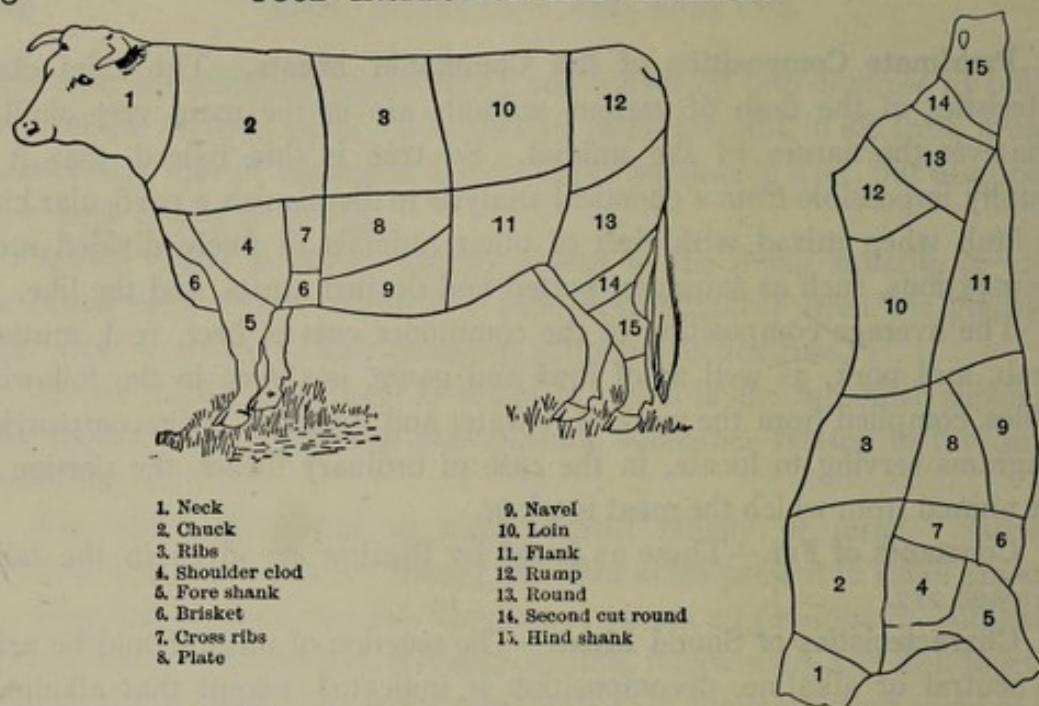


FIG. 56.—Diagram Showing Cuts of Beef.

## COMPOSITION OF BEEF.

Cut.	Number of Analyses.	Refuse.	Water.	Protein.		Fat.	Ash.	Fuel Value per Pound. Cals.
				N × 6.25.	By Difference.			
Chuck: Lean— edible portion..	2	.....	71.3	20.2	19.5	8.2	1.0	720
as purchased..	2	19.5	57.4	16.3	15.7	6.6	0.8	580
Medium—edible portion..	4	.....	68.3	19.6	18.9	11.9	0.9	865
as purchased...	4	15.2	57.9	16.6	16.0	10.1	0.8	735
Fat— edible portion..	4	.....	62.3	18.5	18.0	18.8	0.9	1135
as purchased...	3	14.7	53.3	15.9	15.4	15.9	0.7	965
Ribs: Lean— edible portion..	6	.....	66.0	16.5	16.9	9.8	0.8	790
as purchased...	6	22.6	52.6	15.2	14.8	9.3	0.7	675
Medium—edible portion..	15	.....	55.5	17.5	17.0	26.6	0.9	1450
as purchased...	15	20.8	43.8	13.9	13.5	21.2	0.7	1155
Fat— edible portion..	9	.....	48.5	15.0	15.2	35.6	0.7	1780
as purchased...	8	16.8	39.6	12.7	12.4	30.6	0.6	1525
Loin: Lean— edible portion..	12	.....	67.0	19.7	19.3	12.7	1.0	900
as purchased...	11	13.1	58.2	17.1	16.7	11.1	0.9	785
Medium—edible portion..	32	.....	60.6	18.5	18.2	20.2	1.0	1190
as purchased...	32	13.3	52.5	16.1	15.8	17.5	0.9	1040
Fat— edible portion..	6	.....	54.7	17.5	16.8	27.6	0.9	1490
as purchased...	6	10.2	49.2	15.7	15.0	24.8	0.8	1305
Rump: Lean— edible portion..	4	.....	65.7	20.9	19.6	13.7	1.0	965
as purchased...	3	14.0	56.6	19.1	17.5	11.0	0.9	820
Medium—edible portion..	10	.....	56.7	17.4	16.9	25.5	0.9	1400
as purchased...	10	20.7	45.0	13.8	13.4	20.2	0.7	1110
Fat— edible portion..	5	.....	47.1	16.8	16.4	35.7	0.8	1820
as purchased...	5	23.0	36.2	12.9	12.6	27.6	0.6	1405
Round: Lean— edible portion..	31	.....	70.0	21.3	21.0	7.9	1.1	730
as purchased...	29	8.1	64.4	19.5	19.2	7.3	1.0	670
Medium—edible portion..	18	.....	65.5	20.3	19.8	13.6	1.1	950
as purchased...	14	7.2	60.7	19.0	18.3	12.8	1.0	895
Fat— edible portion..	5	.....	60.4	19.5	19.1	19.5	1.0	1185
as purchased...	3	12.0	54.0	17.5	17.1	16.1	0.8	1005



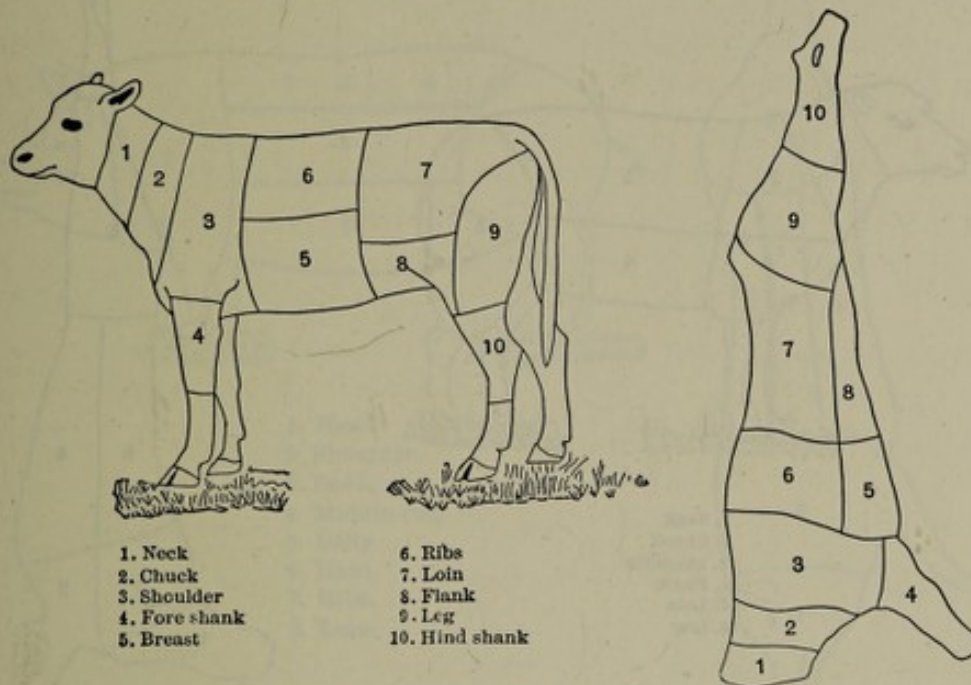


FIG. 57.—Diagram Showing Cuts of Veal.

## COMPOSITION OF VEAL.

Cut.	Number of Analyses.	Refuse.	Water.	Protein.		Fat.	Ash.	Fuel Value per Pound Cals.
				N × 6.25.	By Difference.			
Chuck: Lean—	edible portion..	1	.....	76.3	.....	20.6	1.9	465
	as purchased...	1	19.0	61.8	.....	16.7	1.6	380
	Medium—edible portion..	6	.....	73.3	19.7	19.2	6.5	640
	as purchased...	6	18.9	59.5	16.0	15.6	5.2	515
Ribs: Medium—	edible portion..	9	.....	72.7	20.7	20.1	6.1	640
	as purchased...	9	25.3	54.3	15.5	15.0	4.6	480
	Fat—edible portion..	3	.....	60.9	18.7	18.8	19.3	1160
	as purchased...	3	24.3	46.2	14.2	14.2	14.5	875
Loin: Lean—	edible portion..	5	.....	73.3	20.4	19.9	5.6	615
	as purchased...	5	22.0	57.1	15.9	15.6	4.4	480
	Medium—edible portion..	6	.....	69.0	19.9	19.2	10.8	825
	as purchased...	6	16.5	57.6	16.6	16.0	9.0	690
Fat—	edible portion..	2	.....	61.6	18.7	18.5	18.9	1145
	as purchased...	2	18.3	50.4	15.3	15.1	15.4	935
Leg: Lean—	edible portion..	9	.....	73.5	21.3	21.2	4.1	570
	as purchased...	9	9.1	66.8	19.4	19.3	3.7	520
	Medium—edible portion..	10	.....	70.0	20.2	19.8	9.0	755
	as purchased...	9	14.2	60.1	15.5	16.9	7.9	620



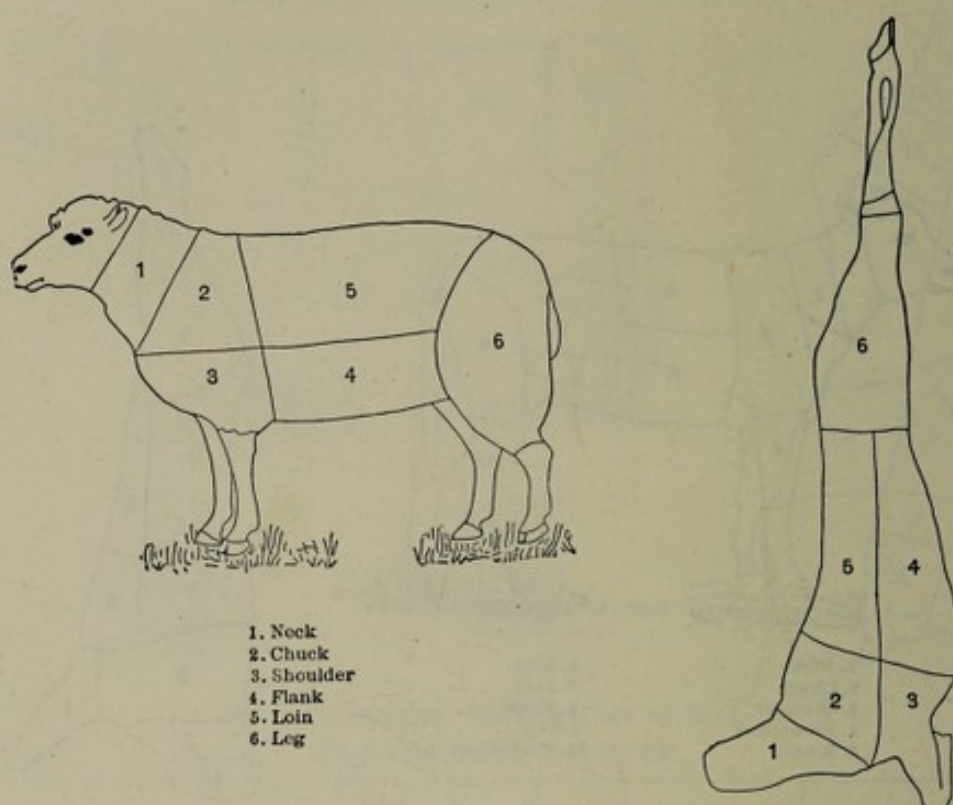


FIG. 58.—Diagram Showing Cuts of Mutton.  
COMPOSITION OF MUTTON AND LAMB.

Cut.	Number of Analyses.	Refuse.	Water.	Protein.		Fat.	Ash.	Fuel Value per Pound. Cals.	
				N× 6.25.	By Difference.				
MUTTON.									
Chuck: Lean—	edible portion..	1	.....	64.7	17.8	18.1	16.3	0.9	1020
	as purchased...	1	19.5	52.1	14.3	14.5	13.1	0.8	820
Medium—	edible portion..	6	.....	50.9	15.1	14.6	33.6	0.9	1700
	as purchased...	6	21.3	39.9	11.9	11.5	26.7	0.6	1350
Fat—	edible portion..	2	.....	40.6	13.9	13.7	44.9	0.8	2155
	as purchased...	2	16.5	33.8	11.6	11.5	37.5	0.7	1800
Loin: Medium—	edible portion..	13	.....	50.2	16.0	15.9	33.1	0.8	1695
	as purchased...	12	16.0	42.0	13.5	13.0	28.3	0.7	1445
Fat—	edible portion..	3	.....	43.3	14.7	14.2	41.7	0.8	2035
	as purchased...	3	11.7	38.3	13.0	12.5	36.8	0.7	1795
Flank: Medium—	edible portion..	8	.....	46.2	15.2	14.8	38.3	0.7	1900
	as purchased...	2	9.9	39.0	13.8	13.6	36.9	0.6	1815
Leg: Lean—	edible portion..	3	.....	67.4	19.8	19.1	12.4	1.1	890
	as purchased...	3	16.8	56.1	16.5	15.9	10.3	0.9	740
Medium—	edible portion..	11	.....	62.8	18.5	18.2	18.0	1.0	1105
	as purchased...	11	18.4	51.2	15.1	14.9	14.7	0.8	900
LAMB.									
Chuck:	edible portion..	1	.....	56.2	19.1	19.2	23.6	1.0	1350
	as purchased...	1	19.1	45.5	15.4	15.5	19.1	0.8	1090
Leg: Medium—	edible portion..	2	.....	63.9	19.2	18.5	16.5	1.1	1055
	as purchased...	2	17.4	52.9	15.9	15.2	13.6	0.9	870
Fat—	edible portion..	1	.....	54.6	18.3	17.1	27.4	0.9	1495
	as purchased...	1	13.4	47.3	15.8	14.8	23.7	0.8	1295
Loin:	edible portion..	4	.....	53.1	18.7	17.6	28.3	1.0	1540
	as purchased...	4	24.8	45.3	16.0	15.0	24.1	0.8	1315



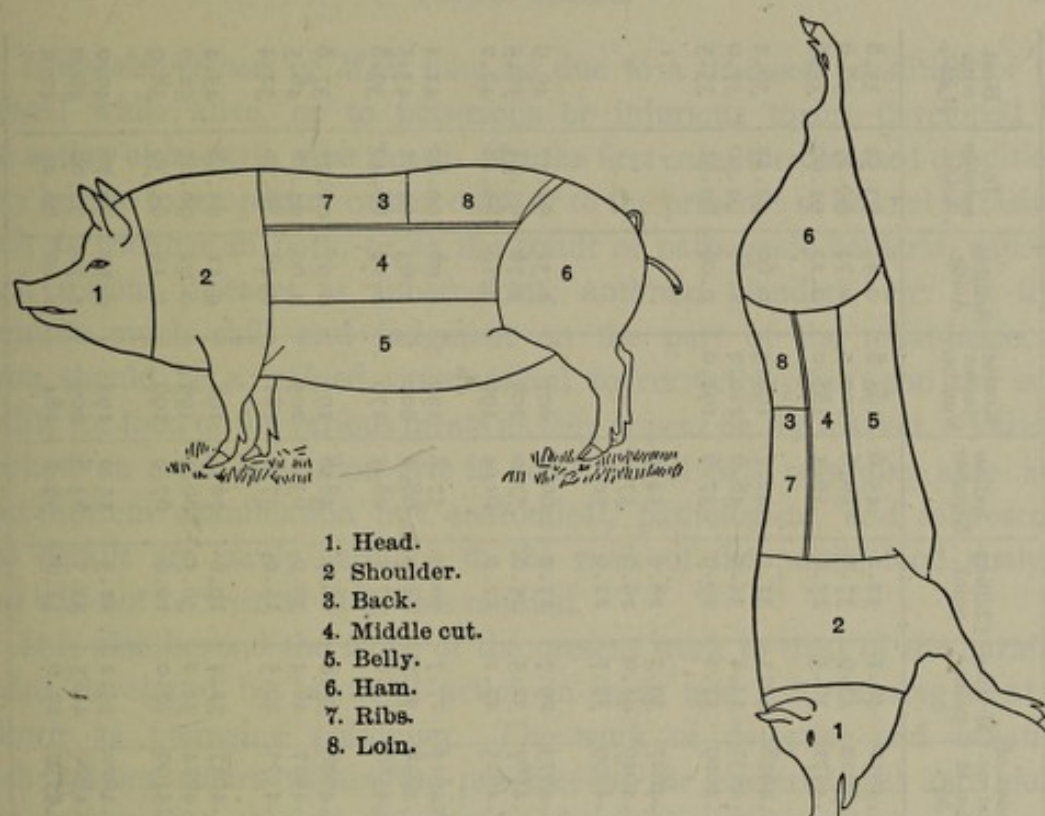


FIG. 59.—Diagram Showing Cuts of Pork.

## COMPOSITION OF PORK, POULTRY, AND GAME.

Cut.	Num- ber of Anal- yses.	Refuse.	Water.	Protein.		Fat.	Ash.	Fuel Value per Pound Cals	
				N× 6.25.	By Differ- ence.				
PORK.									
Shoulder:	edible portion...	19	.....	51.2	13.3	13.8	34.2	0.8	1690
	as purchased...	19	12.4	44.9	12.0	12.2	29.8	0.7	1480
Loin: Lean—	edible portion...	1	.....	60.3	20.3	19.7	19.0	1.0	1180
	as purchased...	1	23.5	46.1	15.5	15.1	14.5	0.8	900
Fat—	edible portion...	4	.....	41.8	14.5	13.1	44.4	0.7	2145
	as purchased...	4	16.5	34.8	11.9	10.9	37.2	0.6	1790
Ham: Lean—	edible portion...	2	.....	60.0	25.0	24.3	14.4	1.3	1075
	as purchased...	2	0.9	59.4	24.8	24.2	14.2	1.3	1060
Fat—	edible portion...	5	.....	38.7	12.4	10.6	50.0	0.1	2345
	as purchased...	5	13.2	33.6	10.7	9.2	43.5	0.5	2035
POULTRY AND GAME.									
Chicken:	edible portion...	3	.....	74.8	21.5	21.6	2.5	1.1	505
	as purchased...	3	41.6	43.7	12.8	12.6	1.4	0.7	295
Fowl:	edible portion...	26	.....	63.7	19.3	19.0	16.3	1.0	1045
	as purchased...	26	25.9	47.1	13.7	14.0	12.3	0.7	775
Goose:	edible portion...	1	.....	46.7	16.3	16.3	36.2	0.8	1830
	as purchased...	1	17.6	38.5	13.4	13.4	29.8	0.7	1505
Turkey:	edible portion...	3	.....	55.5	21.1	20.6	22.9	1.0	1360
	as purchased...	3	22.7	42.4	16.1	15.7	18.4	0.8	1075
Quail:	as purchased...	1	.....	66.9	21.8	.....	8.0	1.7	775



FATS OF VARIOUS MEATS AND MEAT PREPARATIONS.\*

	Specific Gravity 100° 100°	Specific Gravity 100° 15°	Degrees Butyro- refrac- tometer at 35°	Index of Refrac- tion at 35°	M-lting- point.	Chilling- point.	Iodine Number.	Koetts- torfer Number.	Soluble Acids.	In- soluble Acids.	Heat with H <sub>2</sub> SO <sub>4</sub> .
Beef, roast and boiled:	Average.....	0.8953	52.8	1.4610	40.1	32.2	45.6	194.8	0.45	93.54	35.8
	Maximum....	0.9046	55.5	1.4629	43.9	37.0	50.6	200.0	0.74	95.27	36.0
	Minimum...	0.8925	47.0	1.4571	36.5	27.8	36.1	188.0	0.22	91.90	35.6
Beef, canned, corned:	Average.....	0.8934	53.98	1.4618	40.1	31.0	42.7	196.3	0.60	92.37	36.2
	Maximum....	0.8944	56.0	1.4631	43.4	34.5	48.6	210.0	1.62	94.50	37.0
	Minimum...	0.8925	52.7	1.4609	37.2	29.9	37.9	191.0	0.09	89.51	35.5
Beef, canned, dried, and smoked:	Average.....	.....	55.2	1.4627	39.3	26.0	54.1				
	Maximum....	.....	58.5	1.4649	41.6	29.0	57.5				
	Minimum...	.....	51.0	1.4599	37.7	22.0	50.9				
Horse meat:	Average.....	0.9067	62.3	1.4673	29.7	17.2	66.4	201	1.32	91.37	49.9
	Maximum....	0.9184	76.5	1.4762	32.5	25.0	77.0	205	2.76	95.44	56.5
	Minimum...	0.8868	55.2	1.4625	27.2	12.0	61.4	196	0.19	88.18	46.2
Ham and bacon, canned:	Average.....	0.8893	53.4	1.4615	27.7	20.3	57.8	193	0.98	90.80	41.4
	Maximum....	0.9066	58.2	1.4648	30.5	24.0	68.2	207	2.63	94.51	43.5
	Minimum...	0.8699	49.0	1.4586	23.6	17.5	48.5	179	0.19	86.40	39.8
Tongue, canned:	Average.....	0.8929	54.0	1.4628	35.8	26.7	48.8	193	0.97	90.91	37.8
	Maximum....	0.9096	58.0	1.4677	41.4	33.0	63.6	205	2.60	94.78	46.5
	Minimum...	0.8717	47.2	1.4573	26.5	15.0	37.8	181	0.42	86.20	32.4
Fowl:	Average.....	0.8999	56.7	1.4637	31.2	19.6	76.1	198	0.51	95.58	45.4
	Maximum....	0.9157	62.5	1.4674	34.0	36.5	86.4	199	0.92	96.03	52.0
	Minimum...	0.8849	49.0	1.4590	28.0	12.0	67.0	196	0.15	95.39	38.9
Sausage, canned:	Average.....	0.8953	54.6	1.4620	35.2	22.3	55.0	193	0.70	90.76	40.0
	Maximum....	0.9225	60.3	1.4660	41.5	33.5	63.8	200	1.70	92.68	40.0
	Minimum...	0.8786	49.2	1.4559	27.6	12.0	39.8	184	0.10	88.25	40.0

\* U. S. Dept. of Agric., Bur. of Chem., Bul. 13, part 10, p. 1490 et seq.



*Unwholesomeness of Meat* may be due to a diseased condition of the animal while alive, or to poisonous or injurious toxins developed by the action of bacteria after death. In the first case; the diseased conditions may be due to temporary causes only, or to the presence of animal parasites, such as trichinæ in pork, or as the result of pathogenic bacteria, causing such serious diseases as tuberculosis, anthrax, glanders, etc. It thus requires much skill and judgment on the part of the meat-inspector (who should be a trained veterinarian) to correctly pass upon the suitability for food of the various meats as they appear on the market. Various works \* on meat inspection give in detail useful data regarding ante- and post-mortem examination but anatomical, pathological, and microscopical details are rarely germane to the work of the public food analyst, and will not be treated of in this manual.

It is also beyond the scope of the present work to treat of the harmful toxins developed by bacterial action in meat and fish, causing what is known as ptomaine poisoning. The work of detecting and isolating such poisons comes within the province of the bacteriologist and biologist, rather than that of the chemist, involving many experiments upon guinea-pigs, rabbits, or other animals not usually found in the chemist's laboratory. It has furthermore been demonstrated by Vaughn and Novy † that even when these toxins are present in foods in sufficient quantity to produce serious results, very considerable amounts of the food must be taken in order to isolate them by chemical means, more, in fact, than is usually available for analysis.

**U. S. Standards.**‡—*Standard Meat* is any sound, dressed, and properly prepared edible part of animals in good health at the time of slaughter. The term "animals" as herein used includes not only mammals, but fish, fowl, crustaceans, mollusks, and all other animals used as food.

*Standard Fresh Meat* is meat from animals recently slaughtered, or preserved only by refrigeration.

*Standard Salted, Pickled, and Smoked Meats* are unmixed meats preserved by salt, sugar, vinegar, spices, or smoke, singly or in combination, whether in bulk or in packages.

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\* Andrews, *Flesh Foods*; Edelmänn (trans. by Mohler and Eichhorn), *Textbook of Meat Hygiene*; Ostertag (trans. by Wilcox), *Handbook of Meat Inspection*; Robertson, *Meat and Food Inspection*; Vacher, *The Food Inspector's Handbook*; Walley, *A Practical Guide to Meat Inspection*.

† Cellular Toxins.

‡ U. S. Dept. of Agric., Off. of Sec., Circ. No. 19.



*Standard Manufactured Meats* are meats not included in the above divisions, whether simple or mixed, whole or comminuted, with or without the addition of salt, sugar, vinegar, spices, smoke, oils, or rendered fat, if they bear names descriptive of their composition, and when bearing such descriptive names, if force or flavoring meats are used, the kind and quantity thereof are made known.

**Preservation of Meat.**—Raw meat soon begins to decompose, unless precautions are taken to destroy, or at least check the growth of putrefying bacteria. From earlier times the subjection of meat to extreme cold has been practiced in order to enhance its keeping qualities. Bacterial growth is inhibited to a greater or less extent by refrigeration, by subjecting the meat to the various processes of curing, by the use of high temperatures and the exclusion of air as in canning, and by the employment of antiseptics.

**Cold Storage** may consist (1) in actually freezing the meat, in which condition it may be kept without decomposition almost indefinitely, until finally thawed for use, or (2) in keeping the meat at or near the temperature of freezing without actually congealing it, as is done by the use of the ordinary refrigerator. The second method, while much less efficacious than the first, serves to prevent decomposition for a considerable time.

**Effects of Cold Storage on the Composition of Meat.**—Richardson and Scherubel\* made comparative analyses of beef stored at 2 to 4° C. for a week or less and of that held at -11 to -13° C. for various lengths of time ranging from a month to over a year and a half. The results of chemical analysis, as well as histological and bacterial examination, are practically the same in both cases. When stored for some months at 2 to 4° C. with or without previous freezing, they found more or less marked increases in ammonia, coagulable proteins, albumoses, and meat bases.

Wright's investigation† with lamb, mutton, and other meats showed no bacterial change during storage at freezing temperatures and only slight chemical changes.

Pennington,‡ in experiments with cold-storage chickens (the history of which up to the beginning of storage is unknown), found during storage for 14 months or longer a marked increase in certain nitrogenous constituents, notably those soluble in cold water and those coagulable, also marked changes in the fat, especially the lowering of the iodine number and the

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\* Jour. Ind. Eng. Chem., 1, 1909, p. 95.

† Jour. Soc. Chem. Ind., 31, 1912, p. 965.

‡ U. S. Dept. of Agric., Bur. of Chem., Bul. 115, 1908.



liberation of large amounts of free fatty acids. Histological and bacterial changes were also noted.

Richardson,\* on the other hand, observed with chickens frozen 3 to 25 months, some variations in composition but no progressive changes of any kind. He states "More decomposition developed at room temperature (26°) during 24 to 48 hours than when stored below -5° for 9 years."

Pennington, Hepburn, St. John, and Witmer,† in experiments with poultry held at 23.9° C. for 4 days and at 7.2 to 12.8° for 7 days, in a household refrigerator and at 0° for 3 weeks, obtained in all cases an increase of amino acids and basic nitrogen and a decrease of proteins, also an increase in the acid value of the fat as well as changes in histological structure.

**Curing** consists in subjecting the meat to various processes of drying, smoking, pickling, corning, etc., or to a combination of these processes.

*Drying.*—In simple drying, the meat is subjected to the heat of the sun or to artificial heat. Commonly, drying is combined with some other method of preservation such as smoking in the case of beef or salting and spicing as in the case of certain kinds of sausage. Powders made of desiccated meat are as yet of small importance.

*Smoking.*—As commonly practiced with beef, bacon, and ham, the meat, which may or may not be first salted or otherwise treated, is exposed to the smoke of the burning beech or hickory wood, thus becoming impregnated with the antiseptic properties of the creosote and pyroligneous acid, at the same time being dried by the heat. Treatment with crude pyroligneous acid, instead of smoking, is also commonly practiced. In some cases best results are obtained by a slow smoking at a comparatively low temperature, while in others quick, hot smoking is found most efficacious. The character of the meat is decidedly changed by smoking, and, according to Utescher, smoked meat is always alkaline in reaction.

*Pickling.*—The meat may be treated with dry salt and subjected to pressure, so that the meat juice forms the liquid for the brine, in which it is allowed to remain for some time; or, as in the ordinary process of *corning*, the beef is soaked for some days in a strong solution of salt to which a little saltpeter ( $\text{KNO}_3$ ) has been added. In the process of pickling, the salts from the brine slowly diffuse into the interior of the meat by osmosis, a part of the soluble albumin passing out into the brine. The effect of the saltpeter is to preserve the natural red color of the meat, which by the action of salt alone becomes destroyed, or at least impaired.

\* Allen's Coml. Org. Analysis, 4th Ed., 8, p. 426.

† Jour. Biol. Chem., 29, 1917, xxxi.



Bacon and ham are frequently cured by pickling in brine containing salt, saltpetre, and cane sugar, and sometimes also such antiseptics as boric acid and calcium bisulphite.

The curing of bacon is sometimes effected by injecting the pickling fluid into the tissues with a "pickle-pump," capable of exerting a pressure of 40 lbs. to the square inch, and provided with a hollow, perforated needle-nozzle, which penetrates the flesh. After pickling, the bacon or ham may be simply dried, or, if desired, smoked. Oak sawdust is frequently burned for the smoking of ham.

**The Use of Antiseptics in Meat.**—Most of what might be termed the modern preservatives are to be looked for in one or another of the various meat preparations, though some are better adapted than others for use in particular cases, as will be seen by reference to the composition of typical commercial preservative mixtures given on page 878.

*Borax and boric acid*, usually in mixture, have been used more commonly than any other preservatives for the preservation of meat. Like salt, they are used commonly in surface application, in the case of large cuts of meat, or by mixing, in the case of sausage meat. A more recent method of application consists in impregnating the tissue of the meat with a solution of the boric mixture, by means of the above-described pickle-pump. The use of boric acid and its compounds, however, is not permitted under the regulations of the Federal meat inspection law of the United States.

*Sulphurous Acid.*—As much as 1% of a solution of sulphurous acid may be added to meat without being apparent to the taste or smell. Mitchell quotes Fischer as having found that 50% of the preserved meat products (sausages, etc.) sold in Breslau in 1895 contained sulphites, varying in amount from 0.01 to 0.34 per cent of sulphur dioxide. Calcium bisulphite is a salt commonly employed. In Hamburg steak it serves partly as a preservative, but chiefly as a deodorizer and a restorer of the bright red color of fresh meat.

*Sodium Benzoate* is not of such common occurrence in meat products as the other antiseptics mentioned.

*Salicylic Acid* is now seldom used.

Among other preservative substances sometimes used with meat are solutions containing phosphoric acid and aluminum salts.

The toxic effects of these and other antiseptic chemicals in meats, and the most practical means of controlling their use are questions in controversy, presenting no new phases that have not been elsewhere dis-



cussed in treating of the general question of preservatives in food. Methods of detecting preservatives in meats are given elsewhere.

**Drawn vs. Undrawn Poultry.**—Much has been written on this subject but the consensus of opinion at the present time is in favor of undrawn poultry. Boos\* found that undrawn birds taken from cold storage and exposed to 68° F. showed better keeping qualities than the drawn, although freshly killed birds, drawn by a special method not commercially practiced, exposed directly to 68° F., showed perfect keeping qualities. Pennington† concludes that (1) undrawn poultry decomposes more slowly than does poultry which has been either wholly or practically eviscerated, (2) “full drawn” poultry, that is, completely eviscerated, with heads and feet removed, decomposes the most rapidly, and (3) “Boston drawn” and “wire drawn,” the latter being usually the better, are intermediate.

**Spoilage of Meat.**—Bacteriological examination and tests for ptomaines, although falling outside the scope of this work, must be carried on in conjunction with chemical examination in tracing the process of decomposition. Naturally all scientific work is superfluous when the ordinary senses show the product to be offensive. Of the chemical methods which have been proposed, those for the determination of ammonia in the flesh and free acids in the fat are most valuable.

**Bob Veal,** that is the flesh of calves butchered soon after birth, has long been regarded as unwholesome and its sale has been prohibited by Federal and State laws. Reliable methods of deduction are lacking, furthermore there is evidence by Fish and others that the meat is not, as claimed, laxative or otherwise injurious to health. Fetal and still-born calves are obviously unfit for food.

**The Effect of Cooking on Meat** is a subject of great importance in dietetics. Extensive investigations have been carried out by Grindley‡ and associates showing the losses by different methods of cooking and the changes in proteins and other substances brought about. The digestibility of the meat was practically the same regardless of kind, method of cooking and fatness, the meat in all cases being practically all assimilated.

The general result of cooking is to render the meat less tough, to develop an agreeable flavor, and to coagulate more or less of the proteins. When

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\* Mass. State Bd. Health Rep. 1907.

† U. S. Dept. of Agric., Bur. of Chem., Circ. 70, 1911.

‡ U. S. Dept. of Agric., Off. of Exp. Sta., Buls. 102, 141, 162, 193; Bur. of Chem., Bul. 81, 1904, p. 113.



subjected to moist heat, such as boiling and steaming, some of the soluble materials are dissolved, so that when the liquor in which the meat is boiled is thrown away, some of the valuable substances are lost. This is especially true when meat is placed in cold water which is afterwards brought to boiling, a method to be recommended when the liquor with the dissolved extractives is to be used for broth. When the meat to be boiled is placed at once in boiling water, there is less loss of soluble matter by reason of the formation of a more or less impenetrable coating on the outside, by the coagulation of the proteins. Meat that is boiled becomes softer, owing to a partial dissolving of the gelatin formed. In the dry cooking of meat, as by broiling or roasting, there is usually a hardening of the tissues, and a driving out of some of the meat juices, which are, however, often recovered in the form of gravies.

#### CANNED MEAT.

The most effective method of preserving meat and meat preparations of all kinds for long periods of time consists in sterilizing by heat, and sealing in air-tight cans. The process of canning cooked meat and its products does not differ materially from that employed in the similar preparation of vegetables. (See Chapter XXI.) Previous to canning, the meats are usually cooked by boiling, during which process the changes described in the preceding paragraph take place.

**Adulteration.**—The practice of misbranding chopped meat with respect to variety has been very prevalent in the past, and many varieties of so-called potted and devilled meats and game have frequently consisted wholly or in large part of a cheaper variety than that specified on the label. This practice has been largely corrected in this country, owing to the enforcement of the regulations of the Federal meat inspection law. Unfortunately the biological tests for foreign meats are not applicable to the cooked products.

Out of 76 samples examined by McGill,\* 8 contained over 2 per cent of starch, 5 between 1 and 2 per cent, 17 less than 1 per cent, while 46 were free from starch.

Preservatives were at one time added to canned meats especially in the case of dried and smoked beef, ham, and bacon, and in potted and devilled mixtures, but this practice has been discontinued.

**Composition of Canned Meat.**—The following table, compiled from results published by Bigelow and others,† shows the composition of

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\* Lab. Inl. Rev. Dept., Canada., Bul. 164.

† U. S. Dept. of Agric., Bur. of Chem., Bul. 13, part. 10.



COMPOSITION OF CANNED MEATS.

	Num- ber of Analy- ses.	Water.	Fat.	Nitrogen.			Nitrogenous Substances.					Total Ash.	Sodium Chlo- ride.
				Total.	Insolu- ble in Hot Water.	Precipi- tated by Bro- mine.	Meat Bases.	Protein N X 6.25.	Pro- teins Insolu- ble in Hot Water (Coag.).	Gela- tinoids & Pro- teins Ppt'd by Bro- mine.	Meat Bases.		
Fresh beef:	5	68.19	12.60	3.02	2.41	0.19	0.43	18.89	15.08	1.21	1.29	0.96	0.08
	Average.....	71.17	15.33	3.19	2.52	0.24	0.53	19.94	15.75	1.50	1.65	1.13	0.24
	Minimum.....	65.81	9.89	2.83	2.32	0.14	0.33	17.69	14.50	0.87	1.02	0.78	Trace
Canned beef, roast and boiled:	22	58.89	13.99	4.15	3.09	0.57	0.49	25.95	19.29	3.59	1.58	1.28	0.53
	Average.....	66.39	31.78	5.51	4.47	1.16	1.03	34.44	27.94	7.25	3.21	3.51	2.51
	Maximum.....	45.35	5.89	3.31	1.98	0.24	0.09	20.69	12.38	1.50	0.62	0.65	0.04
Canned beef, corned:	11	56.35	11.43	4.26	3.58	0.32	0.36	26.63	22.37	2.02	1.11	4.69	3.37
	Average.....	60.10	22.68	4.89	4.33	0.53	0.47	30.56	27.06	3.31	1.47	7.38	4.68
	Maximum.....	46.94	6.33	3.72	3.01	0.20	0.20	23.25	18.81	1.25	0.62	3.57	2.56
Canned beef, smoked and dried:	11	47.42	7.46	5.21	4.22	0.15	0.85	32.59	26.41	0.92	2.63	12.51	9.67
	Average.....	59.58	12.37	6.49	4.91	0.37	1.29	40.56	30.69	2.31	4.02	17.30	11.33
	Maximum.....	39.22	4.59	4.00	3.30	0.06	0.59	25.00	20.62	0.38	1.59	9.58	7.15
Fresh horse meat:	16	69.81	9.61	3.11	2.37	0.20	0.55	19.47	14.83	1.23	1.70	1.01	0.01
	Average.....	76.91	33.66	3.60	2.97	0.36	1.22	22.50	18.56	2.25	3.81	1.27	0.09
	Maximum.....	52.16	1.24	2.13	1.33	0.12	0.15	13.31	8.31	0.75	0.47	0.63	Trace
Canned ham and bacon:	13	36.77	37.81	3.11	2.38	0.20	0.54	19.43	14.86	1.22	1.67	5.88	5.34
	Average.....	53.30	69.07	6.68	4.75	0.74	1.38	41.75	29.69	4.62	4.31	19.90	17.84
	Maximum.....	15.34	16.84	1.07	0.91	0.06	0.05	6.69	5.69	0.38	0.16	0.78	Trace
Canned tongue:	17	55.17	20.23	3.11	2.50	0.21	0.39	19.43	15.64	1.33	1.23	3.71	2.90
	Average.....	71.80	38.09	3.95	3.20	0.48	0.72	24.69	20.00	3.00	2.25	6.22	4.42
	Maximum.....	39.58	6.84	2.09	1.76	0.02	0.07	13.06	11.00	0.12	0.22	0.78	0.37
Canned fowl:	10	60.42	16.30	3.53	3.05	0.22	0.26	22.07	19.06	1.39	0.81	1.09	0.05
	Average.....	69.06	39.31	5.14	4.45	0.76	0.46	32.12	27.81	4.75	1.44	2.61	0.13
	Maximum.....	47.46	1.82	2.14	1.77	0.11	0.06	13.37	11.06	.....	.....	0.58	Trace
Canned fowl and game:	15	62.44	12.72	3.62	2.86	0.26	0.44	22.61	18.39	1.66	1.39	2.31	1.16
	Average.....	70.60	30.97	5.10	4.35	0.39	0.65	31.87	27.19	2.44	2.03	3.82	2.23
	Maximum.....	44.95	2.02	1.60	1.20	0.12	0.18	10.00	7.50	0.75	0.56	1.00	0.37
Canned sausages:	25	58.51	21.82	2.23	1.82	0.19	0.21	13.92	11.37	1.21	0.67	2.86	1.02
	Average.....	88.61	41.34	3.86	3.86	0.62	0.42	24.12	20.75	3.88	1.31	6.37	4.68
	Maximum.....	44.02	3.53	0.52	0.38	0.03	0.04	3.25	2.38	0.10	0.12	1.35	0.04



various common canned and preserved meats and meat products, and in one or two instances fresh meat has been included for comparison.

In obtaining the results from which the table on page 219 was compiled, but three divisions of nitrogenous substances were made, viz., (1) those insoluble in hot water; (2) those precipitated from the water extract by bromine; and (3) the flesh bases. Owing to the incompleteness of the bromine precipitate, the figures given there for nitrogen precipitated by bromine are somewhat high, and those for nitrogen as meat bases are correspondingly low. This fact was observed during the progress of the work, and pointed out in the text with the statement that "considering the small amount of these bodies contained in meat, the results are believed to be approximately correct."

#### SAUSAGE.

**Nature and Composition.**—Sausages are made from finely chopped meat, highly seasoned with various spices, and, as usually sold, stuffed into casings made of the cleaned and prepared intestine-skin of cattle, sheep, hogs, or even horses. The meat most commonly used is pork. Sausages are frequently home-made, especially in farm communities, the chopped and seasoned meat being stuffed in cloth bags instead of casings. Any and all kinds of meat are used in sausages, and much that is undesirable and even unwholesome is undoubtedly most readily used up in this product. There is little doubt that horse meat occasionally gets into the hands of the marketmen to be worked up in the form of sausages mixed with other meat. The condition in respect to these matters has been greatly improved, however, by the increased vigilance of State and Federal authorities. Sausages are sometimes artificially colored, and in some cases contain so-called "fillers" in the nature of dried bread, corn meal, potato starch, crackers, waste biscuit, boiled rice, etc.

#### CHEMICAL COMPOSITION OF SAUSAGES.\*

Kind.	No. of Analyses.	Ref-use.	Water.	Protein.		Fat.	Total Carbohy-drates.	Ash.	Fuel Value, Cals.
				NX6.25	By Difference.				
Farmer: edible portion...	1	.....	23.2	29.0	27.2	42.0	.....	7.6	2310
as purchased....	1	3.9	22.2	27.9	26.2	40.4	.....	7.3	2225
Pork: as purchased....	11	.....	39.8	13.0	12.7	44.2	1.1	2.2	2125
Bologna: edible portion...	8	.....	60.0	18.7	18.4	17.6	0.3	3.7	1095
as purchased....	4	3.3	55.2	18.2	18.0	19.7	.....	3.8	1170
Frankfort: as purchased. .	8	.....	57.2	19.6	19.7	18.6	1.1	3.4	1170

\* U. S. Dept. of Agric., Off. of Exp. Stations, Bul. 28 (Revised Ed.).



**Adulteration of Sausages with Starchy Materials and Water.—**

Robison, who has made a special study of these forms of adulteration at the Michigan Dairy and Food Department, states as follows: \* “Lean meat carefully chopped has an enormous combining power and can be made to take up a great quantity of water. Frankfurts, bologna, and pork sausage have been found to be adulterated with from 0.5 to 5% of starch, indicating an addition of approximately 1 to 10% of so-called cereal (chiefly corn flour), and from 5 to 40% of water in addition to that contained in the meats when in their fresh condition. The main excuse for the use of water is that it renders the meat of such a consistency that it may be easily stuffed into thin cases, such as are usually used for sausages that are eaten without removing the casing. As a matter of fact, this addition is not necessary where fresh meats are used, nor with those cuts of meat which the American public is in the habit of using in the manufacture of sausages in the home. Without doubt, in sausages composed of ox hearts, ears, snouts, lips, etc., in considerable quantities, the addition of water may facilitate the stuffing into thin casings.

“Starch hastens and increases the absorbing or combining power of lean meat. In many instances where inferior products, such as ears, etc., are used, virtually it is the only absorbing agent present in the product. It then serves a two-fold purpose, first, giving an absorbing power to meat which it has not, or inflating the absorbing power of a meat which naturally is deficient in this respect, and second, acting as a skeleton or framework, thereby disguising shrinkage during the process of cooking. Generally, added water and cereal are evidences of inferiority, and they are by no means infrequently added with the very purpose of concealing such inferiority.

“The evidence of adulteration with water is the discrepancy in the ratio of the water to the protein in the sausage. This ratio in sausage made from the fresh carcass varies from 3 : 1 to 3.6 : 1, being on an average about 3.35 : 1.”

Baumann and Grossfeld † have also adopted the ratio of water to protein but express the latter in the form of nitrogen. Their average ratio is 18.3 : 1 while Robison's calculated to the same basis is 20.9 : 1. The data used in calculating their ratio are (1) 3.43 : 1, the ratio of water to organic fat-free solids as obtained in 640 samples of beef, mutton, pork,

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\* Personal communication.

† Zeits. Unters. Nahr. Genussm., 32, 1916, p. 489.



and horse sausage, and (2) 5.34, the per cent of nitrogen in the fat-free solids. They calculate the per cent of added water ( $W'$ ) from the per cent of total water ( $W$ ) and nitrogen ( $N$ ) by the following formula:

$$W' = W - (N \times 18.3).$$

Other authors have advocated the ratio of water to organic fat-free solids as the criterion. Aachen \* considers 4:1 a liberal maximum, 3:1 being the average, and calculates added water by the formula:

$$W' = W - 4S,$$

in which  $W'$  = per cent of added water,  $W$  = total water, and  $S$  = organic fat-free solids. Feder † also believes 4 : 1 a fair maximum ratio while Seel ‡ finds it too liberal and advocates 3.5 : 1. All of the foreign standards and formulas are not strictly applicable to American sausage; the conditions of manufacture and character of the products are quite different in different countries. Obviously, cooking and drying influence materially the water content. Sausage made from liver and meats other than muscle, blood, etc., belong in special classes.

Casein is said to be used in Europe as an ingredient of sausage. Although it performs a mechanical rôle, it belongs in a different class from starch because of the similarity of its nutritive properties to those of lean meat.

**Artificial Coloring Matter in Sausages.**—Owing to the rapid color changes which freshly chopped meat, especially beef and mutton, naturally undergo, it is a common practice to employ powdered saltpeter (p. 215). Treated in this manner, meat remains pink, owing to the action on the hæmoglobin of the oxides of nitrogen resulting from the nitrate. As much as 4 ounces of niter to 100 lbs. of meat is sometimes used. A larger quantity would result in a shriveled appearance. Sulphites act not merely to prevent decomposition, but also to retain the red color. The use of artificial colors has been common in the past, in order to permanently dye the flesh a bright red, similar to the tint which the oxy-hæmoglobin naturally imparts to the beef when fresh. A variety of colors have been employed for this purpose, such as red ocher, coal-tar dyes, cochineal, etc. They were sometimes used in admixture with preservatives. Their use has been limited to certain colors in this country, owing to the enforce-

\* Zeits. Unters. Nahr. Genussm., 25, 1913, p. 577.

† Chem. Ztg., 38, 1914, p. 709.

‡ Ibid., 39, 1915, p. 409.



ment of the regulations under the Federal meat inspection laws. Spanish red pepper or pimienta is employed more for coloring than for flavoring.

#### ANALYSIS OF MEAT.

In analyzing meats and meat products due regard must be paid to their perishable nature, and, for this reason, immediately after their receipt by the analyst the various determinations should be promptly begun and rapidly carried out. If delays are absolutely necessary, the samples, as well as some of the solutions, especially during the earlier course of the analysis, should be kept on ice to prevent decomposition. Even at low temperatures, however, both bacterial and enzymic decomposition occur, and the nature of the proteins is slowly changed. Refuse material, such as bones, skin, gristle, tendons, etc., are separated as completely as possible by means of a knife from the edible portion and weighed. The visible fat is further separated from the lean, both are weighed, and the latter, cut first into small pieces, is passed repeatedly through a sausage-machine or ordinary household meat-chopper, in order to reduce to a homogeneous, finely divided mass.

**Determination of Water.**—Weigh out 2 to 5 grams of the finely divided material into a tared platinum or porcelain dish, and dry to minimum weight in a boiling-water oven. A slight oxidation of the fat may introduce a trifling error, but, excepting for the most exact work, where the drying should be accomplished in an atmosphere of hydrogen, or at ordinary temperature over sulphuric acid *in vacuo*, the above method is sufficiently close.

**Trowbridge Vacuum Desiccator Method.\***—Fill a paper extraction cartridge, or a glass tube with filter paper bottom, two-thirds full with sand, add fat-free cotton, dry at 103° C., keep in a vacuum for a few hours, and weigh in a weighing bottle. Remove the sand to a porcelain dish, add 5 to 10 grams of the sample, mix thoroughly, transfer to the cartridge, wiping the dish carefully with the cotton. Place in a vacuum desiccator over sulphuric acid, exhaust to 1 mm. or less, close stop cock, and allow to stand 24 to 48 hours, rotating gently every 3 or 4 hours to mix in the watery surface-layer of the acid. Transfer to a desiccator containing fresh acid and proceed as before, weighing after 24 to 48 hours, which time is usually sufficient for constant weight.

**Robison Method for Sausage.**—Dry 100 to 500 grams on a porcelain plate at 70 to 90° C. over a radiator for 10 to 15 hours and grind, then carry

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 122, 1909, p. 219.



out the final drying on a 2 to 5-gram portion at the temperature of boiling water employing in exact work a stream of hydrogen. If the sample is rich in fat, carry out the drying on a sieve over a vessel to collect the fat which is separately weighed and dried for water determination.

**Determination of Fat.**—*Extraction Method.*—Extract 2 grams of the sample dried at  $100^{\circ}$  with anhydrous ether for sixteen hours as in the case of cereal products (p. 286). More complete extraction is obtained by grinding the residue in a mortar and repeating the process and still more complete by digestion with pepsin and intermittent treatment with the fat solvent, but this latter is both tedious and open to other errors.

*Baur and Barschall*\* proceed as follows: Heat 1 to 1.5 grams of the well-ground material in a 250-cc. Erlenmeyer flask under a reflux condenser on a water bath with 5 cc. of concentrated sulphuric acid and 5 cc. of water until the solution is complete, and then for 10 minutes additional. Dilute with 40 cc. of water, cool, add from a pipette 50 cc. of ether, shake vigorously for 2 minutes, then add 50 cc. of petroleum ether (b. pt.  $50$  to  $55^{\circ}$ ), and shake vigorously for 1 minute. Allow to settle for 15 to 20 minutes at  $18^{\circ}$ , pipette off 49.5 cc. of the ethereal solution, which experiments have shown is half of the total amount, filter through a cotton plug into a tared flask, wash with three portions of 2 cc. each of a mixture of ether and petroleum ether, add a minute amount of finely powdered pumice stone (insufficient to appreciably affect the weight), and distil off the solvent. Dry in a water oven, cool, and weigh as usual.

*Kita Centrifugal Method.*†—Place 2.5 grams of the well-ground meat in a Gerber milk test-bottle (open at one end) with 8 cc. of 1:1 sulphuric acid, or 5 grams in a Gerber cheese test-bottle (open at both ends) with 17 cc. of acid and heat in a water-bath at  $60$  to  $70^{\circ}$  with occasional agitation for 5 to 10 minutes or till the solution is complete. Add 1 cc. of amyl alcohol and sufficient 1:1 sulphuric acid to bring the layer of fat within the neck. Whirl in a centrifuge for 3 to 5 minutes, warm on a water-bath, and read the amount of fat on the scale. In some cases the centrifuging must be repeated.

**Examination of Fat.**—Shake a large portion of the original finely divided sample in a corked flask with petroleum ether boiling below  $60^{\circ}$  C., and digest for some hours. Pour off the solvent, remove most of the petroleum ether by distillation, and the last traces by allowing to stand

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\* Arb. Kaisl. Gsndhtsamt., 30, 1909, p. 50; 33, 1910, p. 563.

† Arch. Hyg., 51, 1904, p. 165.



in a vacuum desiccator over freshly ignited calcium chloride. Determine the usual constants as described in Chapter XIII. In minced preparations these constants furnish a possible clue to the variety of meat used.

**Determination of Acidity of Fat.**—*Pennington and Hepburn Method.*\* Weigh 10 grams of the fat, mechanically separated and ground in a meat chopper, directly into a 250 cc. Erlenmeyer flask, add 50 cc. of neutral alcohol, and phenolphthalein as indicator, and bring to a brisk boil. The hot alcohol dissolves the fat. Titrate immediately with tenth normal sodium hydroxide, shaking vigorously, until a pink color appears, which persists for one-quarter of a minute. Calculate the acid value from the amount of sodium hydroxide used, or the free oleic acid by multiplying the acid value by 0.503.

**Determination of Total Nitrogen.**—Proceed according to the Gunning or Kjeldahl method (Chapter IV) employing 2 to 5 grams of the material.

If the meat contains nitrates, as is true of corned beef and some other salted products cured with the addition of saltpeter, follow the method modified to include nitric nitrogen. Richardson does not obtain concordant results by the modified method, when a large amount of sodium chloride is present, and recommends preliminary boiling with 10 cc. of saturated ferrous chloride solution and 5 cc. of concentrated hydrochloric acid to remove nitric nitrogen after which the ordinary Kjeldahl or Gunning method may be employed.

Calculate protein using the factor 6.25. Although nitrogenous substances other than proteins are present and the factors for the individual proteins vary, this factor gives a fairly close approximation to the total nitrogenous substance present.

**Determination of Ammoniacal Nitrogen.**—*Distillation Methods.*—Ordinarily the ammonia, liberated by magnesia (freshly calcined magnesium oxide), is distilled into standard acid and the excess titrated back with standard alkali. This procedure gives results somewhat higher than the truth owing to the decomposition of other nitrogenous compounds.

*Richardson and Scherubel* † proceed as follows: Method I, Distil 100 grams of the material, 10 grams of magnesia, and 450 cc. of water until 200 cc. of the distillate have been collected; Method II, Extract 100 grams of the material three times with 150 cc. of 60% (by vol.) alcohol and distil the combined extracts as in Method I. In both methods employ phenol-

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\* Jour. Amer. Chem. Soc., 32, 1910, p. 568.

† Ibid., 30, 1908, p. 1527.



phthalein as indicator. If in method I the water driven off is replaced and the distillation is repeated, additional ammonia is obtained. After ten distillations in this manner the originators of the method obtained a total of 0.088% of ammoniacal nitrogen in a sample of fresh meat and 0.095% in one of frozen meat, whereas after the first distillation they obtained only 0.030 and 0.033% respectively. Method II gave results about one-third as high as by Method I and after the first distillation the increase was insignificant. Either method is suitable for comparative purposes as the increase during spoilage is relatively great.

*Folin Aeration Method modified by Pennington and Greenlee.\**—The ammonia, set free by sodium carbonate, is evolved at room temperature in a rapid current of air. The ingoing air is purified by passing through sulphuric acid in a flask provided with a safety bulb. It next passes through a liter flask containing 25 grams of the ground meat, 1 gram sodium carbonate, 250 cc. of water, and 25 cc. of alcohol, then through an empty flask to intercept spray into a 250 cc. flask containing tenth normal acid and finally through a 100 cc. flask, to catch acid carried over mechanically, to an air pump operated by an electric motor and provided with an anemometer. One pump and one air purifier suffices for four series of flasks, the current being divided by means of four-way tubes. A volume of 8000 cu. ft. passed through each series in 3 to 6 hours suffices to remove all the ammonia liberated.

**Separation and Determination of Nitrogenous Bodies.**—It is rarely necessary to go further than to divide the nitrogenous bodies into several main groups, according to their solubility in water or other solvents, and their behavior toward certain reagents. The nitrogen may be determined separately in each of these groups and by the approximate factor the corresponding substance or class of substances ascertained.

A portion of the sample is first exhausted with cold water, which removes the soluble proteins (soluble globulins and albumins, proteoses, and peptones) and meat bases, leaving behind the insoluble globulins, the sarcolemma, the albuminoids of the connective tissue (elastin, etc., also insoluble) and the collagen. By next exhausting with boiling water the collagen is removed in the form of soluble gelatin.

The coagulable albumins and globulins are precipitated in the cold water extract by boiling and in the filtrate from these, the proteoses by addition of zinc sulphate. The remainder of the nitrogen obtained by

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\* Jour. Am. Chem. Soc., 32, 1910, p. 561.



difference consists in large part of meat bases with a small amount of peptones, the accurate separation of which is impossible with our present knowledge, although the creatine and creatinine either separately or combined and the total purine bases may be quite accurately estimated.

**Determination of Nitrogenous Substances Insoluble in Cold Water.—**

It is customary to obtain the insoluble nitrogen by difference after determining the nitrogen in an aliquot of the cold water extract. The conversion factor for insoluble protein is 6.25.

*Trowbridge and Grindley Method.\**—Digest 1000 grams for 1 hour with 1500 cc. of ice water and squeeze through cheese cloth. Divide the residue into equal portions in beakers, wash one after the other with the same portion of water, filtering each time through cheese cloth, and repeat until the last filtrate is colorless, neutral to phenolphthalein, and free from protein as shown by the biuret test.

*Emmett Method.†*—Weigh 7 to 25 grams of the material, according to the water content, into a 150-cc. beaker, stir into a homogeneous paste with 5 to 10 cc. of ammonia-free water at 15° C., then add a further portion of 50 cc. Stir frequently for 15 minutes, allow to stand for 2 to 3 minutes, and filter into a 500-cc. graduated flask. Drain off the liquid, using a glass rod to press the meat residue. Wash by decantation with three portions each of 50 cc. and 25 cc. of water, stirring for 5 minutes each time and allowing to settle for 2 to 3 minutes. Transfer the residue to the paper and wash three times with 10 cc. of water. Make up to the mark and determine nitrogen in a 25-cc. aliquot portion.

Three aliquot portions of 150 cc. each may be used for the determination of (1) coagulable proteins and, in the filtrate, proteoses, (2) coagulable proteins and in the filtrate total creatinine (creatine and creatinine) and (3) purine bases. If other determinations are desired employ 14 to 50 grams of material and make up to 1000 cc.

Emmett rightly observes that by direct treatment with water a solution well adapted for the determination of the various forms of soluble nitrogen is obtained, thus avoiding any changes which might result from preliminary drying and extraction with ether.

*Pennington Method.‡*—This method was devised for chicken meat. Shake gently 60 grams of the finely divided meat in a 500-cc. cylindrical bottle with 300 cc. of water for 15 minutes, avoiding sufficient agitation to

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\* Jour. Amer. Chem. Soc., 28, 1906, p. 472.

† U. S. Dept. of Agric., Bur. of Chem., Bul. 162, 1913, p. 153.

‡ Ibid., Bul. 115, 1908, p. 64.



form an emulsion. Centrifuge 20 minutes, decant off the supernatant liquid onto a paper, add 300 cc. of water to the residue, and proceed as before, repeating the treatment until the protein is practically removed as shown by the biuret reaction. Usually a volume of 1500 to 2500 cc. is required. Employ thymol to prevent bacterial decomposition and a low temperature to inhibit the action of natural enzymes. Neutralize the total extract to litmus with N/10 sodium hydroxide. Determine nitrogen in a 100-cc. aliquot portion which is evaporated to 10 cc. before adding the acid. Other aliquot portions may be used for determining coagulable and other forms of soluble nitrogen.

Considerable difficulty is experienced in complete removal of soluble proteins from the dark meat, especially of cold-storage fowls. The extraction should not be continued longer than 26 hours.

Cook shakes 200 grams for 3 hours with 250 cc. of water in a shaking machine and washes in linen bags with 2200 to 2500 cc. of water. Weber\* finds that higher results are obtained by this method using ice water than with water at room temperature.

**Determination of Collagen and Gelatin.**—The connective tissue of fresh meat consists in large part of collagen which on boiling with water is slowly converted into gelatin. In canned meat or other cooked meat products this conversion has been partly effected. In either case the common methods of determination are based on boiling the cold water insoluble material with water until the conversion into gelatin is as complete as possible. The nitrogen is either determined directly in the filtered extract or in the alcohol insoluble portion of the extract as obtained by Stutzer's method. The factor for both collagen and gelatin is 5.55.

*Direct Method.*—The residue from the cold water extract obtained by Emmett's method may be used for the determination. Boil for several days with water, filter, make up to a definite volume, and determine nitrogen in an aliquot of about one-fifth the total volume.

*Stutzer Method.*†—This method was originally designed for meat extracts but has been used by Bigelow for meats. Evaporate another aliquot of one-fifth of the hot-water extract with sand and dry in a water oven. Treat with four portions of 100 cc. of absolute alcohol, decanting each time on a Büchner funnel connected with a suction flask and provided with a layer of long fiber asbestos. Place the beaker in ice water, stir for

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 122, p. 42.

† Zeits. anal. Chem., 34, 1895, p. 568.



2 minutes with 100 cc. of a mixture of 100 grams of alcohol, 300 grams of powdered ice, and 600 grams of ice water, decant onto the filter adding a small piece of ice. Repeat the treatment three times or until the solution is colorless keeping the decanted portions in ice water during filtration, use two or three Büchner funnels if the filtration is slow and take care that the temperature does not rise above  $+5^{\circ}$  C. Finally boil the mixture of sand and residue, together with the asbestos, with several portions of water, filter, wash with hot water, evaporate the filtrate, and determine nitrogen in an aliquot.

**Determination of Myosin.**—König\* states that myosin may be obtained in the residue after boiling with water by digestion with 15% ammonium chloride solution, filtration and precipitation of the myosin in the filtrate by diluting and boiling or by salting out with sodium chloride or magnesium sulphate. The myosin separated by filtration is dissolved in a definite volume of concentrated sulphuric acid and an aliquot used for nitrogen determination. He considers that the nitrogenous matter remaining undissolved after successive treatment with cold water, boiling water, and 15% ammonium chloride solution is sarcolemma or insoluble muscle fiber.

The methods for collagen and myosin are not entirely satisfactory especially in view of our imperfect knowledge of the albuminoids and globulins, present in meat.†

**Determination of Coagulable Protein (Albumin).**—Grindley and Emmett Method.‡—Evaporate 150 cc. of the cold water extract obtained as directed by Emmett to 40 cc. If necessary add carefully very dilute acetic acid or sodium hydroxide solution until faintly acid to litmus paper and boil. Collect the coagulum on a filter, wash with hot water, and determine nitrogen, correcting for any nitrogen that may be present in the paper.  $N \times 6.25 =$  coagulable protein or albumin.

Trowbridge and Grindley § obtain the maximum results in fresh beef by neutralizing one-fourth of the acidity to phenolphthalein before coagulation.

Some analysts filter before neutralization and determine separately

\* *Chemie der Menschlichen Nahrungs- und Genussmittel*, 4. Aufl. III Bd. 2 Th., 1914, p. 24.

† See v. Fürth, *Arch. Path. Pharm.*, 37, 1896, p. 389; *Ergebnisse der Physiologie* Ab. 1, I, 1902, p. 110; *Ibid.*, Ab. 2, I, 1903, p. 575.

‡ *Jour. Amer. Chem. Soc.*, 27, 1905, p. 665; U. S. Dept. of Agric., Bur. of Chem., Bul. 162, p. 146.

§ *Jour. Am. Chem. Soc.*, 28, 1906, p. 494.



any precipitate that forms on boiling after neutralizing. The second precipitate is known as syntonin or acid albumin.

The filtrate from one duplicate may be used for determining proteoses, the filtrate from the other for determining total creatinine.

**Determination of Proteoses, Peptones, Creatine, Creatinine, Purine Bases, and Total Meat Bases.**—Follow the methods as described under meat extracts (pp. 253 to 258).

No accurate method is available for the determination of total meat bases. The practice of obtaining them from the remainder after subtracting the sum of the coagulable and proteose nitrogen from the total water soluble nitrogen, or after subtracting the proteose nitrogen from the total nitrogen in the filtrate from the coagulable nitrogen as followed by Richardson, does not correct for the peptones or related substances thus determined with the bases. Bigelow and Cook's procedure of calculating the nitrogen in the filtrate from the tannin-salt precipitate, correcting for nitrogen in the reagent and ammonia in the sample, and multiplying by 3.12 to obtain the meat bases is subject to error in that part of the creatine is precipitated by the tannin-salt reagent and that the factor is accurate for only one base, creatine; furthermore, according to Richardson, the method is difficult to handle and obtain concordant results due (in part at least) to nitrogen in the reagent. The estimation of the "peptones" by subtracting the sum of the coagulable, proteose, and meat base nitrogen by the tannin-salt method from the total soluble nitrogen and multiplying by 6.25 is likewise unsatisfactory although probably the best procedure now available.

**Determination of Ash.**—Incinerate the residue from the total solids in the original dish at a low red heat. It is usually advantageous, especially in the case of salt meat, to exhaust the charred sample with water, collect the insoluble residue on a filter and ignite. The filtrate is then added, evaporated to dryness, and the whole heated to low redness and weighed. A perfectly white ash is difficult to obtain.

**Determination of Mineral Constituents.**—Determine in the original meat total sulphur and in the ash chlorine, potassium, sodium, phosphoric acid, and other mineral constituents, following the usual methods of analysis. The scheme for ash analysis given on page 310 is applicable to meat ash.

**Determination of Acidity.**—The acidity of meat is due largely to *d*-lactic acid with small amounts of succinic, acetic, and other acids. The results by direct titration are usually calculated in terms of lactic acid.



Mondschein found that about one-third of the acid is held back in the coagulum and proposes the following method:

*Mondschein Method.\**—Suspend 50 grams of the finely ground sample in 60 to 80 cc. of water, coagulate by boiling, filter by suction, and wash with boiling water three times or until the reaction is no longer acid. Titrate the filtrate with N/10 alkali using phenolphthalein as indicator. Calculate as free lactic acid.

Remove the matted coagulum from the filter, stir up in a beaker with 50 cc. of water, add 10 cc. of 10% sodium hydroxide solution, and boil, taking care that the liquid does not froth over, thus liquefying the mass except for a few particles. Add 100 cc. of saturated sodium chloride solution, heat to boiling, and saturate at boiling heat with solid sodium chloride. Filter the precipitate thus formed with the aid of suction, wash with a hot saturated sodium chloride solution, and add to the filtrate sulphuric acid to faint acid reaction, thus precipitating proteins. Boil, add 5 cc. of sulphuric acid, make up to 500 cc., and filter through a dry paper. Heat 250 cc. of the filtrate to boiling and add N/10 potassium permanganate solution until the lactic acid is split up into acetaldehyde, carbon dioxide, and water.† Add an equal bulk of potassium bisulphite solution (12 grams per liter), the strength of which has been determined against N/10 iodine solution, mix, and after 15 minutes titrate with N/10 iodine solution 1 cc. of which equals 0.005 gram lactic acid.‡

Mondschein also describes a more complicated method for use in separating *d*-lactic from  $\beta$ -hydroxybutyric acid, which however does not appear to have practical application in meat analysis.

**Determination of Starch.**—In the following methods it is assumed that glycogen is not present in sufficient amount to appreciably affect the results. If microscopic examination shows the presence of starch and horse meat or liver is suspected, follow the Mayrhofer-Polenske method (p. 234).

*Mayrhofer Method.*—The first paragraph of the description of the Mayrhofer-Polenske method is essentially the Mayrhofer method as originally devised for starch.

*Mayrhofer-Price Method.§*—Heat on a boiling water bath 10 grams of finely-divided meat with 75 cc. of 8% potassium hydroxide in 95% alcohol

\* Biochem. Zeits., 42, 1912, pp. 91, 105.

† v. Fürth and Charnass, Biochem. Zeits, 26, 1910, p. 199.

‡ Ripper, Monatsh. Chem., 21, 1900, p. 1079.

§ U. S. Dept. of Agric. Bur. of Anim. Ind. Circ., 203, 1912.



until all the meat is dissolved (30 to 40 min.). Add an equal volume of 95% alcohol, cool, and after 1 hour decant carefully on a Gooch crucible with a thin layer of asbestos. Wash carefully by decantation twice with 4% potassium hydroxide in 50% alcohol and twice with warm 50% alcohol. Add to the residue and crucible with contents 40 cc. of water and then with constant stirring 25 cc. of concentrated sulphuric acid. After 5 minutes add 40 cc. of water and heat just to boiling with constant stirring. Transfer to a 500-cc. graduated flask, add 2 cc. of 20% phosphotungstic acid solution, cool, make up to the mark, mix, and filter through starch-free filter paper. Neutralize an aliquot portion of the filtrate and determine dextrose by one of the methods described in Chapter XIV. Price recommends Low's method.\*

**Identification of Horse Flesh.**—Although certain authorities have found distinguishing characteristics in color, consistency, odor, etc., between horse flesh on the one hand, and beef and pork on the other, it is extremely difficult, by its physical properties, to detect horse flesh when mixed with other meat, especially when the mixture is chopped. Horse flesh has a much coarser texture and is darker in color than beef. The muscle fibers are, as a rule, shorter in horse flesh. On treating horse flesh with formaldehyde, Ehrlich † has found that a very characteristic odor is developed within forty-eight hours, suggestive of roasted goose flesh.

Certain of the constants of the fat of horse meat differ from those of beef and pork, notably the iodine value and the refractometer readings. These constants are compared as follows:

	Iodine Value.	Butyro-refractometer Readings. Temperature 40°.
Horse fat.....	71-86	53.7
Beef fat.....	38-46	49.0
Hog fat.....	50-70	48.6-51.2

The fact that glycogen usually exists to a much larger extent in horse-flesh than in other meat, and that a considerable amount remains after that of other meat has disappeared, renders it possible in some cases to detect horse flesh, when present in the mixture.

The following table prepared by Bujard shows the relative amount of glycogen in various kinds of meat and sausages:

\* Jour. Amer. Chem. Soc., 24, 1902, p. 1082.

† Zeits. Fleisch Milchhyg., 1895, p. 232.



	Water.	Glycogen Direct.		Glycogen in Dried Substance.	
		Niebel Method.	Mayrhofer Method.	Niebel.	Mayrhofer.
Horse flesh.....	74.44	0.440	0.445	1.721	1.741
".....	74.87	0.600	0.520	2.388	2.069
".....	76.17	1.827	1.727	7.667	7.247
".....	76.00	0.592	0.610	2.466	2.542
Red sausage (Knackwurst).....	69.26	.....	0.038	.....	0.124
Pork sausage.....	67.25	.....	0.24	.....	0.733
Veal.....	74.6	.....	0.086	.....	0.342
Pork.....	75.0	.....	0.186	.....	0.744

In beef Bujard found 0.073 and 0.74 per cent of glycogen calculated in terms of dried substance, and, in sausages made exclusively from horse meat, amounts of glycogen ranging from 0.05 to 5.34, the sample in the latter case being made from the liver. It was formerly thought possible to detect as small an amount as 5% of horse flesh in mixture, but later investigation showed that after the death of the animal, glycogen, though present at first in considerable quantity, decomposes more or less rapidly, going over into muscle sugar (dextrose). Hence, while the presence of much glycogen is suspicious, its absence is by no means proof that horse flesh was not used.

Niebel did not consider the failure of the glycogen test as sufficiently conclusive to establish the absence of horse flesh, on account of the tendency toward decomposition of the glycogen. In the absence of starch, he regards the presence of more than 1% of dextrose in the fat-free meat, after conversion of the carbohydrates, to be proof of the presence of horse-flesh.

**Detection of Glycogen.**—From the well-known reaction produced by iodine on glycogen, horse flesh can often be detected, when present in sausages, unless obscured by the presence of starch or dextrin.

Brautigam and Edelmann\* proceed as follows: 50 grams of the finely divided meat are boiled with 200 cc. of water for an hour, and, after cooling, dilute nitric acid is added to the broth to precipitate the proteins and to decolorize. The broth is then filtered, and a portion of the filtrate is treated in a test-tube with a freshly prepared, saturated, aqueous solution of iodine, or, better, with a mixture of 2 parts iodine to 4 parts potassium iodide and 100 parts water, the reagent being carefully added so as not

\* Pharm. Central., 1898, p. 557.



to mix with the broth, but form a layer above it. If glycogen be present in considerable amount, a wine-colored ring is observable at the junction of the two layers. On heating the test-tube, the coloration disappears if due to glycogen, but it reappears on cooling. This reaction was found to occur with horse flesh and not with beef, mutton, veal, or pork.\*

If the color is not clearly apparent, the chopped meat is heated on the water-bath with a solution of potassium hydroxide (using an amount of potassium hydroxide equivalent to 3% of the weight of the flesh) till the fiber is decomposed, after which the broth is concentrated to half its volume, treated with nitric acid to precipitate the proteins, filtered, and treated with the iodine solution as previously.

**Determination of Glycogen in the Absence of Starch.**—*Pflüger Method.*† —Heat on a boiling-water bath 100 grams of the material with 100 cc. of 60% potassium hydroxide for 3 hours, cool, transfer to a large beaker, dilute to 400 cc., precipitate with 800 cc. of 95% alcohol, and allow to settle over night. Decant off the liquid as completely as possible onto a filter, fill up the beaker with 66% alcohol containing 1 cc. of saturated sodium chloride per liter, and stir vigorously for a long time. After the glycogen settles decant off the liquid, repeat twice the washing with the 66% alcohol, and then wash twice with 95% alcohol, once with absolute alcohol, three times with absolute ether, and three times with absolute alcohol. Dissolve the glycogen in a small amount of hot water, make slightly acid with acetic acid, filter, and fill up to a definite volume. Determine the sugar in the solution either by direct polarization, specific rotation of glycogen  $+196.57^\circ$ , or by conversion into glucose and copper reduction.

In the latter case invert 100 cc. by heating for 3 hours on a water-bath with 5 cc. of hydrochloric acid (sp.gr. 1.19) and proceed according to the Allihn method. Dextrose  $\times 0.927$  = glycogen.

**Separation and Determination of Glycogen and Starch.**—*Mayrhofer-Polenske Method.*‡ —Dissolve 50 grams of the ground meat, containing as little fat as possible, in a 450-cc. beaker with 150 cc. of a solution of 80 grams of potassium hydroxide in 1 liter of 90% (by vol.) alcohol, by warming on a water-bath with occasional stirring which requires about  $\frac{1}{2}$  hour. Add 100 cc. of 50% alcohol to the hot liquid, cool, and filter by suction in Witt's or some other suitable filtering device. Wash the residue with

\* The reaction was found to occur also with the flesh of the human foetus and with the foetus of animals; also with mule meat, but not with the flesh of the dog or cat.

† Pflüger's Arch. Ges. Physiol., 103, 1903, p. 169; 114, 1906, p. 231.

‡ Arb. Kaisl. Gsndtsamt., 24, 1906, p. 576.



30 cc. of alcoholic potash at 50°, then with 90% cold alcohol until the filtrate no longer becomes turbid with a few drops of dilute hydrochloric acid. Transfer the insoluble residue to a 110-cc. graduated flask, add 50 cc. of normal aqueous potassium hydroxide and heat  $\frac{1}{2}$  hour on a water bath to dissolve the glycogen and starch. On cooling acidify the solution with concentrated acetic acid, make up to the mark, and filter. To 100 cc. of the filtrate add 150 cc. of absolute alcohol and after the glycogen and starch have settled (12 hours) collect on a tared Gooch crucible or filter paper. Wash with 70% alcohol until the filtrate contains no more solid matter and finally with a little absolute alcohol and ether. Dry first at 40°, then at 100° to constant weight, determine ash in a portion and deduct. Multiply by 2.2 to obtain the percentage of glycogen and starch in the meat.

The method up to this point is in all essential details the original Mayrhofer\* method for determination of starch, the amount of glycogen present in aged meat products, such as sausage containing no horse flesh, being too small to appreciably viciate the results in determining a considerable addition of starch. Only when starch has been found by microscopic examination or the iodine test and the presence of meat with high glycogen content, such as horse flesh or liver, is suspected, is it necessary to attempt a separation by the following procedure:

Weigh out 0.3 to 0.5 gram of the precipitate, dissolve in 30 to 40 cc. of water, and add double the volume of saturated ammonium sulphate solution. Allow to stand  $\frac{1}{2}$  hour and separate the precipitated starch from the solution of glycogen by filtration. Before washing the precipitate, test the filtrate with a dilute solution of iodine in potassium iodide and if a blue color appears, add saturated ammonium sulphate solution to the filtrate and after the precipitate which forms on standing has settled, filter on the paper containing the former precipitate. If, however, the color obtained with the iodine solution is red-violet by reflected and Bordeaux-red by transmitted light, the second precipitation is unnecessary. Wash the precipitate on the paper three times with half-saturated ammonium sulphate solution, then dissolve with normal sodium hydroxide into a beaker, wash first with normal sodium hydroxide, and finally with water. Neutralize the opalescent filtrate with acetic acid and precipitate with alcohol as above described. Collect on a tared Gooch crucible or filter paper, wash first with 50% alcohol and finally with absolute alcohol, dry at 100°, and weigh.

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\* Forsch. Ber. Lebensm., 3, 1896, pp. 141, 429.



The glycogen may be obtained by difference, subtracting the weight of starch from that of starch and glycogen previously found, or directly as follows: Dilute the filtrate from the precipitated starch with three to four volumes of water, add an equal volume of alcohol, allow to settle 12 hours, filter, wash with 50% alcohol, and dissolve in a small quantity of water. Reprecipitate the glycogen in the strongly opalescent solution with alcohol, collect on a tared Gooch crucible or filter paper, wash with alcohol, dry at 100°, and weigh.

**Identification of Raw Horse Flesh by Biological Tests.**—*Precipitin Test.*—This test depends upon the principle developed by Uhlenhuth and others,\* that when a rabbit has been inoculated with the blood of a particular animal, as for instance that of the horse, the serum of the rabbit's blood will react with the blood of the horse and with that of no other animal. Only raw flesh responds to the test, as heating destroys the reacting substance.

To prepare the serum (antiserum) reagent, inject into a rabbit, either subcutaneously or intravenously, 5 cc. of defibrinated horse blood and repeat the treatment several times allowing intervals of 2 to 5 days between the treatments and increasing the dose up to 10 cc. or more until the serum of the blood drawn from the rabbit shows the proper activity. When of high potency it should react with horse blood serum diluted with 20,000 parts of 0.85% (physiological) salt solution but ordinarily a lower potency is sufficient. To obtain the serum from the blood, allow a few cubic centimeters to coagulate spontaneously. If the blood is replaced by 0.85% salt solution the life of the animal may be preserved.

Prepare an extract of 50 grams of the finely ground sample, previously shaken with chloroform or ether if much fat is present, by soaking for 3 hours at room temperature or over night in an ice box with 100 cc. of 0.85% salt solution. Test the filtered extract to determine if it is of the proper dilution (1 part albumin per 300 cc.) by heating 1 cc. with 1 drop of nitric acid. If a decided turbidity forms in 5 minutes and settles as a precipitate it is of suitable strength. Neutralize with 0.1% sodium hydroxide solution if acid. To 1 cc. of the extract add carefully without mixing 0.1 cc. of the antiserum. Treat in like manner for comparison 1 cc.

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\* Uhlenhuth, Deutsch. Med. Wochs., 1901, p. 780; Wassermann and Schütze, Ibid., 1902, p. 483; Schütze, Ibid., 1902, p. 804; Miessner and Herbst, Arch. wis. prakt. Tierheilk., 1902, p. 359; Wassermann, Zeits. Hyg., 2, 1903, p. 267; Uhlenhuth, Weidanz and Wedemann, Arb. Kaisl. Gsndhtsamt., 1908, p. 449; Gaujoux, Hyg. viande lait, 4, p. 65, 132; Uhlenhuth and Weidanz, Schweiz. Wochs., 48, p. 724.



portions of extracts prepared from horse and other meat. If a cloudiness and finally a decided precipitate forms within 30 minutes the presence of horse meat is indicated.

For further particulars the reader is referred to the papers given in the foot notes.

The preparation of the antiserum, if not the conducting of the actual test, falls properly within the province of the biologist or serologist who has at his command suitable experimental animals and is experienced in judging the tolerance of the rabbit for the injections as well as in carrying out other details of serum work.

*The Compliment Fixation Test* is said to be even more delicate than the foregoing. Details of the process are given by Seiffert.\*

**Determination of Sugars.**—The small amount of dextrose naturally present in meat and the sucrose added to ham and other salted meats in curing are best determined by copper reduction. After removal of interfering substance by suitable reagents, W. B. Smith † precipitates with picric acid and phosphotungstic acid thus removing proteins, which have a solvent action on cuprous oxide, and creatinine, which reduces Fehling solution.

*Smith Method.*—Boil 50 grams of the finely-ground sample, as free as possible from fat, with 150 cc. of water for 15 to 20 minutes., cool, add 1 to 5 grams of solid picric acid and 15 to 20 cc. of 20% phosphotungstic acid solution, and make up to 250 cc. exclusive of the fat. Filter through a dry paper and make up 150 cc. of the filtrate to 160 cc. with 8 cc. of concentrated hydrochloric acid and 2 cc. of water. Mix, filter through a dry paper and without delay determine reducing sugars (as dextrose) in an aliquot, after neutralizing, by one of the usual methods. To another portion of the filtrate add concentrated hydrochloric acid sufficient to bring the total amount present up to one-eleventh of the total volume. Neutralize and determine the dextrose by copper reduction. Deduct the amount obtained by direct inversion and calculate as sucrose.

*Hoagland ‡* precipitates creatinine and other nitrogenous constituents with phosphotungstic acid alone and removes the excess with potassium chloride thus avoiding the possible inversion of sucrose by free hydrochloric acid and the necessity of neutralizing.

\* Zeits. Hyg. Infektionskr., 71, 1912, p. 547; König, Chemie der Menschlichen Nahrung- und Genussmittel, 3, 1 Th., 1914, p. 340; 2 Th., p. 44.

† Jour. Ind. Eng. Chem., 8, 1916, p. 1024.

‡ Jour. Biol. Chem., 31, 1917, p. 67.



**Detection and Determination of Sulphurous Acid.**—Proceed as directed in Chapter XVIII.

Traces should be ignored, as slight reactions for sulphurous acid are obtained with meats that have not been chemically preserved.

Winton and Bailey\* found that in 50 grams of fresh beef, mutton, veal, and pork not more than 0.1 mg. of sulphur dioxide and no hydrogen sulphide was present, whereas on decomposition as high as 2.1 mgs. of sulphur dioxide and 3.4 mgs. of hydrogen sulphide were developed. From these figures it is evident that the examination for sulphur dioxide should be made only on the fresh meat.

Folck and Farreras† use iodate-starch paper for detecting sodium hydrogen sulphite in meat. They prepare the test paper by mixing a carefully prepared starch paste (2.5 grams of starch to 95 cc. of water), after cooling, with a solution of 1 gram of sodium iodate and 2.4 grams of citric acid in 5 cc. of water, saturating filter paper with the mixture, and drying in the dark protected from fumes. In applying the test macerate 5 to 15 grams of the meat for 5 minutes with sufficient water to cover, strain, and test the liquid with the iodate-starch paper which becomes blue in the presence of the sulphite. The method compares favorably with the Rosell method depending on the decolorization of 1% potassium permanganate solution by the water extract of the meat.

**Detection of Boric Acid.**—Boil 25 grams of the ground material with 50 cc. of water, cool, and filter on a wet paper to remove fat and meat fibers. Test the acidulated aqueous extract with turmeric paper as directed under milk.

A more delicate method of procedure consists in burning to an ash a portion of the meat, after treatment with lime water, and testing with turmeric tincture a solution of the ash slightly acidified with hydrochloric acid.

**Determination of Boric Acid.**—See Chapter XVIII.

**Detection of Benzoic Acid.**—Proceed with a portion of the aqueous solution, prepared as above, according to the instructions given in Chapter XVIII or prepare a special solution as follows:

*La Wall and Bradshaw Method modified by Fischer and Gruenert.‡*—Agitate 50 grams of the finely ground sample for 30 minutes with 100 cc. of 50% alcohol acidified with sulphuric acid. Strain through cloth, add

\* Jour. Amer. Chem. Soc., 29, 1907, p. 1499.

† Boll. chim. farm., 53, 1914, p. 106.

‡ Zeits. Unters. Nahr. Genussm., 17, 1909, p. 721.



sodium hydroxide solution to alkaline reaction, and evaporate on the water-bath until all the alcohol is removed. Make up to 50 cc., add 5 grams of sodium chloride, acidify with sulphuric acid, heat to boiling, cool, and filter. Shake the filtrate with ether in a separatory funnel, wash the ethereal solution with a little water, and evaporate to dryness at a gentle heat. Test the residue as described in Chapter XVIII for both benzoic and salicylic acids.

**Determination of Benzoic Acid.**—Prepare the solution as described in the foregoing method, except that the meat is extracted with several portions of 50% alcohol, dealcoholize in an alkaline solution and proceed according to the La Wall and Bradshaw method, page 893.

*Krüger Method.\**—Place 50 grams of the ground sample, containing 70 to 75% of water, in a Kjeldahl flask with 45 cc. of 70% sulphuric acid. If less than 70% or more than 75% of water is present in the sample use less or more of the material and adjust the strength and amount of acid accordingly. Connect with a steam-distillation apparatus and heat cautiously with shaking, using an asbestos pad with a round hole cut in the middle to confine the heat to the portion of the flask in contact with the liquid. When the solution becomes clear distil in a current of steam regulating the heating of the flask so that the volume remains constant and a distillate of 500 cc. is obtained in about 75 minutes.

Filter the distillate, which must be cool as it flows from the condenser, wash with a little cold water, add sodium hydroxide solution to faint alkaline reaction, evaporate to small volume and transfer to a porcelain dish of 100-cc. capacity. Heat on a boiling water-bath and add in small portions with stirring sufficient cold saturated potassium permanganate solution to form a red color that persists for five minutes. Destroy the excess of permanganate with cold saturated sodium sulphite solution and evaporate to about 10 cc. Transfer to a separatory funnel, acidify with 1 : 3 sulphuric acid, dissolve the precipitate remaining in the dish with small portions of the sodium sulphite solution and dilute acid using the mixture to rinse the dish. Extract the solution, which should not exceed 20 cc. in bulk three times, with an equal volume of a mixture of ether and petroleum ether. Wash the combined extract three times with 3 cc. portions of water, and remove the last traces of water by shaking with the quantity of powdered gum tragacanth that is held on the end of a small knife blade. Transfer to a weighed glass dish, using a mixture of ether and petroleum-ether for rinsing, allow

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\* Zeits. Unters. Nahr. Genussm., 26, 1913, p. 12.



to evaporate at room temperature, dry 2 hours over soda lime and weigh. As a check dissolve in neutral alcohol and titrate with N/10 sodium hydroxide using phenolphthalein as indicator. If the weight of benzoic acid is less than 30 mg. the results may be high in which case the benzoic acid is removed by sublimation and the dish reweighed.

**Detection of Salicylic Acid.**—Test a portion of the ether extract, obtained as described for benzoic acid, with ferric chloride solution. A deep-violet coloration indicates salicylic acid.

**Detection of Starch in Sausages, Meat-balls, etc.**—The addition of cracker or bread crumbs is best indicated by the presence of considerable starch, which is readily recognized by the iodine test, applied by boiling up a portion of the sample with water, cooling and adding a drop of iodine reagent to the liquid. The characteristic blue color is produced, if starch be present in notable quantity. Traces of starch may be due to the pepper and spices used in seasoning the sausage. A small admixture of starch is rendered apparent if a small portion of the sausage is treated with a drop of iodine reagent and viewed under the microscope. A microscopical examination will sometimes reveal the character of the starch, whether it is from cereals or from pepper, but in some preparations the starch is thoroughly cooked and its structure destroyed.

**Detection of Coloring Matter.**—*Red Ocher* is indicated by an excessive amount of iron in the ash.

*Cochineal* is most readily tested for by the method of Klinger and Bujard.\* The sausage, finely divided, is heated with two volumes of a mixture of equal parts of glycerin and water for several hours on the water-bath, the mixture being slightly acidified. The yellow solution is passed through a wet filter, and the coloring matter, if present, is precipitated as a lake by adding alum and ammonia, the precipitate is filtered off and washed, after which it is dissolved in a small amount of tartaric acid, and the concentrated solution, contained in a test-tube, is examined through the spectroscope for the characteristic absorption-bands of carmine lake, lying between *b* and *D*.

Spaeth † has shown that both *carmine* (cochineal) and *anilin red*, which are the dyes most commonly used for coloring sausages, can be most readily extracted therefrom by warming the finely divided material a short time on the water-bath with a 5% solution of sodium salicylate.

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\* Zeits. angew. Chem., 1891, p. 515.

† Pharm. Central., 38, 1897, p. 884.



*Vegetable and Coal-tar Colors.*—In addition to the solvents named above various others, such as methylated spirits (Allen), acidified alcohol (A. S. Mitchell), amyl alcohol, ether, ammonia, and those used in the examination of fats and oils (Chapter XIII), are useful in special cases. The solvent, after filtering, is evaporated to small volume, acidified with hydrochloric acid, and white wool is boiled in it. If the wool is distinctly dyed, a coal-tar color is undoubtedly present, and this can often be identified by methods given in Chapter XVII. According to Marpmann, pure normal flesh containing natural color only is completely decolorized by macerating for two hours in 50% alcohol, while artificially colored meat remains colored after this treatment. Richardson\* warns against mistaking for an artificial color the bright red substance often extracted by ether or alcohol and ether from meats cured with saltpeter.

*Marpmann's Microscopical Methods.*†—Moisten a thin section of the sausage with 50% alcohol, and examine under the microscope. Some colors are readily apparent without further treatment. If only traces of color are present, clarify the substance by treatment with xylol, which is removed by the use of carbon tetrachloride. The mass rendered transparent by this treatment is then immersed in cedar oil and examined, the coloring matters, if present, being especially apparent. If the color used is fuchsin (magenta), carmine, logwood, or orchil, the substance of the cell will appear stained. If acid coal-tar dyes are used, the liquid contents of the cell will show the color.

**Detection of Frozen Meat.**—Maljean‡ detects frozen meat by the aid of a microscope. A drop of the blood or meat juice is pressed out upon a slide and immediately examined before it solidifies. Fresh meat juice contains many red blood corpuscles, floating in a clear colorless serum, and readily apparent. In blood from frozen meat, the red corpuscles are nearly always completely dissolved in the serum, due to freezing, or, if not dissolved, are much distorted and entirely decolorized, the liquid portion being darker than usual.

Megascopically, the fresh meat juice is more abundant than that of frozen meat, and its color is deeper. According to C. A. Mitchell, if a small piece of meat once frozen be shaken in a test-tube with water, color is imparted to the water much more quickly than with fresh meat, and the color is deeper.

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\* Allen's Commercial Organic Analysis, Phila., 1914, 8, p. 364.

† Zeits. angew. Mikros, 1895, p. 2.

‡ Jour. pharm. chim., 25, 1892, p. 348.



## MEAT EXTRACTS AND SIMILAR PRODUCTS.

**Meat Extracts.**—*Methods of Manufacture.*—Numerous preparations sold under the name of meat extracts have been on the market for many years. At the beginning of the nineteenth century the value of such extracts was known, but Liebig was the first some fifty years later to produce a commercial extract of meat. Liebig's preparation, as originally made, consisted of a cold-water extract of chopped lean meat, strained free from fiber, heated, filtered, and evaporated, thus containing little if any gelatin or proteins. Later, however, Liebig advocated the use of warm and even boiling water for extraction, by which method of preparation a greater amount of gelatin is brought into solution. He, however, condemned the use of salt.

The best modern meat extracts are prepared from meat freed from bone and superfluous fat by treatment with hot or boiling water, the time and temperature of extraction varying greatly with the different processes. While in Argentina, in former times when cattle were plentiful, meat extract was the main product and the extracted residue was considered of little value, at the present time, at least in the United States, the extract is commonly a by-product obtained by evaporating the liquor in which meat has been cooked for canning. The concentration of the liquor is carried on in vacuum kettles to a water content of about 50% for liquid extracts or 18 to 25% for solid or pasty extracts. As corned beef is the most popular canned meat, the liquor in which it is cooked is the chief source of supply. It contains a considerable amount of salt as well as a little saltpeter and sugar, the salt according to Richardson \* being removed in sufficient amount by concentrating and centrifuging to comply with the standards as given on page 252. While in certain cases salt is a willful addition, under conditions now existing in the United States, it is more apt to be an impurity which the manufacturer is concerned in removing.

Meat extracts are commonly packed in glass or earthenware jars. The use of tin containers has been found objectionable because of the blackening of the cans due, according to Beveridge,† to tin sulphide, iron sulphide, and iron oleate.

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\* Allen's Commercial Organic Analysis, Phila., 1914, 8, p. 396.

† Third Rep. Com. Physiol. Effects of Food, Training and Clothing on the Soldier, London, 1908, 73.



*Constituents.*—The chief constituents are coagulable proteins belonging to the globulin and albumin groups, proteoses (albumoses), meat bases, phosphates, and chlorides. Small amounts of lactic acid, inosite, and other minor constituents of meat soluble in hot water are also present. True peptones are usually either not present or else the test is obscured by interfering substances. According to Micko\* although gelatin in small amounts is present in the liquor from which meat extract is prepared, the finished product does not contain gelatin as such, but rather in the form of acid-glutin or gelatose which respond to the biuret test like gelatin, but do not form a jelly. This change is due to the action of lactic acid during concentration. Adam† reports formic acid in all the samples of extracts and related products examined. He states it is formed by the action of nitric acid on starch used in the process of manufacture.

By far the most important constituents from the physiological standpoint are the meat bases to which the preparations owe their well-known stimulating properties. Indeed, a properly prepared extract has very little actual food value, but is rather to be regarded as a stimulant and condiment serving both purposes in an analogous manner to tea and coffee.

*Creatine and Creatinine*, aside from their value as stimulants, are of importance, as was first pointed out by Micko, in distinguishing true meat extracts from yeast extracts, which formerly, if not at the present time, were used as substitutes. These are usually determined together and the results expressed in terms of creatinine ("total creatinine") after dehydrolyzing the creatine with acid.

*Carnosine, Carnitine, and Methyl Guanidine*, according to Krimberg, occur in meat extracts as well as in the living muscle.

*The Purine Bases* of meat extracts have been exhaustively studied by Micko.‡ who found hypoxanthine in the largest amount while xanthine and adenine were present in smaller amounts. He was unable to find either guanine, which according to Kossel is present in meat, or carnine which Weidel§ reported in American meat extract. The former Micko considers to have been eliminated in the manufacture of the extract while the latter he believes not to be present in either. Carnine and hypo-

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\* Zeits. Unters. Nahr. Genussm., 14, 1907, p. 284.

† Arch. Chem. Mikros., 9, 1916, p. 77.

‡ Zeits. Unters. Nahr. Genussm., 6, 1903, p. 781; 8, 1904, p. 225.

§ Ann. Chem. Pharm., 158, p. 353.



xanthine are very similar in their reactions and the latter might easily be mistaken for the former although there could be no question of identity if nitrogen were determined, as carnine contains 28.57% and hypoxanthine 41.18%. In yeast extracts Micko found adenine as the chief purine base; guanine, hypoxanthine, and xanthine were also present, the quantities being in the order named, while carnine was not found.

*Products of Hydrolysis.*—By hydrolyzing meat extracts, according to Fischer's method, Micko\* obtained glutaminic acid, alanine, leucine, isoleucine, aspartic acid, and glycol. In addition other amino acids were obtained but in too small quantities for identification. Hydrolysis of the precipitate obtained by salting out with ammonium sulphate showed that it consisted almost entirely of true proteoses and did not contain gelatin. The filtrate from the proteoses yielded on hydrolysis amino acids of which glutaminic acid and glycol were the most abundant while alanine, leucine, and aspartic acid occurred in smaller amounts. Proline and phenyl-alanine were not found. Taurine not previously reported was isolated and identified.

*Chlorine* in meat extract is usually calculated in terms of sodium chloride, which Richardson † points out is not scientifically accurate, since the chlorine derived from the meat exists chiefly if not wholly as potassium chloride. He considers that after allowing 0.06% of sodium chloride for every unit per cent of dry solid matter present any excess may be fairly considered as added salt.

*Analyses* of both solid and liquid meat extracts by Micko, ‡ Bigelow and Cook, § Street, || and Wright, ¶ appear on pages 246 to 249. In a number of instances the results have been recalculated or rearranged to facilitate comparison.

**Meat Juices.**—A true meat juice, as prepared by expressing the liquid portion of meat, is a food product of high nutritive value and differs markedly in this respect from liquid extract and similar preparations on the market, some of which have been sold with misleading claims. While it is impracticable to concentrate a meat juice without precipitation or

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\* Zeits. Unters. Nahr. Genussm., 5, 1902, p. 193.

† Allen's Commercial Organic Analysis, Phila., 1914, 8, p. 394.

‡ Zeits. Unters. Nahr. Genussm., 5, 1902, p. 193; 20, 1910, p. 537; 26, 1913, p. 321.

§ U. S. Dept. of Agric., Bur. of Chem., Bul. 114.

|| Conn. Agric. Exp. Sta., Rep. 1907-8, p. 606.

¶ Jour. Soc. Chem. Ind., 31, 1912, p. 176.



alteration, the name meat juice is perhaps warranted in the case of some of the preparations now on the market, which unlike liquid extracts give strong tests for hemoglobin and contain considerable amounts of coagulable nitrogen. In view, however, of the difficulties of accurate classification, juices and fluid extracts are included under the same head in the tables on pages 246 to 248.

**Peptones and Meat Seasonings.**—Certain meat preparations, known in Germany as "*Speisewürzen*," are made by digesting in various ways meat, or meat residues after extracting the soluble constituents, so as to obtain the constituents in a "predigested" condition and develop agreeable flavors. These preparations are generally known as "peptones" in English, although this term is in many, if not all, cases inappropriate in view of their composition. Etienne and Delhayé obtained an English patent in 1890 for preparing a "peptone" from meat by heating the pulp in an autoclave from 150 to 175° C., separating the liquid from the insoluble matter, and digesting the latter with hydrochloric acid until the fibers are destroyed. The liquid obtained by the acid treatment, after neutralizing with sodium carbonate, is added to the concentrated aqueous extract. Micko,\* in following this process, obtained by the acid treatment of the extracted meat a product with little odor but a decided meaty flavor which appeared to be due to amino acids. He obtained similarly flavored products by the hydrolysis of casein and silk fibroin. Examination of commercial preparations, made by the hydrolysis of protein matter, showed that they contained no coagulable or insoluble proteins, little if any proteoses or peptones, but often considerable amounts of ammonia. The amount of phosphoric acid present depended on the protein material employed but compared with that present in true meat extract was usually greater. Peptones are commonly used in soup preparations, often in conjunction with true meat extract, and various vegetable extracts for flavors.

Soy sauce, the characteristic seasoning of chop suey, although a vegetable product rich in carbohydrates, resembles in its other constituents seasonings prepared from meat. Like the peptones, it contains only a trace of purine bases and little or no total creatinine. Suzuki, Aso, and Mitarai † separated from two liters of this sauce the following constituents in the quantities (grams) named: alanine 6.6, leucine 6, proline 3, lysine 2.6,

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\* Zeits. Unters. Nahr. Genussm., 26, 1913, p. 322.

† Bul. Col. Agr. Tokyo. Imp. Univ., 7, 1907, p. 477.



COMPOSITION OF MEAT EXTRACTS AND SIMILAR PRODUCTS (MICKO).

Brand.	MINERAL CONSTITUENTS.			NITROGEN.										Hemoglobin.	Ether Extract.	Sugars.	Starch, Dextrin, and Glycogen.	Glycerin.
	Water.	Total Ash.	Sodium Chloride Equiv. to Chlorine.	Phosphoric Acid.	Total.	Insoluble.	Coagulable.	Protease.	Peptone.	Creatine.	Creatinine.	Purine Base.	Ammonia.					
<b>Solid Meat Extracts:</b>																		
Liebig's.....	17.44	22.10	2.98	7.93	9.27	.....	0.00	1.63	tr.	.....	.....	0.65	0.39	.....	.....	.....	*	
Toril.....	28.20	28.15	16.73	4.50	6.58	0.03	0.00	1.94	tr.	.....	.....	0.39	0.22	.....	.....	.....	*	
Bovos.....	28.65	28.92	15.45	4.76	4.84	.....	0.00	0.61	tr.	.....	.....	0.89	0.24	.....	.....	.....	*	
Luos.....	26.52	26.32	8.57	5.82	7.05	0.06	0.00	0.18	pos.	.....	.....	0.51	0.01	.....	.....	.....	*	
<b>Fluid Meat Extracts:</b>																		
Bovos.....	61.67	17.51	11.71	2.44	2.27	.....	0.00	0.28	tr.	.....	.....	.....	0.12	.....	.....	.....		
Maggi's Soup Seasoning.....	56.93	22.11	18.77	1.11	3.10	.....	0.00	0.07	tr.	.....	.....	0.01	0.67	.....	.....	.....		
Brand's Meat Juice.....	77.25	9.39	5.76	1.28	1.83	.....	0.25	0.12	.....	0.20	0.25	.....	.....	pos.	.....	0.00	.....	pos.
Valentine's Meat Juice.....	65.09	11.49	1.52	3.89	3.14	.....	0.01	0.07	.....	0.22	0.21	.....	.....	?	.....	0.00	.....	pos.
Wyeth's Meat Juice.....	59.56	16.49	7.84	3.26	3.02	0.02	0.54	0.06	.....	0.17	0.18	.....	.....	pos.	.....	0.00	.....	pos.
Pick's Karsan.....	52.36	13.29	6.04	2.22	4.63	0.53	0.62	1.05	0.00	0.45	0.33	.....	0.18	pos.	.....	0.00	.....	pos.
<b>Miscellaneous:</b>																		
Carnine Lefranco.....	38.24	0.92	0.05	0.30	0.98	0.03	0.56	0.01	.....	0.06	0.03	.....	.....	pos.	.....	44.29†	.....	pos.
Armour's Fluid Beef, Seasoned†.....	70.46	16.75	13.40	0.76	1.45	0.07	0.00	0.32	0.00	0.10	0.11	.....	0.06	0.00	0.86	0.00	1.04	0.00
Bovril§.....	42.24	17.55	9.95	2.87	6.28	0.96	0.00	1.67	tr.	0.53	0.40	.....	0.21	0.00	.....	0.00	.....	0.00
Brunnengraber's Carvis.....	89.89	2.46	1.52	0.37	1.25	.....	tr.	0.51	pos.	0.12	.....	.....	.....	0.00	.....	0.00	.....	0.00
Vir.....	76.60	14.70	12.67	0.69	1.22	.....	0.00	0.24	tr.	.....	.....	0.09	0.06	.....	.....	.....	.....	0.00
Soup Seasoning.....	15.82	42.12	38.66	0.61	6.04	.....	.....	0.04	.....	0.06	0.06	0.02	0.98	.....	.....	.....	.....	0.00
Graf's Soup Seasoning.....	51.56	16.97	16.24	.....	4.84	.....	.....	.....	.....	.....	0.02	0.02	1.09	.....	.....	.....	.....	0.00
Karna Soup Extract.....	10.32	54.44	50.81	1.64	3.17	.....	.....	.....	.....	.....	.....	0.29	0.43	.....	10.33	.....	.....	0.00
Karna Goulash Seasoning  .....	13.60	27.18	23.96	1.46	2.14	.....	.....	.....	.....	.....	.....	0.17	.....	.....	16.21	.....	.....	0.00
Seasoning from Meat Proteins*.....	64.51	21.66	21.28	.....	1.99	.....	.....	.....	.....	.....	.....	.....	0.22	.....	.....	.....	.....	0.00
Seasoning from Casein†.....	63.25	22.40	21.22	.....	2.00	.....	.....	.....	.....	.....	.....	tr.	0.25	.....	9.39	.....	.....	0.00
Ochsen.....	12.95	49.92	45.24	0.60	3.17	.....	.....	0.07	.....	.....	.....	.....	0.25	.....	.....	6.71	.....	0.00
Soy Sauce.....	69.57	15.08	14.01	0.51	1.27	.....	.....	0.16	.....	.....	.....	0.01	0.11	.....	.....	.....	.....	0.00
<b>Bouillon Cubes:</b>																		
I.....	3.05	63.28	58.09	1.71	3.85	.....	.....	1.34	.....	0.53	0.17	0.13	0.13	.....	7.22	.....	.....	0.00
II.....	2.94	68.73	64.71	1.21	2.96	.....	.....	0.45	.....	0.32	0.10	0.24	0.28	.....	9.28	.....	.....	0.00
III.....	7.25	69.87	67.45	0.43	2.40	.....	.....	0.10	.....	0.11	0.04	0.36	0.84	.....	4.85	.....	.....	0.00
IV.....	4.28	72.34	68.33	0.98	2.06	.....	.....	0.26	.....	0.31	0.08	0.16	0.16	.....	8.76	.....	.....	0.00

\* Glycogen present. † Sucrose 38.91; invert sugar 5.38. ‡ Microscopic examination: tomatoes, capsicum, pepper. § Microscopic examination: meat fibers, pepper, capsicum. || Microscopic examination: wheat flour, onion, paprika. ¶ Made in the laboratory.



COMPOSITION OF MEAT EXTRACTS AND SIMILAR PRODUCTS (BIGELOW AND COOK).

Brand.	MINERAL CONSTITUENTS.						ACIDITY.		NITROGEN.								Ether Extract.	Undetermined.
	Total Ash.	Sodium Chloride Equiv. to Chlorine.	Total Phosphoric Acid.	Organic Phosphoric Acid.	Inorganic Phosphoric Acid.	N/10 Sodium Hydroxide, cc. per gm.	As Lactic Acid.	Total.	Insoluble and Coagulable.	Proteose.	"Peptone" (see p. 254).	Total Meat Base.	Creatine and Creatinine.	Purine Base.	Other Meat Base.	Ammonia.		
Solid Meat Extracts:																		
Cudahy's Rex.....	26.50	24.06	8.54	2.29	0.35	1.94	6.67	7.30	0.32	1.65	1.57	3.56	0.87	0.38	2.31	0.20	1.30	8.66
Liebig's.....	21.14	21.03	3.11	2.40	0.61	1.79	9.04	9.07	0.19	2.01	2.08	3.82	1.14	0.33	2.65	0.37	0.94	5.89
Armour's.....	21.66	20.46	5.47	4.55	0.49	4.06	9.35	7.66	0.48	2.02	1.90	3.05	0.75	0.04	2.26	0.21	0.50	11.67
Libby's Premier.....	21.86	30.92	18.32	2.53	0.24	2.29	5.72	6.02	0.20	0.77	1.33	3.20	1.01	0.11	2.08	0.43	0.53	16.11
Swift's.....	20.16	27.28	13.51	2.89	0.18	2.71	4.61	6.60	0.35	1.62	1.09	3.43	0.81	0.45	2.17	0.71	0.43	21.04
Hammond's Coin Special.....	12.39	31.68	13.25	3.19	0.21	2.98	7.16	6.86	0.06	0.86	1.48	4.21	1.24	0.52	2.45	0.25	0.43	20.61
Fluid Meat Extracts:																		
Armour's Conc.....	57.75	17.23	8.27	2.32	0.26	2.06	3.46	2.85	0.04	0.34	0.70	1.66	0.38	0.23	1.05	0.11	0.09	9.75
Wyeth's Beef Juice.....	58.84	16.21	6.71	3.27	0.43	3.23	3.92	3.15	*0.46	0.10	0.47	1.92	0.26	0.26	1.40	0.20	0.23	8.12
Valentine's Meat Juice.....	57.64	10.26	1.77	3.41	0.45	2.96	5.04	3.06	0.03	0.10	0.77	1.94	0.35	0.22	1.37	0.22	0.50	15.12
Armour's Vigor.....	49.94	15.91	7.02	3.29	0.46	2.83	5.29	3.87	*0.29	0.69	0.74	2.02	0.48	0.17	1.37	0.13	0.04	12.14
Cudahy's Rex.....	55.99	16.99	8.48	2.48	0.38	2.10	5.51	3.95	*0.17	0.54	0.41	2.63	0.80	0.04	1.79	0.20	0.05	6.60
Cibil's.....	64.63	16.13	11.38	0.95	0.14	0.81	2.70	3.18	*0.31	0.44	0.89	1.36	0.50	0.77	0.09	0.18	0.06	2.04
Mosquera Jelly.....	68.97	13.85	10.05	0.80	0.18	0.62	2.45	2.41	*0.08	0.31	0.91	0.98	0.26	0.08	0.64	0.13	0.09	3.54
Miscellaneous:																		
Royal Bouillon Capsules.....	14.75	39.75	20.72	2.50	.....	.....	6.44	5.93	0.33	2.04	1.18	2.22	0.46	0.13	1.63	0.16	0.73	9.66
Bovril Seasoned.....	43.30	16.00	8.73	2.26	0.25	2.01	4.30	5.62	*1.21	1.68	0.64	1.93	0.51	0.16	1.26	0.16	0.56	7.82
Beef Jelly, Mosquera.....	27.82	17.31	8.39	2.80	0.34	2.46	8.37	7.83	0.19	1.96	2.43	2.96	0.58	0.05	2.33	0.29	0.15	8.97
Brand & Co.'s Essence of Beef.....	90.93	1.34	0.09	0.48	0.11	0.37	9.98	1.28	0.03	0.60	0.18	0.43	0.11	0.01	0.31	0.04	0.51	12
Mulford's Predigested Beef.....	91.69	0.18	0.01	0.04	0.01	0.03	1.06	0.42	0.01	0.03	0.15	0.22	0.02	0.01	0.19	0.01	1.11	4.17
Armour's Soluble Beef.....	30.15	14.55	5.21	2.39	0.47	1.92	6.07	8.41	.....	3.15	2.38	2.14	0.42	0.22	1.50	0.23	0.32	4.80
Bovox Essence of Beef.....	65.77	17.29	9.73	0.88	0.25	0.63	3.23	3.71	0.63	1.15	1.47	0.89	0.05	0.01	0.83	0.17	0.12	5.05
Johnson's Fluid Beef (Bovril).....	47.22	9.80	4.37	0.59	0.19	0.40	5.40	5.77	*0.21	1.72	2.15	1.24	0.20	0.18	0.86	0.25	0.48	1.72
American Extract of Beef.....	27.54	34.73	24.73	2.53	1.10	1.43	5.56	5.91	*0.20	1.47	2.51	1.15	.....	0.03	1.12	0.21	0.62	0.65
Bovine Concentrated Beef.....	80.40	1.55	1.05	0.09	0.04	0.03	1.36	2.61	*0.11	1.12	0.69	0.57	0.17	0.04	0.36	0.12	0.12	0.05
London Ess. of Mutton.....	82.03	2.25	0.18	0.82	0.08	0.74	1.80	2.61	*0.11	1.12	0.69	0.57	0.17	0.04	0.36	0.12	0.12	0.05
Murdoch's Liquid Food.....	86.00	0.65	0.20	0.10	0.03	0.07	1.35	1.84	*0.31	0.05	1.35	0.08	0.00	0.03	0.05	0.05	0.57	0.48
Maggi's Bouillon.....	50.56	21.94	17.53	1.04	0.06	0.98	4.50	2.76	0.02	0.15	0.17	1.87	0.07	0.04	1.70	0.55	0.12	8.05
Rose's Peptonized Beef.....	45.13	3.52	1.63	0.43	0.04	0.39	2.31	0.98	*0.22	0.83	2.50	3.17	0.07	0.10	3.00	0.26	1.11	15.75
Armour's Beef Ext. and Veg. Tab.....	22.20	23.66	18.14	1.82	.....	.....	5.29	4.10	*1.69	0.69	0.64	1.01	0.15	0.16	0.70	0.07	0.43	26.76
Leube-Rosenthal's Beef Solution.....	72.68	3.91	1.84	0.60	0.09	0.51	2.82	3.13	*1.58	0.49	0.51	0.43	0.08	0.01	0.34	0.12	2.48	0.77
American Malted Meat Beef Extract.....	8.61	7.87	3.48	0.75	0.20	0.56	0.93	2.02	*1.23	0.24	0.10	0.45	tr.	0.02	0.43	0.00	2.97	68.49
Arlington Beef Peptinoids.....	5.72	5.63	0.40	1.43	0.42	1.01	0.39	4.12	*3.23	0.50	0.00	0.39	tr.	0.04	0.35	0.00	1.95	61.81

\*All coagulable. † Largely insoluble. ‡ Largely insoluble. § Beef, mutton and fruits. || Largely coagulable.



COMPOSITION OF MEAT EXTRACTS AND SIMILAR PRODUCTS (STREET).

Brand.	MINERAL CONSTITUENTS.					ACIDITY CC. N/10 KOH PER GRAM.		FORMS OF NITROGEN.												
	Water.	Total Ash.	Sodium Chloride Equiv.	Phosphoric Acid.	Potash.	Phenol-phthalein.	Litmus.	Total.	Insoluble.	Coagulable.	Zinc Sulphate Ppt.	Tannin-salt Ppt.	Meat Base (by Diff.).	Creatinine.	Creatinine.	Purine Base.	Other Meat Base.	Ammonia.	Petroleum Ether Extract.	
Solid Meat Extracts:																				
Premium.....	10.02	20.26	17.42	1.74	9.75	11.0	5.5	9.25	0.11	.....	1.55	3.72	2.13	0.35	0.45	0.38	0.95	0.29	0.05	
Armour's.....	19.06	21.77	6.62	5.27	8.30	11.8	5.4	8.07	0.16	0.13	2.10	4.27	3.21	0.49	0.36	0.62	1.74	0.30	0.08	
Armour's Sol. Beef.....	17.95	14.45	4.13	3.44	4.08	8.4	5.0	10.47	0.08	0.15	5.04	7.89	3.00	0.10	0.22	0.44	1.24	0.35	0.09	
Cudahy's Rex.....	16.08	27.96	11.50	3.47	8.02	9.3	4.8	8.19	0.02	0.11	1.80	3.97	3.66	0.62	0.64	0.53	1.87	0.43	0.08	
Ingram's Michigan.....	18.21	28.61	15.89	2.43	5.24	7.1	3.8	7.36	0.17	0.07	1.96	4.61	1.31	0.07	0.46	0.48	0.30	0.47	0.12	
Liebig's.....	20.49	17.57	3.17	5.01	7.59	12.7	8.4	9.41	0.09	.....	2.36	4.51	4.44	1.30	0.86	0.76	1.52	0.37	0.17	
Mosquera Jelly.....	24.88	24.35	13.61	3.59	5.31	8.0	3.2	7.72	.....	0.24	2.30	4.70	2.20	0.38	0.24	0.38	1.29	0.40	0.04	
Marshall's (Liebig).....	20.15	30.34	18.03	2.44	6.21	8.2	4.0	5.94	0.11	0.11	1.32	3.10	2.41	0.16	0.42	0.40	1.43	0.21	0.05	
Marshall's (Orig.).....	18.54	32.14	19.24	1.55	6.15	7.9	5.0	5.68	0.22	.....	1.27	2.93	2.34	0.41	0.56	0.43	0.94	0.19	0.05	
Southwick.....	30.37	28.25	22.47	1.15	3.20	5.5	4.8	5.54	0.34	.....	3.16	3.94	1.04	0.06	0.15	0.38	0.45	0.22	0.44	
Swift's.....	22.81	25.44	12.71	3.95	7.23	12.5	7.4	7.14	0.10	.....	1.72	3.16	3.14	0.87	0.46	0.64	1.17	0.74	0.08	
Fluid Meat Extracts:																				
Asparox †.....	68.37	14.51	10.07	0.96	2.27	2.4	1.6	1.78	0.10	0.01	0.38	50.64	0.92	0.13	0.16	0.14	0.49	0.11	0.02	
Armour's †.....	42.03	21.56	10.43	2.72	5.46	6.1	4.2	4.01	0.00	0.10	0.45	1.50	1.99	0.48	0.43	0.36	0.72	0.42	0.00	
Armour's Vigoral †.....	43.15	15.71	5.84	2.85	5.34	6.6	3.8	4.09	0.26	0.07	0.55	1.53	1.07	0.15	0.46	0.31	1.05	0.26	0.19	
Bovox.....	51.74	18.01	14.07	0.88	1.70	3.0	1.2	4.02	0.06	0.00	1.58	2.80	1.00	0.10	0.19	0.07	0.04	0.16	0.29	
Bovril.....	43.12	15.81	7.89	2.48	3.55	5.2	3.1	5.36	0.09	0.04	1.33	2.33	1.55	0.30	0.49	0.35	0.41	0.45	0.62	
Cibil's.....	64.21	15.84	12.56	1.38	1.98	3.0	2.0	2.79	0.14	0.00	0.60	1.40	1.07	0.18	0.28	0.17	0.44	0.18	0.00	
Cudahy's Rex.....	51.53	15.18	4.93	2.87	4.81	5.8	3.8	4.48	0.10	0.06	0.66	1.83	2.30	0.58	0.31	0.34	1.07	0.10	0.05	
Maggi's Bouillon.....	49.34	21.34	18.88	1.01	1.64	4.3	2.3	2.88	0.00	0.00	0.33	0.82	1.88	0.01	0.05	0.06	1.76	0.18	0.00	
Morris.....	51.80	15.81	9.83	1.78	3.36	3.7	2.2	4.04	0.13	0.00	0.81	1.02	1.48	0.00	0.00	0.15	1.33	0.42	0.13	
Mosquera Jelly †.....	64.99	11.28	8.22	0.99	1.73	3.4	1.8	2.90	0.00	0.06	0.31	1.41	1.30	0.07	0.19	0.12	0.92	0.13	0.10	
Sinnox.....	57.33	15.91	11.72	1.36	2.37	4.7	3.3	2.28	0.00	0.00	0.57	0.83	0.87	0.04	0.29	0.18	0.36	0.58	0.00	
Sinnox for Invalids.....	51.57	18.92	12.46	2.18	3.48	5.9	3.6	2.70	0.00	0.05	0.31	0.95	1.42	0.22	0.42	0.26	0.52	0.28	0.16	
Valentine's Meat Juice.....	55.72	11.53	1.49	2.67	5.18	6.0	4.3	3.13	0.05	0.00	0.12	0.00	1.92	0.13	0.22	0.37	1.20	0.26	0.10	
Wyeth's Beef Juice.....	53.81	17.17	7.84	3.06	4.48	6.6	4.7	3.25	0.12	0.41	0.20	0.85	1.08	0.11	0.18	0.16	1.23	0.19	0.02	
Miscellaneous:																				
Bovine.....	68.21	1.61	1.22	tr.	0.18	0.4	0.3	2.57	0.06	0.14	2.39	2.26	0.07	0.00	0.00	0.03	0.04	0.04	0.09	
Mason's Beef Ess.....	88.18	1.36	0.23	0.42	0.62	1.0	0.4	1.30	0.00	0.05	0.77	0.85	0.34	0.05	0.08	0.05	0.16	0.06	0.09	
Beef Peptonoids.....	5.06	5.25	1.17	0.02	1.16	1.2	0.8	4.07	1.42	1.83	0.58	50.68	0.08	0.00	0.00	0.03	0.05	0.06	3.29	
Somatox.....	11.19	6.09	0.03	1.47	0.13	1.7	1.5	12.64	0.00	0.05	10.60	12.19	0.26	tr.	0.00	0.04	0.22	0.14	0.09	
Mosquera Beef Meal.....	12.58	5.19	0.99	1.45	2.24	6.1	2.3	12.25	6.73	0.00	0.63	4.15	1.11	0.11	0.17	0.04	0.79	0.26	8.56	
Armour's Beef Ext. and Vegetable Tab.....	10.21	29.57	22.42	2.24	3.73	5.5	3.7	4.43	1.63	0.14	0.52	1.85	0.68	0.07	0.13	0.40	0.08	0.13	0.36	

\* Traces of nitrates. † Nitrate nitrogen, 0.73. ‡ Contains salicylic acid. § Biuret reaction for peptones in zinc sulphate filtrate.



COMPOSITION OF MEAT EXTRACTS (WRIGHT).

	MINERAL CONSTITUENTS.				NITROGEN											
	Water.	Total Ash.	Sodium Chloride Equiv. to Chlorine.	Phosphoric Acid.	Potash.	Acidity as Lactic Acid.	Total	Insoluble.	Coagulable.	Proteose.	Peptone-like and Polypeptide.	Creatine and Creatinine.	Purine Base.	Other Meat Base.	Ammonia.	Ether Extract.
No. 1.....	14.49	19.01	4.16	6.24	7.60	12.60	8.87	0.20	0.11	2.48	0.86	2.11	0.40	1.97	0.74	0.40
No. 2.....	15.34	19.51	5.07	5.29	7.80	11.10	8.12	0.20	0.10	2.30	1.00	1.25	0.15	2.58	0.54	0.42
No. 3.....	16.95	16.57	3.09	5.54	6.76	11.20	8.47	0.18	0.06	1.81	1.57	1.77	0.28	2.11	0.67	0.33
No. 4.....	17.54	20.03	4.47	4.60	8.32	11.00	8.31	0.18	0.07	1.62	1.67	1.73	0.33	2.26	0.43	0.45
No. 5.....	17.39	21.30	4.42	5.24	8.14	10.90	7.88	0.16	0.07	1.89	1.71	1.33	0.40	1.84	0.52	0.41
No. 6.....	15.71	21.53	5.03	5.58	7.92	11.60	8.05	0.16	0.07	1.84	1.76	1.27	0.60	1.73	0.62	0.35



ammonia 4.2, protein 5.4, formic acid 0.1, acetic acid 0.4, lactic acid 3.2, and two new bases 1 and 0.2. Tyrosin, aspartic acid, substances resembling polypeptides, and cystin were also found. A process has been patented in Germany for preparing a similar seasoning from peas.

*Analyses.*—Under the head of "Miscellaneous" are included in the tables on pages 246 to 248, especially on page 246, a number of preparations known as soup seasoning, etc., which were doubtless obtained in large part by the hydrolysis of meat residues or other protein substances. They are characterized by their low content of purine bases and total creatinine.

**Bouillon Cubes.**—These are about the size of large dice. They are wrapped with tin foil and for the convenience of travelers are packed in metal boxes. They consist of various mixtures of meat extract, peptones, extracts of soup vegetables, salt, spices, and gelatin or some other stiffening material. A single cube mixed with boiling water suffices for a cup of bouillon which, although containing a very small amount of food material, furnishes a most agreeable concomitant for bread or dry biscuit and also a safe and palatable beverage when the character of the water is questionable. The chief value of analyses is not to show their nutritive value, which is obviously small, but from the percentages of total creatinine and purine bases to gain an insight into the proportion of meat extract present. The percentage of meat extract having been determined by analysis, dependence must then be placed on the palate to determine the quality of the product.

*Analyses* by Micko of bouillon cubes appear on page 246; those by Cook \* on page 251 are of cubes bought in New York in 1912.

Kappeler and Gottfried † in analyses of 35 samples obtained the following maxima and minima: water 10.0–1.1, protein 26.9–0.4, phosphoric acid 1.43–0.25, ash 83.7–56.8, salt 83.3–53.7, total creatinine 1.2–0, fat 9.6–0, and sugar 14.7–0.

*Standards.*—Micko, ‡ after discussing the widely differing standards proposed by Sudendorf, § Geret, || Serger, ¶ and Lebbin, \*\* suggests a minimum of 15% fat-free organic matter (with 15% total nitrogen) of which

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\* Jour. Ind. Eng. Chem., 5, 1913, p. 989.

† Zeits. Unters. Nahr. Genussm., 31, 1916, p. 1.

‡ Loc. cit.

§ Zeits. Unters. Nahr. Genussm., 23, 1912, p. 577.

|| Ibid., 24, 1912, p. 570; Kons. Ztg., 14, 1913, p. 5.

¶ Kons. Ztg., 13, 1912, p. 378.

\*\* Ibid., 14, 1913, pp. 1, 65.



## COMPOSITION OF BOUILLON CUBES.

Brand.	Solids.	Organic Matter.	Ether Extract.	Ash.	Sodium Chloride Equiv. to Chlorine.	Phosphoric Acid.	Acidity cc. N/20 NaOH per Gram.	Total Nitrogen.	Nitrogen pptd. by Alcohol from HCl Sol.	Total Creatinine.
Behrend.....	96.60	22.86	1.93	73.74	72.13	1.02	6.20	2.19	0.13	0.84
Oxo.....	95.06	25.31	3.10	69.75	65.00	1.51	6.50	2.97	0.86	1.07
Steero.....	96.05	28.41	1.20	67.64	62.15	1.83	9.15	3.62	0.76	1.67
Burnham....	96.87	41.94	1.00	54.93	52.90	0.58	6.10	2.11	0.05	0.88
Sunbeam....	95.73	45.23	1.44	50.50	49.26	0.54	7.30	2.36	0.02	0.92
Armour.....	96.05	26.48	0.96	69.57	67.44	0.62	6.00	2.79	0.17	1.07
Morris.....	96.77	33.00	3.79	63.77	59.17	1.69	9.68	3.67	0.56	1.07
Standard....	95.81	21.76	4.19	74.05	72.22	0.48	5.01	2.09	0.07	0.50
Liggitt.....	96.00	21.91	4.58	74.09	71.98	0.41	4.75	2.11	0.05	0.49
Knorr.....	95.44	26.24	4.57	69.20	65.00	1.55	7.40	3.20	0.91	1.38

half is from meat extract, furthermore that for every 100 parts of organic matter from meat extract there should be present at least 10 parts of total creatinine, 1.1 parts of purine nitrogen, and 11 parts of phosphoric acid ( $P_2O_5$ ). Calculated as percentages of the material as purchased these limits would be as follows: total nitrogen 2.25%, total creatinine 0.75% (nitrogen as creatinine 0.28%), purine nitrogen 0.08%, phosphoric acid 0.82%, and meat extract 12.5-15.0%. The author considers that both meat extract and peptones would contain about the same amount of nitrogen (15%) in the dry matter, but that total creatinine, purine bases, and phosphoric acid would be present in considerable amount only in the meat extract. Yeast extract contains even more purine nitrogen and phosphoric acid than meat extract, but the absence of total creatinine serves for its detection. He further notes that albumoses are much higher in meat extracts than in peptones, while on the other hand ammonia is decidedly lower.

Beythien\* considers that bouillon cubes should contain 15-20% of meat extract (0.6-0.8 gram per 4-gram cube) and a maximum of 65% of salt. Gerlach† places the minimum limit for meat extract at 7.5% (0.3 gram per 4-gram cube) but considers 65% a suitable maximum for salt.

\* Zeits. Unters. Nahr. Genussm., 31, 1916, p. 33.

† Chem. Ztg., 40, 1916, p. 587.



**Miscellaneous Preparations.**—Under the head of "Miscellaneous" in the tables are included a variety of preparations containing various mixtures of meat extracts or other meat derivatives with protein substances from other sources, vegetable matter, spices, and other products. Some are foods for invalids while others are merely condiments. The amounts of meat extract can be roughly estimated from the creatinine content.

**Yeast Extracts.**—The absence of creatine and creatinine and the presence of the purine bases (adenine, guanine, hypoxanthine, and xanthine) in yeast extracts have already been noted under meat extracts. Analyses of three yeast extracts by Micko\* are given in the following table:

COMPOSITION OF YEAST EXTRACTS.

	Water.	MINERAL CONSTITUENTS.			Fat-free Organic Matter.	NITROGEN.			
		Total Ash.	Sodium Chloride Equiv. to Chlorine.	Phosphoric Acid.		Total.	Proteose.	Creatine and Creatinine.	Purine Base.
Sitogen.....	32.50	22.00	.....	6.54	45.50	5.81	.....	0	1.14
Ovos.....	53.67	16.87	10.45	2.79	29.46	2.99	.....	0	0.50
X.....	65.93	15.73	10.85	2.11	18.35	2.36	0.21	0	0.30

The author calls attention to the small amount of proteoses (albumoses) and peptones and expresses an opinion that the nitrogen is probably in large part in the form of nucleoproteins.

**Standards.**—The following standards were adopted by the U. S. Joint Committee in 1907.

1. *Meat Extract* is the product obtained by extracting fresh meat with boiling water, and concentrating the liquid portion by evaporation after the removal of fat, and contains not less than 75% of total solids, of which not over 27% is ash, and not over 12% is sodium chloride (calculated from the total chlorine present), not over 0.6% is fat, and not less than 8% is nitrogen. The nitrogenous compounds contain not less than 40% of meat bases, and not less than 10% of creatine and creatinine.

2. *Fluid meat extract* is identical with meat extract, except that it is concentrated to a lower degree, and contains not more than 75, and not less than 50% of total solids.

\* Zeits. Unters. Nahr. Genussm., 5, 1902, p. 193.



3. *Bone extract* is the product obtained by extracting fresh trimmed bones with boiling water and concentrating the liquid portion by evaporation after removal of fat, and contains not less than 75% of total solids.

4. *Fluid bone extract* is identical with bone extract, except that it is concentrated to a lower degree and contains not more than 75 and not less than 50% of total solids.

5. *Meat juice* is the fluid portion of muscle fiber, obtained by pressure or otherwise, and may be concentrated by evaporation at a temperature below the coagulating point of the soluble proteins. The solids contain not more than 15% of ash, not more than 2.5% of sodium chloride (calculated from the total chlorine present), not more than 4 nor less than 2% of phosphoric acid ( $P_2O_5$ ), and not less than 12% of nitrogen. The nitrogenous bodies contain not less than 35% of coagulable proteins, and not more than 40% of meat bases.

6. *Peptones* are products prepared by the digestion of protein material by means of enzymes or otherwise, and contain not less than 90% of proteoses and peptones.

7. *Gelatin (edible gelatin)* is a purified, dried, inodorous product of the hydrolysis, by treatment with boiling water, of certain tissues, as skin, ligaments, and bones, from sound animals, and contains not more than 2% of ash and not less than 15% of nitrogen.

#### ANALYSIS OF MEAT EXTRACTS, ETC.

**Determination of Water.**—Dry from 2–20 grams of the material, according to the water content, in a flat-bottom dish in a boiling water oven to constant weight. It is well to dissolve pasty preparations in water and to use a sufficient amount of asbestos or sand to absorb the material. If fat is to be determined on the same portion employ a tin, or lead dish or Hoffmeister shell; if ash, a platinum or porcelain dish, omitting sand or asbestos.

**Determination of Ash.**—Proceed as in the case of meats (p. 230).

**Determination of Fat.**—Dry as described for the determination of water with asbestos or sand and extract with anhydrous ether in a continuous extractor. Evaporate off the ether, dry the residue, and weigh as in the case of milk (page 121).

**Determination of Total and Ammoniacal Nitrogen.**—Proceed as directed under meat (p. 225).

**Determination of Insoluble Protein.**—Weigh a quantity of the material corresponding to about 10 grams of dry matter into a 500-cc. graduated



flask. Add cold water, mix well, make up to the mark, and shake at intervals for an hour or until the material appears to have gone into solution, and filter on a dry paper. Determine nitrogen in an aliquot of 50 cc. of the filtrate, thus obtaining the amount soluble in cold water which, subtracted from the total nitrogen, gives the insoluble nitrogen.  $N \times 6.25 = \text{insoluble protein}$ .

**Determination of Coagulable Protein.**—Evaporate 400 cc. of the filtrate obtained in the preceding section, to 100 cc., neutralize, boil, filter, and determine nitrogen in the precipitate as in the Grindley and Emmett method (p. 229).  $N \times 6.25 = \text{coagulable protein (albumin)}$ .

Make the filtrate up to 500 cc. and use aliquots for the determination of (1) proteoses, (2) tannin-salt precipitate, and (3) creatine and creatinine.

**Determination of Proteoses.**—*Bömer Method.\**—To a 50-cc. aliquot of the solution prepared as above, add 1 cc. of sulphuric acid (1 : 4) and finely powdered zinc sulphate in small quantities with stirring until on long standing the precipitate of proteoses separates on the surface of the liquid and a small quantity of undissolved zinc sulphate settles to the bottom. Collect the precipitate on a paper free from nitrogen, wash with cold saturated zinc sulphate solution, keeping the funnel covered with a watch glass during filtration, and determine nitrogen in the precipitate without removal from the paper.  $N \times 6.25 = \text{proteoses (albumoses)}$ .

**Determination of Tannin-Salt Precipitate.**—*Schjerning Method Modified by Bigelow and Cook.†*—Concentrate 100 cc. of the filtrate from the coagulable proteins to 20 cc. or less, transfer to a 100-cc. graduated flask, add 50 cc. of sodium chloride solution (300 grams per liter), and shake thoroughly. Place in an ice box at about 12° C. and after the temperature becomes constant add 30 cc. of 24% tannin solution (also at ice-box temperature), make up to the mark, and shake. Keep in the ice box overnight, filter at ice-box temperature, through a dry paper, and determine nitrogen in 50 cc. of the filtrate. Correct for the nitrogen in the reagent, as found by a blank determination, and calculate the percentage in the material. The nitrogen thus obtained includes that present as meat bases, except about one-quarter of the creatine present in the tannin-salt precipitate, and as ammonia. Bigelow and Cook estimate peptones by adding the percentages of tannin-salt filtrate, insoluble, coagulable

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\* Zeits. anal. Chem., 34, 1895, p. 562.

† Jour. Amer. Chem. Soc., 28, 1906, p. 1496. Schjerning himself (Zeits. anal. Chem., 39, 1900, p. 562) regarded tannic acid as of little value in the separation of the proteins. In his method for total protein nitrogen he uses uranium acetate.



and proteose nitrogen, deducting from the total nitrogen, and multiplying the difference by 6.25, although the figure probably represents peptoids, formed by the action of the hot solution on gelatin, and polypeptides. True peptones are apparently not present as the tannin-salt filtrate seldom gives the biuret reaction. See also page 247.

Bigelow and Cook suggest that a correction be introduced for the creatine after determining the amount present before and after precipitation with tannin-salt reagent. Street believes this correction impracticable owing to the difficulty of removing tannin from the filtrate, the slightest trace of which interferes with the color reaction.

**Determination of Creatine and Creatinine.**—*Folin Method Modified by Benedict and Myers.*\*—This method is based on the color produced by creatinine with picric acid in alkaline solution (Jaffé reaction). Place 50 cc. of the filtrate from the coagulable protein, obtained as described above, in a 100-cc. graduated flask, boil down to about 10 cc., add 10 cc. of normal hydrochloric acid, and heat in an autoclave 30 minutes at 117–119°. Cool, add 10 cc. of normal sodium hydroxide solution, make up to the mark, and shake. Pipette 25 cc. into a 500-cc. graduated flask, add 30 cc. of saturated (1.2%) picric acid solution, and 10 cc. of 10% sodium hydroxide solution, make up to the mark, and shake. Compare in a colorimeter with a half-normal potassium bichromate solution (24.54 grams per liter) set at 8 mm. on the scale, which corresponds to 9.88 mg. of pure creatinine. If the reading of the unknown varies more than 2 or 3 mm. from 8 mm. prepare a new solution, using an aliquot that will give approximately that reading. From the results obtained calculate the creatinine which includes not only the creatinine that exists ready formed in the product, but also that obtained by dehydrolyzing the creatine.

If separate figures for both bases are desired, place an aliquot of the filtrate from the coagulable nitrogen directly in a 500-cc. graduated flask, add 15 cc. of the picric acid solution and 10 cc. of the sodium hydroxide solution, make up to the mark, shake, and compare with the bichromate solution as before. Calculate the percentage of creatinine and subtract from the percentage of total creatinine previously obtained and the difference is the creatine in the material calculated in terms of creatinine. To obtain the nitrogen corresponding to either base or the sum of the two, expressed in each case as creatinine, multiply by 0.372. To convert

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\* Amer. Jour. Phys., 18, 1907, p. 397.



creatine expressed as creatinine into true percentage of creatine multiply by 1.16.

If an autoclave is not at hand, invert by the *Baur and Barschall Method* \* as follows: Evaporate 200 cc. of the filtrate from the coagulable protein to a volume of less than 38 cc., transfer to a graduated cylinder, and make up to 38 cc. with the rinsings. Transfer to a 100-cc. graduated flask, rinsing with 17 cc. of normal hydrochloric acid, and heat 4 hours under a reflux condenser in a boiling water-bath. Neutralize the acid with 17 cc. of normal sodium hydroxide solution, make up to 100 cc., and proceed as above described, using, however, smaller aliquots because of the greater concentration of the solution. Micko obtains practically the same results by the two methods of dehydration.

Sudendorf and Lahrmann † find that in bouillon cubes the presence of yeast extract, caramel, tomato juice, and other substances give misleading colors in the determination of creatinine and therefore lead to wrong conclusions as to the content of meat extract. They eliminate this source of error by preliminary treatment with potassium permanganate, as proposed by Lendrich and Nottbohm, as follows:

Prepare a 10% solution, filter to remove fat, and place 10–20 cc. of the filtrate in a 75-cc. capsule, add 10 cc. of normal hydrochloric acid, and evaporate to dryness on a boiling water-bath. Dissolve the residue in water, neutralize to litmus with N/2 sodium hydroxide solution, rinse into an Erlenmeyer flask, dilute to 75 cc., and run in 1% potassium permanganate solution containing 2.5% sodium chloride, drop by drop with shaking until a red color persists for several minutes. If the solution becomes too thick, dilute with water. Destroy the excess of potassium permanganate by adding drop by drop 3% hydrogen peroxide solution containing 1 cc. of glacial acetic acid per 100 cc. Heat on a boiling water-bath 5–10 minutes until the manganese oxide separates in flocks. Filter with the aid of suction, wash until chlorine is removed, evaporate to small volume, rinse into a 500-cc. flask, using about 20 cc. of water, cool, then treat with 10 cc. of 10% sodium hydroxide solution and 20 cc. of saturated picric acid solution. After 5 minutes make up to the mark and proceed as above described.

**Determination of Purine (Xanthine) Bases.**—*Krüger-Micko Method*. ‡—To 5 grams of the material add 100 cc. of water and 10 cc. of sulphuric

\* Arb. Kaisl. Gsndhtsamt., 24, 1906, p. 552.

† Zeits. Unters. Nahr. Genussm., 29, 1915, p. 1.

‡ Ibid., 5, 1902, pp. 204, 209; 26, 1913, p. 334.



acid (1:3), and boil under a reflux condenser 3 hours. Neutralize with sodium hydroxide solution, add 20 cc. each of copper sulphate solution (130 grams per liter) and saturated sodium bisulphite solution (200 grams per liter), and boil 2-3 minutes. After cooling, filter and wash with water containing a little of both the reagents to prevent oxidation. Remove paper and filter to a flask, mix well with water, and add sufficient hydrochloric acid to dissolve the precipitate. Heat to boiling to reduce the filter to a pulp and remove sulphur dioxide, and precipitate the copper with hydrogen sulphide gas. After some hours filter on a close filter and wash with water containing a little hydrogen sulphide and a few drops of hydrochloric acid.

Evaporate the filtrate to small volume, take up in hot water if necessary, with the addition of a few drops of hydrochloric acid, nearly neutralize with ammonia, dilute to 70 cc., and after cooling add 20 cc. of ammonia water (1:1).

Precipitate with a mixture of 25 cc. of 10% silver nitrate and 25 cc. of 1:1 ammonia water, added drop by drop with stirring. After standing some hours in a dark place, filter, wash three times with 4% ammonia water (1:6), then with water, containing 2.5 cc. of strong ammonia per 100 cc., until free from nitrates. Wash out the ammonia with 70% alcohol, dry in a water oven, and determine nitrogen.

The results are most accurately expressed as percentage of purine nitrogen, since the factors for the different purine bases differ somewhat. Some authors calculate the results in terms of xanthine, using the factor 2.71.

*Other Methods for Separating Meat Nitrogenous Compounds.*—Among the methods which need be but briefly mentioned, as they have been largely displaced in recent years, are those of Liebig (alcohol solubility), Bruylants\* (fractional precipitation by various strengths of alcohol), Hehner† (precipitation by methylated spirits), Rideal and Stewart‡ (precipitation by chlorine), Allen and Searle§ (precipitation by bromine), and various methods based on precipitation by phosphotungstic acid.

**Determination of Acidity.**—Titrate with N/10 alkali a weighed portion of the material dissolved in water, using phenolphthalein or delicate litmus paper as indicator. Calculate as lactic acid.

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\* Jour. Pharm. Chem., 5, 1897, p. 515.

† Analyst, 10, 1885, p. 221.

‡ Ibid., 22, 1897, p. 228.

§ Ibid., 22, 1897, p. 259.



**Determination of Coagulation Point.**—Dilute with water to overcome the influence of coloring matter and other interfering substances. Place in a test tube 2 cm. in diameter and introduce a thermometer through a perforated cork. Heat the test tube in a beaker of water, stirring the solution with the thermometer during the heating, and note the coagulation point.

**Determination of Sugars.**—*W. B. Smith Method.\**—To a solution of 5 grams of the meat extract in 25 cc. of water add an excess (4–6 grams) of solid picric acid and an excess (40–60 cc.) of 20% phosphotungstic acid solution. Mix, make up to 100 cc., shake, and filter through a dry paper. To 60 cc. of the filtrate add 3 cc. of concentrated hydrochloric acid, make up to 66 cc., and filter quickly. Determine copper reducing power of the filtrate both directly and after inversion, neutralizing in both cases. See Chapter XIV.

**Determination of Glycerol.**—This substance is sometimes used as a preservative for fluid preparations. Bigelow and Cook † evaporate to dryness, extract with acetone, remove the meat bases by precipitation with silver nitrate, followed by phosphotungstic acid, and determine the glycerin in the filtrate by Hehner's method.‡

**Detection of Preservatives in Meat Extracts.**—Boric acid is sometimes used as a preservative in these preparations, and is tested for by the usual methods (Chapter XVIII).

### GELATIN.

Gelatin may be regarded as a food adjunct rather than as a food. The food value of preparations containing gelatin is due chiefly to the sugar and other constituents as the amount of gelatin is small and what is present serves chiefly as fuel and not as a substitute for true proteins since a diet containing gelatin as the only nitrogenous constituent will not support life.

The product used for food should be obtained from clean material such as bones, calves' feet, etc. It consists essentially of *glutin* and its derivative *glutose*.

According to the U. S. Standard it should contain at least 15% of nitrogen and not more than 2% of ash.

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\* Jour. Ind. Eng. Chem., 8, 1916, p. 1024.

† U. S. Dept. of Agric., Bur. of Chem., Bul. 114, p. 42.

‡ Jour. Soc. Chem. Ind., 8, 1889, p. 4.



Common impurities are sulphur dioxide and traces of arsenic. Copper, zinc (indicating glue stock preserved with zinc salts), and lead are occasionally present.

In detecting sulphur dioxide, Alexander\* recommends distillation and determination by the gravimetric rather than the iodimetric method.

### FISH.

**General Composition.**—Fish resembles meat both structurally and in the nature of its constituents, but differs from it in the relative proportions of its various components. Thus, there is considerably more refuse matter such as skin and bones in fish than in meat, and in the edible portion of fish the amount of water and ash is usually greater and of fat is less. Comparing the nitrogenous components of each, we find in fish more of the gelatin-yielding matter (collagen) and less of the extractives than in meat. There is much less hæmoglobin or allied coloring substance in the flesh and blood of fish than in meat, which accounts for the white color usually characteristic of the former. Certain fish, however, like the salmon, probably owe their distinctive color to a pigment belonging to the lipochrome class, while the colored areas in salt herring are due, according to Griebel,† to a red pigment, soluble in trimethylamin and similar weak bases, derived from the eyes of marine animals eaten by the fish. The common belief that fish contains markedly more phosphorus and is a better brain food than meat is not in accordance with the facts, although fish roe and milt are rich in phosphates and lecithin as well as in proteins. As regards digestibility, fish is regarded as superior to meat although certain varieties rich in fat and all crustaceans are exceptions to the rule.

According to Atwater and Bryant‡ the composition of different varieties of true or vertebrate fish is as given on page 260.

**Fat Content of Fish.**—Hutchison§ classifies fish as follows with reference to their content of fat: (1) lean, with less than 2% of fat, such as cod and haddock; (2) medium, with 2-5% of fat, such as halibut and mackerel; (3) fat, with more than 5% of fat, such as salmon, eels, turbot, and herring.

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\* Jour. Amer. Chem. Soc., 29, 1907, p. 783.

† Zeits. Unters. Nahr. Genussm., 19, 1910, p. 424.

‡ U. S. Dept. of Agric., Off. of Exp. Sta., Bul. 28, p. 47 *et seq.*

§ Food and the Principles of Dietetics.



## COMPOSITION OF FISH.

		Refuse.	Water.	Protein.		Fat.	Ash.	Fuel Value per Pound.
				N× 6.25.	By Difference.			
Bass—	edible portion.....	.....	77.7	18.6	18.3	2.8	1.2	465
	as purchased.....	55.0	35.1	8.4	8.3	1.1	0.5	200
Bluefish—	edible portion.....	.....	78.5	19.4	19.0	1.2	1.3	410
	as purchased.....	48.6	40.3	10.0	9.8	0.6	0.7	210
Cod—	edible portion.....	.....	82.6	16.5	15.8	0.4	1.2	325
	as purchased.....	52.5	38.7	8.4	8.0	0.2	0.6	165
Eel—	edible portion.....	.....	71.6	18.6	18.3	9.1	1.0	730
	as purchased.....	20.2	57.2	14.8	14.6	7.2	0.8	580
Haddock—	edible portion.....	.....	81.7	17.2	16.8	0.3	1.2	335
	as purchased.....	51.0	40.0	8.4	8.2	0.2	0.6	165
Halibut—	edible portion.....	.....	75.4	18.6	18.4	5.2	1.0	565
	as purchased.....	17.7	61.9	15.3	15.1	4.4	0.9	470
Herring—	edible portion.....	.....	72.5	19.5	18.9	7.1	1.5	660
	as purchased.....	42.6	41.7	11.2	10.9	3.9	0.9	375
Mackerel—	edible portion.....	.....	73.4	18.7	18.3	7.1	1.2	645
	as purchased.....	44.7	40.4	10.2	10.0	4.2	0.7	365
Perch—	edible portion.....	.....	75.7	19.3	19.1	4.0	1.2	530
	as purchased.....	62.5	28.4	7.3	7.2	1.5	0.4	200
Pickrel—	edible portion.....	.....	79.8	18.7	18.6	0.5	1.1	370
	as purchased.....	47.1	42.2	9.9	10.7	0.3	0.6	210
Salmon—	edible portion.....	.....	64.6	22.0	21.2	12.8	1.4	950
	as purchased.....	34.9	40.9	15.3	14.4	8.9	0.9	660
Shad—	edible portion.....	.....	70.6	18.8	18.6	9.5	1.3	750
	as purchased.....	50.1	35.2	9.4	9.2	4.8	0.7	380
Skate—	edible portion.....	.....	82.2	18.2	15.3	1.6	1.1	400
	as purchased.....	51.0	40.2	8.9	7.5	0.7	0.6	195
Smelt—	edible portion.....	.....	79.2	17.6	17.3	1.8	1.7	405
	as purchased.....	41.9	46.1	10.1	10.0	1.0	1.0	230
Trout—	edible portion.....	.....	77.8	19.2	18.9	2.1	1.2	445
	as purchased.....	48.1	40.4	9.9	9.8	1.1	0.6	230
Turbot—	edible portion.....	.....	71.4	14.8	12.9	14.4	1.3	885
	as purchased.....	47.7	37.3	7.7	6.8	7.5	0.7	460
Whitefish—	edible portion.....	.....	69.8	22.9	22.1	6.5	1.6	700
	as purchased.....	53.5	32.5	10.6	10.3	3.0	0.7	325

Clark and Almy\* note considerable variation in the fat of migratory fish such as shad and mackerel, due to season, age, time of spawning, and feeding, but little in bottom fish such as haddock and flounders.

Röhrig† bases his distinction of sardines from French and Brabant anchovies on the differences in fat content, the former containing 7–10%, the latter 0.8–3.10%. This distinction is of no value in the examination of sardines in oil.

Behre and Frerichs‡ employ the percentage of fat and the fat con-

\* Jour. Biol. Chem., 29, 1917, xxii.

† Ber. Chem. Unters. Anst. Leipzig, 1910, p. 13.

‡ Zeits. Unters. Nahr. Genussm., 24, 1912, p. 676.



stants as a means of distinguishing anchovy butter from imitations made from herring and other foreign fish. The following data are given by them:

	Number of Samples.	Water.	Fat.	Iodine Number of Fat.	Refraction of Fat, 40° C.
Butter.....	.....	13.40	83.51	41.38	45
Anchovies...	3	65.07-74.46	1.14-1.36	138.7-176.7	74-75
Herring.....	2	52.40-56.20	10.62-11.76	101.1-109.1	64-66

From the above figures the authors named conclude that anchovy butter with less than 10% of fat probably contains no herring; if the fat content reaches 15% the iodine number should not be over 60 and the refraction over 50.

**Characteristics of Fresh Fish.**—Fish of all kinds should be eaten when perfectly fresh, as it undergoes decomposition much sooner than meat when killed. While with meat aging is often beneficial to bring out requisite tenderness and flavor, in the case of fish deterioration begins almost immediately after death. Even though certain varieties of fish may be kept firm and wholesome for some days on ice, the flavor is distinctly impaired by long keeping. When, however, fish is frozen while perfectly fresh changes are arrested. Smith\* could detect no appreciable change in composition, food value, or sanitary condition in fish frozen for 9 months and Perlzweig and Gies† found none in fish frozen 2 years. Fish that is not perfectly firm to the touch, or that has abnormally dry scales, or that shows blubber at the gills, or that possesses the marked odor that accompanies incipient decomposition, should not be used as food.

**The Roe** of shad, cod, and other fish is eaten fresh and is valuable not only for its protein, fat, and carbohydrates but also for its lecithin and cholesterol. Fish milt is also used for food.

**Caviar** is salted sturgeon roe. The best grades are obtained from southern Russia, inferior grades from the river Elbe. Salted cod roe, a common substitute, is characterized by its lower fat content. From the data available it is difficult to give a minimum limit for fat in caviar. Analyses by Rimini‡ and Grossfeld§ give a minimum fat content of 14.67% for true caviar and a maximum fat of 4.44% for cod roe pre-

\* Biochem. Bul., 3, 1913, p. 54.

† Ibid., p. 69.

‡ Staz., sper. agrar. Ital., 36, 1903, p. 249.

§ König's Chemie der Mensch. Nahrungs- und Genussmittel 4 Aufl. III, 2, p. 159.



pared in a similar manner. Buitenberg's analyses,\* however, show a minimum of 7.59% of fat in true caviar and 1.27% of fat in a single sample of salted cod roe.

**Crustaceans and Mollusks.**—These differ from the meats and common fish in that they contain considerable carbohydrate (glycogen) but no creatine or creatinine. The *lobster* and *crab* are nearly alike in composition, the flesh being made up of coarse, dense, thick-walled fibers.

Payen gives the following composition of the flesh and body of lobster:

	Flesh (contained in Claws and Tail).	Body (consisting mainly of Liver).
Water.....	76.6	84.31
Protein.....	19.17	12.14
Fat.....	1.17	1.14

*Clams* and *Oysters* are low in solid nutriment, and are more digestible when eaten raw than cooked. Oysters contain 3% or more of glycogen, and distinct amounts of copper the physiological role of which is analogous to that of iron in the blood of higher animals.

The following analyses are from Atwater and Bryant's compilation:†

#### COMPOSITION OF SHELL FISH, ETC.

	Refuse.	Water.	Pro- tein. N X 6.25.	Fat.	Car- bohy- drates.	Ash.	Fuel Value per Pound. Cals.
Clams— edible portion.....	....	85.8	8.6	1.0	2.0	2.6	240
as purchased.....	41.9	49.9	5.0	0.6	1.1	1.5	140
Crabs— edible portion.....	....	77.1	16.6	2.0	1.2	3.1	415
as purchased.....	52.4	36.7	7.9	0.9	0.6	1.5	195
Lobster— edible portion.....	....	79.2	16.4	1.8	0.4	2.2	390
as purchased.....	61.7	30.7	5.9	0.7	0.2	0.8	140
Mussels— edible portion.....	....	84.2	8.7	1.1	4.1	1.9	285
as purchased.....	46.7	44.9	4.6	0.6	2.2	1.0	150
Oysters— edible portion.....	....	86.9	6.2	1.2	3.7	2.0	235
as purchased.....	81.4	16.1	1.2	0.2	0.7	0.4	45
Scallops— as purchased.....	....	80.3	14.8	0.1	3.4	1.4	345
Terrapin— edible portion.....	....	74.5	21.2	3.5	....	1.0	545
as purchased.....	75.4	18.3	5.2	0.9	....	0.2	135
Turtle— edible portion.....	....	79.8	19.8	0.5	....	1.2	390
as purchased.....	76.0	19.2	4.7	0.1	....	0.3	90
Frogs' Legs— edible portion.....	....	83.7	15.5	0.2	....	1.0	295
as purchased.....	32.0	56.9	10.5	0.1	....	0.7	200

\* Ber. Hyg. Inst. Hamburg, 1900-2, p. 13.

† U. S. Dept. of Agric., Off. of Exp. Sta., Bul. 28, p. 52.



**Canned Fish.**—Modern methods of canning are particularly valuable in the fish industry owing to the perishable nature of the product and often the remoteness of the supply. With us salmon, tunny, and lobster are the best known varieties preserved by simple canning, while sardines and herring are the most popular fish treated by salting, smoking, or the addition of oil, spices, and various condiments previous to canning.

While the packing of decomposed fish, spoilage due to imperfect sterilization, and the substitution of inferior varieties have not been uncommon, the detection of these irregularities or defects, now fortunately infrequent, falls to the bacteriologist or zoologist; the examination for foreign oils and tin, however, is a frequent duty of the chemist. Olive oil is used in packing the best grade of French and Norwegian sardines, but cotton seed oil is commonly substituted in the preparation of the cheaper American product, the nature of the oil being distinctly stated on the label, thus complying with the requirements of food laws.

*Tin*, in the form of salts dissolved from the can, is still a matter of concern, although packers have made serious effort to reduce the amount by using lacquered cans and paper or veneer linings. In samples of sardines put up in mustard, vinegar, and oil, the Massachusetts Board has found as high as 0.376 gram of tin in a half-pound can, the corrosion of the can being very marked. Crustaceans, because of their content of free amino compounds, act with special avidity on tin. Bigelow and Bacon\* found that monomethylamin in canned shrimps attacks the can as do free amino acids in certain vegetables.

The United States Government, pending further investigation, has allowed 0.300 gram of tin per kilo in canned fish, as well as in canned meats, vegetables, and fruits (F. I. D. 126). The unavoidable amount of tin present, however objectionable, is more than offset by the advantages of the canning system. For the detection of tin see Chapter XXI.

**Salted and Smoked Fish.**—Preservation by drying, salting, or smoking as well as a combination of two or all of these methods, is as ancient as canning is modern. While it is of first importance that the fish is true to name, sound when packed, and kept sound by proper handling, the chemist is chiefly concerned with the examination for colors and preservatives as noted on page 265.

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\* Jour. Ind. Eng. Chem., 3, 1911, p. 832.



**Floating of Shellfish.**—*Oysters* and other shellfish, either in the shell, or, more commonly, after shucking, are often subjected to "floating" or "drinking" in fresh or brackish water or else shipped in direct contact with lumps of ice. Both practices cause the shellfish to greatly increase in size, owing to the absorption of an undue amount of water, and if not labelled "floated" the product is adulterated under the federal law and the laws of certain states.

It is, however, not regarded as improper to drink oysters in water of a saline content equal to that in which they will grow to maturity or to wash the shucked oysters in unpolluted, cold or iced water for the minimum time required for cleaning and chilling. After washing they should be drained and packed for shipment in tight receptacles surrounded by ice but protected from the absorption of the water resulting from the melting of the ice.

Nelson advocates the floating of oysters in clean water with a lower salt content than that of the beds because (1) dirt is eliminated, (2) the volume of flesh is increased, (3) a better color and texture are secured, (4) shrinkage is decreased, and (5) the water content during transportation and storage is retained.

Often shellfish is polluted by growing or floating in impure water, handling under insanitary conditions, or packing in unclean receptacles.

As oysters cannot reach the consumer in satisfactory condition if shipped in their own liquor, the loss of food constituents on draining comes up for consideration. Baylac\* reports in a liter of the liquor about 2 grams of albumin as well as determinable amounts of urea, ammonium salts, and inorganic matter. The amount of organic matter in the liquor from Mediterranean oysters is greater than in that of oysters from the ocean.

**Scallops.**—According to the Maine Experiment Station,† scallops properly handled should contain not less than 20% of dry matter. The following is a summary of analyses of soaked and unsoaked scallops by Sullivan,‡ the swelling of the meats being sufficient in some cases to make 4½ gallons fill a 7-gallon keg:

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\* Comp. rend. soc. biol., 62, 1907, p. 250.

† Offic. Insp., 55, 1913, p. 149.

‡ Amer. Food Jour., 10, 1915, p. 472.



	UNSOAKED (31 SAMPLES).			SOAKED (12 SAMPLES).		
	Max.	Min.	Aver.	Max.	Min.	Aver.
Solids.....	24.17	19.21	22.48	19.64	14.18	16.20
Protein.....	15.69	12.62	14.38	11.80	9.06	10.57
Ash.....	1.81	1.33	1.56	1.17	0.93	1.02

*Clams.*—At the Maine Station it was found that the drained meats of clams, which contained when opened 24.9% of solid matter, took up on soaking over night in salt water sufficient liquid to reduce the percentage of dry matter to 15.3. While it is recognized that clams will not keep in their own liquor, it is insisted that they be rinsed not longer than 1 minute in cold water or else 2 minutes in hot water, followed by 2 minutes in cold water.

**Preservatives in Fish and Oysters.**—Boric acid and borax in mixture and sodium benzoate form the most common preservatives of salt dried fish and of oysters. In the case of salt codfish, the preservative is sprinkled on the surface.\* Such surface application is allowed under the laws of some states, as for example, Massachusetts, and under the federal law, provided directions for the removal of the preservative are given on the package. In opened oysters sold in casks and kegs, boric mixture has been used commonly in solution in the oyster liquor, but is now infrequent. Ishida † calls attention to the presence of a trace of formaldehyde in fresh crab meat and distinct amounts after being preserved 8 months.

**Artificial Colors** of the coal-tar group are used to give smoked fish a rich brown color. The New York City Board of Health has brought to notice the coloring of cheaper fish in imitation of salmon.

**Methods of Analysis.**—These are similar to the methods given for meat.

#### CONCENTRATED FOODS.

Under the name of "condensed" or "concentrated foods" or "emergency rations" a number of canned preparations are sold for the use of campers, travelers, armies in the field, etc. These consist usually of mixtures of dried ground meats and vegetables, pressed together in compact form, and preserved in tin cans. The claims made for the food value of these preparations are, as a rule, extravagant and erroneous, as shown

\* Bitting, U. S. Dept. of Agric., Bur. of Chem., Bul. 133, 1911.

† Jour. Pharm. Soc. Japan, 422, 1917, p. 300.



by Woods and Merrill,\* who give the following analyses of some of these foods:

	Net Weight Con- tents.	Weight of Materials in Package.					Total Fuel Value
		Water.	Pro- teins.	Fat.	Carbo- hy- drates.	Ash.	
	Grams.	Grams.	Grams.	Grams.	Grams.	Grams.	Gals.
Ration cartridge, pea, beef, etc. ....	241	34.2	52.9	42.0	98.0	13.9	1071
Blue ration campaigning food, <i>a</i> .....	169	76.1	37.5	9.0	37.9	8.5	432
" " " " <i>b</i> .....	78	1.0	5.6	23.1	46.9	1.4	436
Red ration campaigning food, <i>a</i> .....	122	33.8	26.2	18.5	37.8	5.7	496
" " " " <i>b</i> .....	77	1.2	5.0	23.0	46.6	1.2	424
Ration cartridge, potatoes, beef, etc. ....	283	117.9	62.3	12.6	76.4	13.8	772
Emergency ration, <i>a</i> .....	120	14.2	56.1	29.6	11.9	7.8	617
" " <i>b</i> .....	113	1.9	8.2	32.7	68.0	2.2	622
Emergency ration, <i>a</i> .....	121	4.5	71.8	32.6	6.7	5.4	776
" " <i>b</i> .....	127	5.7	8.3	15.3	94.8	2.9	588
Nao meat food .....	437	231.3	56.9	90.1	46.2	12.5	1328
Army rations .....	661	420.2	101.2	84.3	47.9	7.4	1542
Standard emergency ration .....	418	23.6	129.6	90.5	160.3	14.0	2198
" " " <i>a</i> .....	270	17.0	50.6	54.8	137.0	10.6	1402
" " " <i>b</i> .....	49	0.5	3.2	10.5	34.0	0.8	254
Arctic food .....	423	30.7	75.1	167.3	119.8	30.1	2430
Tanty emergency ration .....	475	313.5	60.2	48.6	41.9	10.8	1482
F-A Food Company's stew .....	964	638.0	149.2	114.5	52.5	9.8	2460

\* Maine Exp. Sta., Bul. 75, p. 103.



## CHAPTER IX.

### EGGS.

**Nature and Structure.**—Though eggs of various birds are used to some extent as food, it is the egg of the hen that is in universal use for this purpose, and therefore the one which is here for the most part discussed, bearing in mind that the structure and composition of all varieties of birds' eggs are closely analogous.

Fig. 60 shows the longitudinal section of a hen's egg.

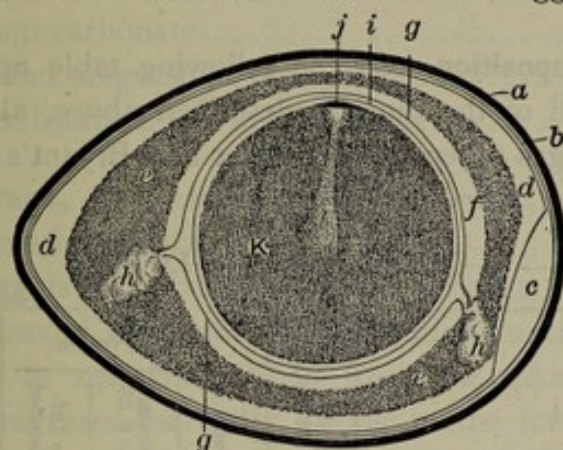


FIG. 60.—Longitudinal Section of a Hen's Egg. *a*, Shell; *b*, Double Membrane of Shell; *c*, Air-chamber; *d*, Outer, or Fluid Albuminous Layer; *e*, Thick, Middle Albuminous Layer; *f*, Inner Albuminous Layer; *g*, Membrane of the Chalaza; *hh*, the Chalaza; *i*, Vitelline Membrane; *j*, Germ; *k*, Yolk; *l*, Latebra. (After Macé.)

**Weight of Eggs.**—The average weights of whole and parts of hens' eggs, as given by Langworthy \* and Serono and Palazzi † (the latter for 1000 eggs), are as follows:

	Langworthy, grams.	Serono and Palazzi, grams.
Shell.....	6	7
White.....	33	32
Yolk.....	18	19
Total.....	57	58

\* U. S. Dept. Agric., Farmers' Bul. 128, 1901.

† Arch. farm. sper., 11, p. 553.



The data given in the following table were obtained by Woods and Merrill:\*

AVERAGE WEIGHTS OF EGGS AND PARTS AS PREPARED FOR ANALYSIS.

	Weight as Received.	Weight Boiled.				Shell (Refuse).	White.	Yolk.
		Shell (Refuse).	White.	Yolk.	Total. <sup>1</sup>			
	Grams.	Grams.	Grams.	Grams.	Grams.	Per Cent.	Per Cent.	Per Cent.
Turkey.....	105.5	11.7	60.1	30.9	102.7	11.4	56.5	30.1
Goose.....	190.4	24.1	98.5	64.8	187.4	12.8	52.6	34.6
Duck.....	70.6	7.2	36.5	24.4	68.1	10.6	53.6	35.8
Guinea fowl...	40.2	5.6	20.9	12.5	39.0	14.4	53.6	32.0

<sup>1</sup>Shrinkage due to loss in preparation and cooking.

**Proximate Composition.**—In the following table appear analyses by Woods and Merrill of the samples described above, also the average of analyses of hens' eggs taken from Atwater and Bryant's Compilation:

COMPOSITION OF EGGS.

		Refuse (Shells).	Water.	Protein.		Fat.	Ash.	Fuel Value per Pound.
				Nitrogen $\times 6.25$ .	By Difference.			
Turkey—	white.....		86.7	11.5	12.5	Trace	0.8	Cal. 325
	yolk.....		48.3	17.4	17.6	32.9	1.2	1875
	entire edible portion.....		73.3	13.4	14.2	11.2	0.9	850
	as purchased.....	13.8	63.5	11.6	12.2	9.7	0.8	735
Goose—	white.....		86.3	11.6	12.9	Trace	0.8	330
	yolk.....		44.1	17.3	18.4	36.2	1.3	1975
	entire edible portion.....		69.5	13.8	15.1	14.4	1.0	985
	as purchased.....	14.2	59.7	11.5	12.9	12.3	0.9	860
Duck—	white.....		87.0	11.1	12.2	Trace	0.8	315
	yolk.....		45.8	16.8	16.8	36.2	1.2	1980
	entire edible portion.....		70.5	13.3	14.0	14.5	1.0	985
	as purchased.....	13.7	60.9	11.5	12.1	12.5	0.8	880
Guinea fowl—	white.....		86.6	11.6	12.6	Trace	0.8	325
	yolk.....		49.7	16.7	17.3	31.8	1.2	1800
	entire edible portion.....		72.8	13.5	14.3	12.0	0.9	875
	as purchased.....	16.9	60.6	11.2	11.9	9.9	0.7	730
Hen—	white.....		86.2	12.3	13.0	0.2	0.6	250
	yolk.....		49.5	15.7	16.1	33.3	1.1	1705
	entire edible portion.....		73.7	13.4	14.8	10.5	1.0	720
	as purchased.....	11.2	65.5	11.9	13.1	9.3	0.9	635

\* Maine Exp. Sta., Bul. 75, 1901.



**Ash.**—The mineral content of the egg is thus shown by König:

COMPOSITION OF THE ASH OF EGGS.

	Ash of the Dry Sub- stance.	Potash.	Soda.	Lime.	Mag- nesia.	Iron Oxide.	Phos- phoric Acid.	Sul- phuric Acid.	Silica.	Chlo- rine.
Hen's egg: entire.	3.48	17.37	22.87	10.91	1.14	0.39	37.62	0.32	0.31	8.98
white.	4.61	31.41	31.57	2.78	2.79	0.57	4.41	2.12	1.06	28.82
yolk..	2.91	9.29	5.87	13.04	2.13	1.65	65.46	.....	0.86	1.95

**Egg Shell**, according to König, has the following composition:

Calcium carbonate.....	89.0-97%
Magnesium carbonate.....	0.0- 2%
Calcium and magnesium phosphate.....	0.5- 5%
Organic substances.....	2.0- 5%

**Egg Membrane**, the skin covering the white, consists chiefly of a protein of the keratin group.

**White of Egg** is an albuminous fluid without cellular structure. It has a specific gravity of 1.038-1.045, contains 12-18% of solids, and is always alkaline in reaction. When fresh the alkalinity is slight, but during ageing increases markedly, owing to the formation of ammonia. Determinations of ammonia are accordingly valuable in detecting deterioration. A blue color forms in the portion of the white adjacent to the yolk on boiling; this is due to iron sulphide formed by the action of hydrogen sulphide, liberated from the white, on iron of the yolk. The well-known blackening of silver by eggs is due to silver sulphide formed in a similar manner.

The average composition of the white of hens' eggs is as follows:

Water.....	86.2%
Fat, lecithin, cholesterol, etc.....	traces
Protein.....	12.7%
Dextrose.....	0.5%
Ash.....	0.6%
	<hr/> 100.0%

*Fats, Lecithin, and Cholesterol* are present only in negligible quantities.



*Proteins.*—According to Osborne and Campbell\* the proteins of the white of egg are four in number: ovalbumin, conalbumin, ovomucin, and ovomucoid. No sharp and distinct separation of these bodies has yet been made.

*Ovalbumin* is a crystallizable protein and forms with conalbumin the largest portion of the protein of the egg white. In a 2.5% solution in water, the ovalbumin starts to coagulate at 60° and yields a dense coagulum at 64°. Stronger solutions require a somewhat higher temperature for coagulation.

*Conalbumin* bears a close resemblance to ovalbumin, but is not crystalline, coagulates at a lower temperature (below 60°), and the coagulum is more flocculent.

*Ovomucin* is a globulin-like substance, precipitated from egg white by dilution with water. When dried and washed with alcohol it is a light, white powder soluble in strong sodium chloride solution. Some authors consider it as a part of "ovoglobulin" which is precipitated completely by saturation with magnesium sulphate, or half saturation with ammonium sulphate.

*Ovomucoid* is not coagulable by heat and may be separated (imperfectly) from the filtrate from the coagulable proteins. It is precipitated by alcohol and saturation with ammonium sulphate, but not by half saturation or by any proportion of sodium chloride, sodium sulphate, or magnesium sulphate.

*Carbohydrates.*—Kojo† reports 0.55% and Morner‡ 0.3–0.5% of dextrose. Diamare§ suggests that the sugar is not present as such, at least at the outstart, but is formed by the action of an amylolytic enzyme.

*Ash* is present to the extent of 0.04–0.07% and consists, as shown by the analyses on page 269, chiefly of alkali chlorides.

*Egg Yolk.*—This contains over 50% of solids or about four times that of the white and is also much more complex in composition. In addition to proteins it contains large amounts of fat, lecithin, and other phospholipins, also glucolipins, cholesterol, lutein (a lipochrome), hematogen (a nuclein?), salts, and other constituents.

Because of the complex composition of egg yolk which is as yet im-

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\* Jour. Amer. Chem. Soc., 22, 1900, p. 422.

† Zeits. physiol. Chem., 75, 1911, p. 1.

‡ Ibid., 80, p. 430.

§ Chem. Zentbl., 1, 1910, p. 1732.



perfectly understood, an accurate statement of composition is impossible. The following is based on the data at present available:

Water.....	49.5%
Fat.....	18.0%
Lecithin and other phospholipins.....	11.0%
Protein (ovovitellin, etc.) .....	14.5%
Dextrose.....	0.3%
Lutein, cholesterol, hematogen, cerasin (?), etc....	5.7%
Ash.....	1.0%
	<hr/> 100.0%

The Fat of egg yolk, extracted in various ways, has been studied by several investigators, but their results are far from comparable.

Pennington \* first dried the yolk by extraction for 2 days with absolute alcohol, evaporated the extract to dryness, and added the residue to the portion insoluble in the alcohol; she then ground the mass and extracted the fat for 2 days with petroleum ether (b. pt. 60° C.). Spaeth † and also Kitt, ‡ secured the fat by extracting the dried egg yolk with ether, while Palladino and Toso § expressed it from the yolk after boiling.

The constants of the fat obtained by the authors named, also Serono and Palazzi, || follow:

Analysts.	Specific Grav- ity at 15°.	Refractive Index at 25°.	Solidifying Point.	Melting Point.	Iodine Number.	Saponification Number.	Reichert-Meissl Number.	Acetyl Number.	Hehner Number.	Acid Number.	Melting Pt. of Fatty Acids.
Serono and Palazzi.	0.9121	.....	.....	.....	82.3	198.9	.....	3.82	.....	.....	37°
Pennington.....	.....	1.4627	.....	.....	62.8	179.9	.....	.....	76.1	206	.....
Spaeth.....	0.881*	1.4713	.....	.....	68.5†	184.4	0.7	.....	.....	.....	36°
Kitt.....	0.9144	.....	.....	.....	72.1	190.2	0.4	.....	95.2	195	.....
Palladino and Toso.	0.9156‡	.....	9°	23°	81.4	185.8	.....	.....	.....	.....	35°

\* At 100°.

† Of fatty acids 72.6.

‡ At 20°.

*Lecithin* (C<sub>42</sub>H<sub>84</sub>NPO<sub>9</sub>) is a characteristic constituent of egg yolk especially useful in detecting the presence and approximate amount of eggs in alimentary paste.

\* Jour. Biol. Chem., 7, 1910, p. 109.

† Zeits. Nahr. Unters. Hyg., 10, 1896, p. 171.

‡ Chem. Ztg., 21, 1897, p. 303.

§ Gior. pharm. chim.; abs. Jour. pharm. chim., 6, 1896, p. 247.

|| Arch. farm. sper., 11, p. 553.



*Lutein* ( $C_{40}H_{56}O_2$ ), the chief coloring matter of egg yolk, has the same empirical formula as xanthophyl and carotin, but has a lower melting point than xanthophyl and is more soluble in alcohol than carotin.

*Cholesterol* ( $C_{27}H_{44}O$ ) is present, according to Berg and Angerhausen,\* in amounts equivalent to about 1.40% in hens' and duck eggs.

*Ovovitellin*, the characteristic phosphoprotein of eggs, has been studied by Osborne and Campbell, who believe the substance as ordinarily isolated to be a mixture of compounds of true vitellin and lecithin.

*Hematogen* has been isolated by Bunge,† who believes it to be the mother substance of hemaglobin. It is similar to the nucleins and contains sulphur, phosphorus, and iron.

*Sugar*.—A small amount of dextrose is present in egg yolk.

**Grades of Eggs.**—Various systems of sorting are in vogue in different regions, but the following classification appears to be most common:

1. *Extras*.—Eggs of good size and uniform color, free from all defects.
2. *Firsts*.—Same as extras, but not of uniform color.
3. *Seconds*.—Small, dirty, checked, "weak" (with thin whites), and "leaker" eggs.
4. "*Spots*."—showing on candling dark areas due to mold or developing embryo ("blood rings").
5. "*Rots*."—Opaque on candling and offensive in odor on opening.

In addition the following are distinguished: *Green Eggs* with a greenish color in the white, *Musty Eggs* with a characteristic disagreeable taste, and *Sour Eggs*, with a sour taste.

**Preservation of Eggs.**—Owing to the porous nature of the shell, the moisture of the contents gradually grows less by evaporation, and the egg loses in weight. Air also passes in through the shell pores, carrying various microbes, which result in ultimate decomposition and spoiling of the egg. Nature has provided the shell with a thin surface coating of mucilaginous matter, which, however, is easily washed off. This coating tends to partially close the pores, and for best results in keeping should not be removed by washing.

Eggs are commonly preserved by protecting them as far as possible from the air. This is accomplished in a variety of ways, the most common being to pack the eggs in salt or bran, so that the packing medium fills up the interstices between the eggs. Eggs thus packed will keep considerably longer than when exposed to the air. A solution of salt is some-

\* Zeits. Unters. Nahr. Genussm., 29, 1915, p. 9.

† Jour. Chem. Soc., 93-94, p. 1500.



times employed, and also lime water, the eggs being simply packed in the solution. The use of lime water is, however, open to the serious objection that a disagreeable odor and taste are imparted to the eggs.

Eggs are sometimes coated with gelatin, vaseline, wax, or gum, so as to cover them with an impervious layer, either by dipping them in the coating medium, or by varnishing or otherwise applying the substance to the egg shell. By far the most efficacious egg coating has been shown by experiments in the North Dakota Experiment Station,\* and also in Germany, to be sodium and potassium silicate, or water glass. The fresh eggs, preferably unwashed, are packed in a jar, and a solution of water glass (1 part of syrupy water glass to 9 parts of boiled water) is poured over them. According to the North Dakota experiments, at the end of three and a half months, eggs packed in this manner the first of August appeared to be perfectly fresh.

One drawback to this method is that eggs so treated break more easily on boiling, but this may be prevented by carefully piercing the shell with a strong needle.

Cadet de Vanx has proposed immersing the egg in boiling water for twenty seconds, the result being that a very thin layer of the egg-white next the shell becomes coagulated, thus forming an impervious coating inside the shell.

**Cold-storage Eggs.**—The preservation of eggs by storage at low temperatures has become an enormous industry. The temperature employed differs somewhat, but a little below the freezing-point of water ( $-1^{\circ}$  to  $-2^{\circ}$  C.) gives the best results. The length of storage varies usually from one to ten months.

Experiments conducted by Wiley,† under authorization from Congress, brought out certain points as to the physical and chemical changes found to take place during cold storage. After breaking the shell and keeping at room temperature one day, the odor of eggs stored for 3.5 months was different from that of fresh eggs, but was not disagreeable. This odor increased on longer storage, and after 12.6 months became very characteristic. After 16.6 months, a musty odor was noticed immediately after opening the egg.

Chemical analysis by Cook showed that eggs in storage for one year lost 10% of the total weight, due to evaporation of water from the whites.

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\* Farmer's Bul. 103, U. S. Dept. of Agric., p. 18.

† U. S. Dept. of Agric., Bur. of Chem., Bul. 115.



Storage also caused a lowering of the amount of coagulable protein and of lecithin phosphorus, but an increase in lower nitrogen bodies, proteoses, and peptones. The acid reaction of yolks diminished during storage.

Microscopical examination by Howard and Read brought to light occasional rosette crystals in the yolk of eggs stored for 12 months or longer, but the nature of these crystals and their diagnostic value do not appear to have been established.

**Spoilage of Eggs.**—Pennington\* and her co-workers have found an average of 2 and 6 organisms per gram respectively in the white and yolk of strictly fresh eggs when incubated at 37° C., and 7 and 9 organisms per gram when incubated at 20° C. The average percentage of ammoniacal nitrogen in the whole egg was 0.0013. Resistance to spoilage appears to be greatest in the Spring when the moisture content is least and least in August and September when the moisture content is greatest. On keeping both the bacterial count and the per cent of ammoniacal nitrogen increase, but the latter to a much lesser degree than the former. The authors state that for certain constituents at least the count must approach 100,000,000 per gram before chemical methods for the detection of bacterial activity are of value.

Seconds, including medium stale, hatch spots, heavy rollers, dirties, checks, and eggs with yolks partially mixed with the white, opened aseptically, contained less than 1000 organisms per gram, while 26.5% of eggs with adhering yolks, 50% with dead embryos, 75.9% of moldy eggs, 66.7% of white rots, and 100% of black rots contained over 1000 per gram. With the exception of rots few contained *B. coli*.

Firsts opened commercially in July had low counts and the same was true of most clean-shelled seconds, only 8.3% containing over 1,000,000 organisms per gram, while 16.6% of the dirties, 18.8% of the checks, and 20% of the eggs with yolk partially mixed with the white exceeded that limit. *B. coli* ranged up to 100,000 per gram being no greater in the last named grades than in clean-shelled seconds. Market seconds in the producing sections during Summer averaged 0.0017–0.0022% of ammoniacal nitrogen, while other grades better than white rots varied up to 0.0030% or more.

Houghton and Weber† obtained the following percentages of ammoniacal nitrogen calculated to the dry substance of the liquid egg:

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\* U. S. Dept. of Agric., Bul. 51, 1914 and 224, 1916. Bur. of Chem. Circ., 98, 1912.

† Biochem. Bul. 3, 1914, 447.



	Folin Titration Method.	Folin Nesslerization Method.
Seconds.....	0.0114%	0.0124%
Spots.....	0.0141%	0.0200%
Light rots.....	0.0173%	0.0215%
Rots.....	0.0262%	0.0299%
Black rots.....	0.1696%	0.1486%

The authors also found Klein's modification of the Van Slyke method useful in detecting blood rings, spots and light rots, and the acidity of the fat in detecting spots and lower grades.

### FROZEN EGGS.

In the handling of eggs many become cracked or otherwise injured to an extent which renders them unfit for transportation. These are either sold to bakers for immediate use, or else opened and kept from spoiling by freezing, or drying. The portions of "spot eggs" that do not show evidence of damage are also treated by one of these methods, but together with white rots are now legitimately sold only for tanning certain kinds of leather.

Pennington and co-workers found that frozen eggs having less than 5,000,000 bacteria and less than 100,000 *B. coli* per gram, and with less than 0.0024% of ammoniacal nitrogen on the wet basis (0.0087% dry basis) can be made from most of the regular breaking stock.

The preservatives formerly much employed in opened eggs are boric acid and formaldehyde. The latter is especially effective as an egg preservative. If a small quantity be added and stirred into opened eggs that have become absolutely putrid, the result is astonishing. The product is completely deodorized, and exhibits the outward appearance at least of fresh eggs.

Formaldehyde, if present, may readily be detected by heating some of the egg directly with the hydrochloric-acid ferric-chloride reagent used in testing milk for formaldehyde, carrying out the process exactly as in the case of milk.



## DESICCATED EGGS.

This product is placed on the market as a coarse orange-yellow powder. It is particularly valuable because of its concentrated form and excellent keeping qualities without storing at freezing temperatures.

**Preparation of Desiccated Eggs.**—Breaking stock suitable for freezing may be preserved by drying, using the same precautions as to cleanliness in opening and handling.

Bailey\* who is located in one of the principal breaking sections (Kansas) states that the drying is carried out by spreading over cylinders or belts which move in a current of warm air or by heating in a vacuum. In some processes the drying is finished in wire baskets. The temperature is kept below 120° F. (49° C.) to prevent coagulation of the albumin. Salt or sugar are sometimes added to aid in preservation.

If properly prepared the powder mixes readily with water, assuming much the same form as before drying.

**Composition.**—The following are analyses of two samples one (A) made by the Bureau of Chemistry, the other (B) by the Massachusetts State Board of Health:

	A.	B.
Water.....	6.80	5.95
Protein (N×6.25) .....	45.20	48.15
Protein by difference.....	51.20	
Fat.....	38.5	40.56
Ash.....	3.5	5.34

**Inspection.**—Pennington states that the percentage of ammoniacal nitrogen is not a reliable index of the quality of the stock, owing to the volatilization of more or less of this constituent during desiccation. As in the case of frozen eggs, factory inspection is desirable, particularly as the drying is not carried out at a sterilizing temperature.†

## ANALYSIS OF EGGS.

**Physical Examination of Eggs.**—Various physical tests, based on the gradual increase in size of the air chamber (Fig. 60), have been prescribed for ascertaining the approximate age of an egg. Thus, accord-

\* Source, Chemistry, and Use of Food Products, Phila., 1914, p. 438.

† See Maurer, Kan. Agric. Exp. Sta., Bul. 180, p. 345.



ing to Delarne, if the egg, when placed in a 10% salt solution sinks to the bottom, it may be considered perfectly fresh; if it remains immersed in the liquid, it is to be considered at least three days old; and if it rises to the surface and floats thereon it is more than five days old. This test is a very rough one, and is useful only for eggs that have been kept in the air. Preserved eggs cannot be gauged by this means.

The commercial method of examining eggs is by "candling," consisting in placing the egg in front of an opening in a screen between a bright light and the eye. If the egg is fresh, it will show a uniform rose-colored tint, without dark spots, the air-chamber being small and occupying about one-twentieth the capacity of the egg. If the egg is not fresh, it will appear more or less cloudy, being darker as the egg grows older, becoming in extreme cases opaque. At the same time the air-chamber grows larger as the age increases. So-called "spots" show on candling black patches.

Greenlee\* has proposed the moisture content as an index of age and has devised a "rate formula" for predicting the condition after holding for a definite time at a definite temperature.

**Preparation of the Sample.**†—The egg is first weighed as a whole and afterwards boiled hard, cooled, and again weighed. The shell, white, and yolk are then carefully separated and each weighed. After rejecting the shell, the yolk and white are separately reduced by a chopping-knife to the size of wheat grains. These portions are dried partially at a temperature not exceeding 45° C., weighed, and afterwards ground to a fine powder in a mortar.

Pennington separates the yolk from the white by draining on a piece of wire gauze, then washes off any adhering white with water and dries by extraction with alcohol as described on page 271.

Fat constants are determined on the petroleum ether extract.

**Determination of Water, Ether Extract, Total Nitrogen, and Ash** are made in practically the same manner as with flesh foods. It is well to determine water with the addition of sand, after which the residue may be ground up for extraction with ether, using a continuous flow extractor.

Little attention has been paid as yet to the complete separation and determination of the nitrogen compounds in the white and yolk, and it

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 83, 1911.

† Woods and Merrill, Maine Exp. Sta., Bul. 75, p. 92.



is customary to calculate the protein by the use of the factor 6.25 or by difference.

**Determination of Lecithin.**—*The Juckenack Method* (page 366), devised for noodles, is also applicable to eggs, previously dried at a moderate temperature with sand, and to commercial dried eggs. If desired, the free lecithin, or that present in the ether extract, and the combined lecithin, obtained by extraction with absolute alcohol of the residue after removal of the ether extract, may be separately determined.

**Detection of Preservatives.**—*Formaldehyde* may be readily detected by heating directly with the acid ferric chloride reagent as described for milk.

*Boric Acid.* See Chapter XVIII. Bertrand and Agulhon\* find by their spectroscopic method 0.116–0.136 mg. of boron as hydroxide per kilo of egg white and 0.008 mg. per kilo of egg yolk, to which no preservative has been added. These amounts are not detectable by ordinary methods.

*Salicylic Acid.*—Froideaux† proceeds as follows: Mix 25 grams of desiccated or 30 grams of liquid egg with 250 cc. of water, stir in 125 cc. of 8% sodium hydroxide solution, and warm for 45 minutes on a water-bath. Break up the mass with a rod and filter. Acidulate the filtrate with hydrochloric acid, precipitate proteins with sodium phosphomolybdate, filter, extract the filtrate with ether, and proceed as usual.

#### EGG SUBSTITUTES.

There have been many preparations in powdered form sold under this name, nearly all claiming to contain all the ingredients of eggs, but most of them falling far short of these claims. Some of them, as, for instance, those made from desiccated skimmed milk, do contain nitrogenous matter, but as a rule little, if any, fat.

Two samples of "egg substitute" sold in Massachusetts were analyzed with the following results:‡

	A.	B.
Protein.....	16.94	18.72
Fat.....	3.43	3.40
Water.....	6.71	7.01
Corn starch, salts, and coloring matter.	72.92	70.87

\* Compt. rend., 1913, 156, p. 2027.

† Jour. pharm. chim., 10, p. 18.

‡ An. Rep. Mass. State Board of Health, 1895, p. 675.



A ten-cent package of sample A, weighing about 2 ounces, was alleged to be equivalent to 12 eggs. Starch furnished the chief ingredient in both samples.

One of the most flagrant examples of fraud in this connection was a product sold under the name "N'egg," advertised to contain the nutritive equivalent of the whites and yolks of a dozen eggs, "their composition being based on careful scientific analysis of natural eggs." It was put up in two small boxes, one containing a white and the other a yellow dry powder. Both were entirely devoid of nitrogen, and consisted of nearly pure tapioca starch with a little common salt, the color of the "yolk" being due to Victoria yellow.

Some egg substitutes are sold under the name of "custard powders," and are alleged to take the place of eggs in cooking. These are variously made up of mixtures of skim-milk powder, coloring matter, and baking powder ingredients as shown from the following analyses:\*

## CUSTARD POWDERS.

	1	2	3	4	5	6
Starch .....	86.25	84.45	51.03	26.38	52.32	53.82
Albuminous compounds .....	0.59	0.58	6.01	2.96	6.00	5.06
Soluble coloring matter .....	0.88	0.98				
Baking soda .....			15.33	50.70	22.11	26.71
Tartaric acid .....			13.69	10.33	11.37	6.19
Phosphates .....			0.24			
Carbonates of lime and magnesia .....			2.70			
Water .....	11.83	13.69	11.00	9.63	8.20	8.22
Ash .....	0.45	0.38				

\* Food and Sanitation, Nov. 25, 1893.



## CHAPTER X.

### CEREALS AND THEIR PRODUCTS, VEGETABLES, FRUITS, AND NUTS.

THE chief points of difference in composition between the animal foods already treated of, and those of the vegetable kingdom, are apparent in the relative amounts of proteins and carbohydrates. The proteins present in the cereals differ materially both in character and amount from those in the flesh foods, being, as a rule, present in much smaller amount. The leguminous foods, such as peas, beans, and lentils, and nuts as a rule, are, however, comparatively high in nitrogenous content.

The carbohydrates, which in the flesh foods are almost entirely lacking, and in milk in the form of lactose make up about one-third of the solid matter, constitute the greater part of the cereals and legumes, being present chiefly in the form of starch.

In nuts, with few exceptions such as the chestnut and peanut, starch is absent.

The composition of the principal cereal grains is tabulated as follows by Villier and Collin:

	Wheat.	Barley.	Rye.	Oats.	Rice.	Corn.	Millet.	Buck-wheat.
Water.....	13.65	13.77	15.06	12.37	13.11	13.12	11.66	12.93
Nitrogenous substances.	12.35	11.14	11.52	10.41	7.85	9.85	9.25	10.30
Fat.....	1.75	2.16	1.79	5.32	0.88	4.62	3.50	2.81
Sugar.....	1.45	1.56	0.95	1.91	16.52	2.46	65.95	55.81
Gum and dextrin.....	2.38	1.70	4.86	1.79		3.38		
Starch.....	64.08	61.67	62.00	54.08		62.57		
Cellulose.....	2.53	5.31	2.01	11.19	0.63	2.49	7.29	16.43
Ash.....	1.81	2.69	1.81	3.02	1.01	1.51	2.35	2.72

The following results of the analyses of cereal grains are summarized from the work of the Division of Chemistry, United States Department of Agriculture:\*

\* Bulletin 13, part 9.



## CEREAL GRAINS.

	Num- ber of Analy- ses.	Weight of 100 Ker- nels, Grams.	Moist- ure.	Pro- teins.	Ether Ex- tract.	Crude Fiber.	Ash.	Carbohydrates, other than Crude Fiber.	Wet Gluten.	Dry Gluten.
Barley:	14									
Mean.....		4.533	6.47	11.52	2.67	3.81	2.87	72.66		
Buckwheat:	10									
Mean.....		3.069	12.31	10.86	2.06	10.57	1.85	63.34		
Corn, domestic:										
Maximum.....		48.312	12.32	11.55	5.06	2.00	1.55	75.07		
Minimum.....		10.608	9.58	8.58	2.94	1.00	1.19	68.97		
Mean.....		38.979	10.93	9.88	4.17	1.71	1.36	71.95		
Oats, domestic:										
Maximum.....		3.891	13.02	15.05	6.14	16.65	4.37	61.44		
Minimum.....		2.038	7.87	9.10	0.93	8.57	1.47	53.70		
Mean.....		2.918	10.06	12.15	4.33	12.07	3.46	58.75		
Rice:										
Unhulled.....	4	2.929	10.28	7.95	1.65	10.42	4.09	65.60		
Unpolished....	6	2.466	11.88	8.02	1.96	0.93	1.15	76.05		
Polished*.....	14	2.132	12.34	7.18	0.26	0.40	0.46	79.36		
Rye, domestic:										
Maximum.....		4.201	11.45	18.99	2.30	2.50	2.41	75.36		
Minimum.....		1.932	9.54	8.40	1.16	1.65	1.71	63.61		
Mean.....		2.493	10.62	12.43	1.65	2.09	1.92	71.37		
Wheat, domestic:										
Maximum.....		6.190	14.53	17.15	2.50	3.72	2.35	76.05	39.05	14.65
Minimum.....		2.125	7.11	8.58	0.28	1.70	1.40	66.67	12.33	4.77
Mean.....		3.866	10.62	12.23	1.77	2.36	1.82	71.18	26.46	10.31
Wheat, foreign:										
Maximum.....		5.723	12.97	14.52	2.26	2.89	2.04	76.14	32.57	12.33
Minimum.....		2.250	8.52	8.58	0.73	1.87	1.67	67.01	18.72	7.00
Mean.....		4.076	11.47	12.08	1.78	2.28	1.73	70.66	25.36	9.82

\* Polished rice in the United States is commonly coated with glucose and talc, ostensibly as a protection against dust and the ravages of insects. Such coating is allowed if declared on the label and directions for its removal are also given.

Ballard † gives the following percentage composition of beans, lentils, and peas:

	Beans.		Lentils.		Peas.	
	Min.	Max.	Min.	Max.	Min.	Max.
Water.....	10.10	20.40	11.70	13.50	10.60	14.20
Nitrogenous substances....	13.81	25.46	20.42	24.24	18.88	22.48
Fat.....	0.98	2.46	0.58	1.45	1.22	1.40
Sugars and starches.....	52.91	60.98	56.07	62.45	56.21	61.10
Cellulose.....	2.46	4.62	2.96	3.56	2.90	5.52
Ash.....	2.38	4.20	1.99	2.66	2.26	3.51*

† Jour. Pharm. Chem., 1897, pp. 196, 197.



The composition of potatoes, according to Balland,\* is as follows:

	Water.	Nitrogenous Substances.	Fat.	Sugar and Starch.	Cellulose.	Ash.
Normal state—minimum ..	66.10	1.43	0.04	15.58	0.37	0.44
maximum..	80.60	2.81	0.14	29.85	0.68	1.18
Dried—                  minimum ..	.....	5.98	0.18	80.28	1.40	1.66
maximum..	.....	13.24	0.56	89.78	3.06	4.38

The composition of the common vegetables, fruits, and berries is thus given by Atwater and Bryant.†

#### VEGETABLES.

		Number of Analyses.	Refuse.	Water.	Protein.	Fat.	Total Carbohy- drates.	Crude Fiber.	Ash.	Fuel Value per Pound, Calories.
Asparagus—	as purchased...	3	.....	94.0	1.8	0.2	3.3	.8	.7	105
Beans, dried—	as purchased...	11	.....	12.6	22.5	1.8	59.6	4.4	3.5	1605
Beans, fresh Lima—	edible portion..	1	.....	68.5	7.1	.7	22.0	1.7	1.7	570
	as purchased...	.....	55.0	30.8	3.2	.3	9.9	.8	.8	255
Beets, fresh—	edible portion..	24	.....	87.5	1.6	.1	9.7	.9	1.1	215
	as purchased...	.....	20.0	70.0	1.3	.1	7.7	.....	.9	170
Cabbage—	edible portion..	16	.....	91.5	1.6	.3	5.6	1.1	1.0	145
	as purchased...	.....	15.0	77.7	1.4	.2	4.8	.....	.9	125
Carrot, fresh—	edible portion..	18	.....	88.2	1.1	.4	9.3	1.1	1.0	210
	as purchased...	.....	20.0	70.6	.9	.2	7.4	.....	.9	160
Celery—	edible portion..	5	.....	94.5	1.1	.1	3.3	.....	1.0	85
	as purchased...	.....	20.0	75.6	.9	.1	2.6	.....	.8	70
Cauliflower—	as purchased...	2	.....	92.3	1.8	.5	4.7	1.0	.7	140
Cucumber—	edible portion..	4	.....	95.4	.8	.2	3.1	.....	.5	80
	as purchased...	.....	15.0	81.1	.7	.2	2.6	.....	.4	70
Lettuce—	edible portion..	8	.....	94.7	1.2	.3	2.9	.7	.9	90
	as purchased...	.....	15.0	80.5	1.0	.2	2.5	.....	.8	75
Mushrooms—	as purchased...	11	.....	88.1	3.5	.4	6.8	.8	1.2	210
Onion, fresh—	edible portion..	15	.....	87.6	1.6	.3	9.9	.8	.6	225
	as purchased...	.....	10.0	78.9	1.4	.3	8.9	.....	.5	205
Parsnip—	edible portion..	3	.....	83.0	1.6	.5	13.5	2.5	1.4	300
	as purchased...	.....	20.0	66.4	1.3	.4	10.8	.....	1.1	240
Pumpkin—	edible portion..	3	.....	93.1	1.0	.1	5.2	1.2	.6	120
	as purchased...	.....	50.0	46.5	.5	.1	2.6	.....	.3	60
Radish—	edible portion..	4	.....	91.8	1.3	.3	8.3	.7	1.0	135
	as purchased...	.....	30.0	64.3	.9	.1	5.8	.7	.7	95
Rhubarb—	edible portion..	2	.....	94.4	.6	.7	3.6	1.1	.7	105
	as purchased...	.....	40.0	56.6	.4	.4	2.2	.....	.4	65
Squash—	edible portion..	10	.....	88.3	1.4	.5	9.0	.8	.8	215
	as purchased...	.....	50.0	44.2	.7	.2	4.5	.....	.4	105
Tomato, fresh—	as purchased...	27	.....	94.3	.9	.4	3.9	.6	.5	105
Turnip—	edible portion..	19	.....	89.6	1.3	.2	8.1	1.3	.8	185
	as purchased...	.....	30.0	62.7	.9	.1	5.7	.....	.6	125

\* Jour. Pharm. Chem., 1897, pp. 298-300.

† Bul. 28, Office of Exp. Station U. S. Dept. of Agriculture.



## FRUITS.

		Num. ber of Analyses.	Refuse.	Water.	Protein.	Fat.	Total Carbo- hydrates.	Crude Fiber.	Ash.	Fuel Value per Pound, Calories.
Apples—	edible portion . . . .	29	.....	84.6	.4	.5	14.2	1.2	.3	290
	as purchased . . . . .	.....	25.0	63.3	.3	.3	10.8	.....	.3	220
Apricots—	edible portion . . . . .	11	.....	85.0	1.1	.....	13.4	.....	.5	270
	as purchased . . . . .	.....	6.0	79.9	1.0	.....	12.6	.....	.5	255
Bananas—	edible portion . . . . .	6	.....	75.3	1.3	.6	22.0	1.6	.8	460
	as purchased . . . . .	.....	35.0	48.9	.8	.4	14.3	.....	.6	300
Blackberries—	as purchased . . . . .	9	.....	86.3	1.3	1.0	10.9	2.5	.5	270
Cherries—	edible portion . . . . .	16	.....	80.9	1.0	.8	16.7	.2	.6	365
	as purchased . . . . .	.....	5.0	76.8	.9	.8	15.9	.....	.6	345
Cranberries—	as purchased . . . . .	3	.....	88.9	.4	.6	9.9	1.5	.2	215
Currants—	as purchased . . . . .	1	.....	85.0	1.5	.....	12.8	.....	.7	265
Figs, fresh—	as purchased . . . . .	28	.....	79.1	1.5	.....	18.8	.....	.6	380
Grapes—	edible portion . . . . .	5	.....	77.4	1.3	1.6	19.2	4.3	.5	450
	as purchased . . . . .	.....	25.0	58.0	1.0	1.2	14.4	.....	.4	335
Huckleberries—	edible portion . . . . .	1	.....	81.9	.6	.6	16.6	.....	.3	345
Lemons—	edible portion . . . . .	4	.....	89.3	1.0	.7	8.5	1.1	.5	205
	as purchased . . . . .	.....	30.0	62.5	.7	.5	5.9	.....	.4	145
Muskmelons—	edible portion . . . . .	1	.....	89.5	.6	.....	9.3	2.1	.6	185
	as purchased . . . . .	.....	50.0	44.8	.3	.....	4.6	.....	.3	90
Oranges—	edible portion . . . . .	23	.....	86.9	.8	.2	11.6	.....	.5	240
	as purchased . . . . .	.....	27.0	63.4	.6	.1	8.5	.....	.4	170
Pears—	edible portion . . . . .	2	.....	84.4	.6	.5	14.1	2.7	.4	295
	as purchased . . . . .	.....	10.0	76.0	.5	.4	12.7	.....	.4	260
Pineapple—	edible portion . . . . .	1	.....	89.3	.4	.3	9.7	.4	.3	200
Plums—	edible portion . . . . .	3	.....	78.4	1.0	.....	20.1	.....	.5	395
	as purchased . . . . .	.....	5.0	74.5	.9	.....	19.1	.....	.5	370
Prunes—	edible portion . . . . .	24	.....	79.6	.9	.....	18.9	.....	.6	370
	as purchased . . . . .	.....	5.8	75.6	.7	.....	17.4	.....	.5	335
Raspberries—	as purchased . . . . .	1	.....	85.8	1.0	.....	12.6	2.9	.6	255
Strawberries—	edible portion . . . . .	22	.....	90.4	1.0	.6	7.4	1.4	.6	180
	as purchased . . . . .	.....	5.0	85.9	.9	.6	7.0	.....	.6	175
Watermelon—	edible portion . . . . .	2	.....	92.4	.4	.2	6.7	.....	.3	140
	as purchased . . . . .	.....	59.4	37.5	.2	.1	2.7	.....	.1	60

The following analyses of apples made by Browne\* are of interest. The first four analyses show the changes that occur in the composition of a Baldwin apple at different stages of its growth. Below these is given the average of the analysis of 160 samples, representing 27 varieties of apples.

## COMPOSITION OF A BALDWIN APPLE AT DIFFERENT PERIODS.

Condition.	Water.	Solids.	Invert Sugar.	Su- crose.	Total Sugar.	Total Sugar after In- version.	Starch.	Free Malic Acid.	Ash.	Sugar Co- efficient.
Very green..	81.53	18.47	6.40	1.63	8.03	8.11	4.14	1.14	0.27	47.16
Green. ....	79.81	20.19	6.46	4.05	10.51	10.72	3.67	.....	.....	53.10
Ripe. ....	80.36	19.64	7.70	6.81	14.51	14.87	0.17	0.65	0.27	75.71
Over-ripe. ..	80.30	19.70	8.81	5.26	14.07	14.35	.....	0.48	0.28	72.84

\* Penn. Dept. of Agriculture, Bulletin 58.



## AVERAGE COMPOSITION OF 27 VARIETIES OF APPLES.

Water.....	83.57
Solids.....	16.43
Invert sugar.....	7.92
Sucrose.....	3.99
Total sugar.....	11.91
Total sugar after inversion.....	12.12
Free malic acid.....	0.61
Ash.....	0.27
Sugar coefficient.....	73.76

The composition of the commoner nuts as compiled by Atwater and Bryant \* is shown in the following table:

## NUTS.

		Number of Analyses.	Refuse.	Water.	Protein.	Fat.	Total Carbo- hydrates.	Crude Fiber.	Ash.	Fuel Value per Pound Cal's.
Almonds—	edible portion..	11	.....	4.8	20.0	54.9	17.3	2.0	2.0	3030
	as purchased..	.....	45.0	2.7	11.5	30.2	9.5	.....	1.1	1660
Beechnuts—	edible portion..	1	.....	4.0	21.9	57.4	13.2	.....	3.5	3075
	as purchased..	.....	40.8	2.3	13.0	34.0	7.8	.....	2.1	1820
Brazil-nuts—	edible portion..	1	.....	5.3	17.0	66.8	7.0	.....	3.9	3265
	as purchased..	.....	49.6	2.6	8.6	33.7	3.5	.....	2.0	1655
Butternuts—	edible portion..	1	.....	4.4	27.9	61.2	3.5	.....	2.9	3165
	as purchased..	.....	86.4	.6	3.8	8.3	.5	.....	.4	430
Chestnuts, fresh—	edible portion..	9	.....	45.0	6.2	5.4	42.1	1.8	1.3	1125
	as purchased..	.....	16.0	37.8	5.2	4.5	35.4	.....	1.1	945
Cocanuts—	edible portion..	1	.....	14.1	5.7	50.6	27.9	.....	1.7	2760
	as purchased..	.....	48.8	7.2	2.9	25.9	14.3	.....	.9	1413
Filberts—	edible portion..	1	.....	3.7	15.6	65.3	13.0	.....	2.4	3290
	as purchased..	.....	52.1	1.8	7.5	31.3	6.2	.....	1.1	1575
Hickory-nuts—	edible portion..	1	.....	3.7	15.4	67.4	11.4	.....	2.1	3345
	as purchased..	.....	62.2	1.4	5.8	25.5	4.3	.....	.8	1265
Peanuts—	edible portion..	4	.....	9.2	25.8	38.6	24.4	2.5	2.0	2560
	as purchased..	.....	24.5	6.9	19.5	29.1	18.5	.....	1.5	1935
Pecans—	edible portion..	1	.....	3.0	11.0	71.2	13.3	.....	1.5	3455
	as purchased..	.....	53.2	1.4	5.2	33.3	6.2	.....	.7	1620
Pistachios—	edible portion..	1	.....	4.2	22.3	54.0	16.3	.....	3.2	2995
Walnuts, Calif'nia—	edible portion..	1	.....	2.5	18.4	64.4	13.0	1.4	1.7	3306
	as purchased..	.....	73.1	.7	4.9	17.3	3.5	.....	.5	885

\* U. S. Dept. of Agric., Off. of Exp. Station, Bul. 28.



## GENERAL METHODS OF ANALYSIS.

**Preparation of the Sample.**—Except otherwise stated grind so as to pass a sieve with round holes 1 mm. ( $\frac{1}{25}$  inch) in diameter using a hand mill, iron mortar, or other suitable device.

The following methods for water (standard method), ash, protein, fiber, fat, and nitrogen-free extract, in essential details, have been in use since the middle of the nineteenth century and have been adopted by the A. O. A. C.

**Determination of Water.**—*Standard Method.*—Dry 2 grams of the material to constant weight (about four hours) at the temperature of boiling water, in a current of dry hydrogen or *in vacuo*. The apparatus described on page 50 may be used.

**Determination of Moisture in Grain, Legumes, Oil Seeds, etc.**—*Brown and Duvel Method.\**—This method is especially useful in guarding

against an excessive amount of moisture in grain, which not only adds weight but also causes deterioration through the growth of bacteria and moulds.

The apparatus (Fig. 61) consists of a condenser-tank (A) and an evaporating-chamber (B) with a cover (k) and a mica window (m), the whole supported on a stand (C). It is arranged for conducting six distillations at the same time.

The distilling-flask is shown at the left (p') in the wooden rack used only during filling and at the right (p) in position for distillation.

Weigh into the distilling-flask 100 grams (corn, barley, wheat, rye, unhulled rice, kafir, flaxseed, soy bean) or 50 grams (oats, cottonseed) of the whole grain and add 150 cc. of hydrocarbon engine oil with a flash-point in an open cup of 200°—

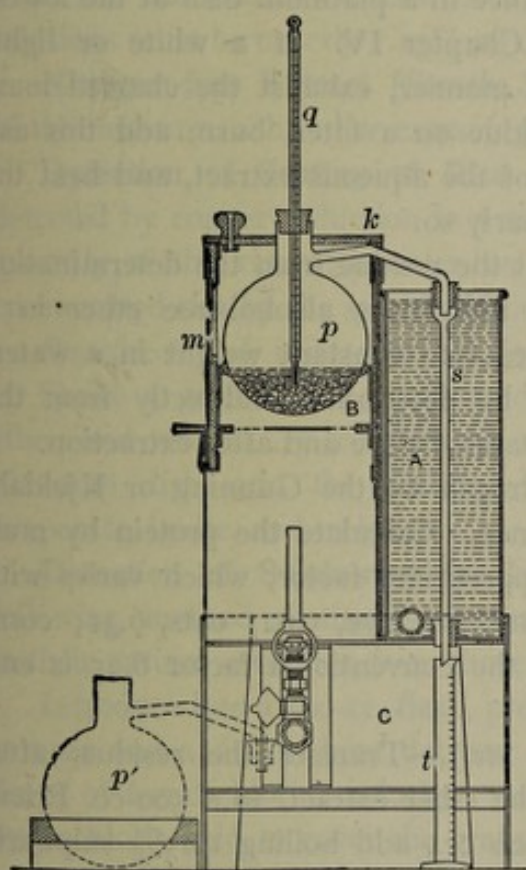


FIG. 61.—Brown and Duvel Apparatus for Determination of Moisture in Grain. End View.

\* U. S. Dept. of Agric., Bur. of Plant. Ind., Bul. 99, and Circular 72.



205° C. Close the neck of the flask with a rubber stopper carrying a thermometer (*q*), the bulb of which extends well into the mixture of oil and corn; connect the side tube by means of another cork with the condenser-tube (*s*), and heat with the Bunsen burner, so as to bring (in twenty minutes) to the proper temperature, which for corn, barley, rice, kafir, and cottonseed is 190°, for wheat 180°, for rye and flaxseed 175°, for soy bean 170°, and for oats 195° C. When the desired temperature is reached turn off the flame, and allow to stand until the moisture ceases to drop from the condenser-tube into the graduate (*l*). The number of cc. in the graduate represents the amount of moisture in the grain.

The results agree closely with those by drying to constant weight in a water-oven at 100°.

After the determination is finished empty the contents of the flask on a suitable strainer, thus recovering the oil for further use.

**Ash.**—Burn 2 grams of the substance in a platinum dish at the lowest possible red heat, as described in Chapter IV. If a white or light-gray ash cannot be obtained in this manner, exhaust the charred mass with water, collect the insoluble residue on a filter, burn, add this ash to the residue from the evaporation of the aqueous extract, and heat the whole at low redness until white or nearly so.

**Ether Extract** (*Fat, etc.*).—Extract the residue from the determination of moisture for sixteen hours with anhydrous alcohol-free ether in a continuous extractor and dry the extract to constant weight in a water-oven. The ether extract may also be determined indirectly from the difference in weight of the dried substance before and after extraction.

**Protein.**—Determine the total nitrogen by the Gunning or Kjeldahl method, using 1 gram of the substance. Calculate the protein by multiplying the total nitrogen by the appropriate factor, which varies with the different cereals as follows: wheat, 5.70; rye, 5.62; oats, 6.31; corn, 6.39; and barley, 5.82. Ordinarily the conventional factor 6.25 is employed.

**Crude Fiber.** (*Cellulose, Lignin, etc.*).—Transfer the residue, after extraction for the determination of the ether extract, to a 500-cc. Erlenmeyer flask, with a mark showing 200 cc., add boiling 1.25% sulphuric acid to the mark, heat at once to boiling, and boil gently for thirty minutes, shaking cautiously from time to time to prevent the material from crawling up on the sides of the flask. Filter through paper, and wash once with boiling water. Rinse the substance back into the same flask with 200 cc. of a boiling 1.25% solution of sodium hydroxide, free,



or nearly so, from sodium carbonate, boil at once, and continue the boiling for thirty minutes in the same manner as directed above for the treatment with acid. Filter on a tared filter-paper or Gooch crucible, and wash with boiling water till the washings are neutral. Dry at  $110^{\circ}$  and weigh, after which incinerate completely and correct for ash.

If a tared filter is used, it should be previously dried at  $110^{\circ}$  for one hour in a glass-stoppered weighing bottle, cooled for fifteen minutes and weighed. After collecting the fiber on the filter, it is well to wash successively with alcohol and ether to facilitate drying. The filter should not be pushed down into the weighing bottle until the fiber is dry to the touch after which two hours' drying at  $110^{\circ}$  will be sufficient. A blank should be made to ascertain the loss sustained by the filter on treatment with alkali and the necessary correction introduced. This error and others can be avoided by filtering on a Gooch crucible, but with many materials this cannot be used because of clogging. The acid and alkali solutions must be exactly 1.25% as determined by titration.

**Nitrogen-free Extract** (*Starch, Sugar, Gums, etc.*)—Subtract the sum of the moisture, ash, ether extract, protein, and crude fiber from 100.

**Detection of Coating of Rice with Talc, Glucose, etc.**—*Glucose* is detected by copper reduction in a solution obtained by shaking a weighed quantity of the rice with water, using for comparison the results obtained on uncoated rice by the same method.

*Steatite or Talc* is detected by Jones\* as follows:

Shake 5 grams of the unground material successively with 20 cc. of ether and several 15-cc. portions of water. Evaporate the ether in a platinum dish, add the settlings from the combined aqueous liquids, evaporate, ignite, and weigh.

**Detection of Sulphuring in Grain.**—Carroll† has shown that the following method distinguishes sharply barley, oats and other grains in their natural condition from those bleached by sulphurous acid.

Introduce into a 500-cc. flask, provided with a desulphurized perforated stopper and a double-bent delivery tube, 10 grams of mossy zinc, a few drops of ferric chloride solution, 100 grams of the grain and enough 8% hydrochloric acid to cover the grain. Place a loose plug of cotton in the neck, attach the stopper, and run the delivery tube into a test-tube containing 10 cc. of 2% lead acetate solution. If a brownish black precipitate of lead sulphide forms, the grain has been sulphured. Particles

\* Chem. News, 108, 1913, p. 176.

† U. S. Dept. of Agric., Bur. of Plant Ind., Circ. 40.



of dust or zinc, occasionally carried over mechanically, are distinguished from lead sulphide by their insolubility in 10% ferric chloride solution.

#### CARBOHYDRATES OF CEREALS AND VEGETABLES.

**Classification.**—As a rule the same carbohydrates are found in all cereals, being present, however, in varying proportions. By far the greater part of the carbohydrate content of cereals is starch, the other carbohydrates being comparatively small in amount, so that in rough work it is sometimes customary, though incorrect, to assume the entire amount of so-called “nitrogen-free extract” or carbohydrates (as determined by difference) to be starch.

The carbohydrates occurring in cereals may be classified as follows:

Principal carbohydrates of cereals:	{	Insoluble.....	{	Starch
			{	Cellulose
			{	Pentosans
	{	Soluble.....	{	Sucrose
			{	Dextrose
			{	Dextrin
			{	Raffinose (traces)

**STARCH** ( $C_5H_{10}O_5$ )<sub>n</sub>.—Pure starch is a glistening, white, granular powder having a peculiar feeling when rubbed between the thumb and finger. It is a very hygroscopic, commercial starch containing about 18% of moisture. Starch is very widely distributed in the vegetable kingdom, occurring in almost every plant at some stage in its growth.

Starch is insoluble in cold water, alcohol, and ether; it is soluble in hot water, though not without change. By boiling with dilute acids, starch is first converted by hydrolysis into a mixture of dextrin and maltose, and finally by prolonged boiling into dextrose. Malt extract also hydrolyzes starch in solution.

**Detection.**—Starch is best detected, when present to any appreciable extent in any mixture, by boiling a portion of the sample in water, cooling, and applying a solution of iodine. A characteristic blue color is produced if starch is present. Very small amounts of starch are best identified in powdered mixtures by applying a drop of a solution of iodine to the dry powder on a microscope slide, or, better, to the powder previously rubbed out with water on a slide under a cover-glass; the starch granules, if present, will be colored intensely blue by the iodine, and are at once rendered apparent when viewed through the microscope.



Though the cereal and vegetable starches, whatever their origin, are identical chemically, the various starch granules have certain characteristics, when viewed under the microscope, that render their identification easy in most cases. A knowledge of the microscopical appearance of the common vegetable starches is of the utmost importance to the public analyst, who frequently finds them as adulterants of various foods, such as coffee, cocoa, spices, etc. For microscopical examination, powdered samples should be ground fine enough to pass through a 60 or 80 mesh sieve.

**Classification.**—As seen under the microscope the starch granules of various grains and vegetables differ in form, size, and often in their manner of grouping. Thus, at the outset, the common starches may be divided as to the microscopical form of their granules into three classes, viz., lenticular, irregularly oval, and polygonal. To the first class, in which the starch granule has in general the circular disk form, belong rye, wheat, and barley. Representing the second or irregularly elliptical class are the pea, bean, potato, and arrowroot. In the third, or polygonal class, should be included corn, oats, buckwheat, and rice. In thus characterizing the distinguishing forms as lenticular, oval, and polygonal, it should be borne in mind that while the tendency of the most typical starch granules in each class, when viewed in normal position, is toward the circular, the oval, or the polygonal as the case may be, it is not by any means true that all or even most of the granules in any one instance perfectly conform to one of these shapes throughout. Thus, lenticular wheat granules, when viewed edgewise, will appear elliptical, and are often distorted in shape, especially when roasted; and polygonal buckwheat granules may in many instances have such obtuse angles as to appear circular. It is the general trend of all the starches toward one or another of these shapes that suggests the classification.

The identification of the various starches morphologically is indeed the most natural and ready method. Not only the character of the starch, but also its approximate amount, when present in mixtures, can in many instances be ascertained by a careful examination with the microscope. The analyst should be provided with samples of starches of known purity conveniently at hand, and in all doubtful cases these should be referred to for comparison.

*Wheat Starch* (Fig. 152, Pl. VIII).—This starch is frequently present in adulterated pepper, mustard, ginger, cocoa, coffee, and other foods. Its granules occur for the most part in two sizes, of which the larger



are lenticular, varying from 0.021 mm. to 0.041 mm., or rarely 0.050 mm., in diameter, while the smaller are rounded or polygonal, averaging about 0.005 mm. in diameter. The smaller granules are grouped irregularly in and around the larger, there being six to ten of the former to one of the latter. The larger granules are, however, the most distinctly characteristic, and are usually readily recognized in a mixture, not only by their shape, but by reason of the concentric rings with which they are provided, and which are generally but not always apparent.

*Barley Starch* (Fig. 124, Pl. I).—This much resembles wheat, in that it has two sizes of granules, but both sizes are respectively smaller than those of wheat, though present in about the same proportion. The larger lenticular disk-like granules vary from 0.013 mm. to 0.035 mm. in diameter, while the smaller average 0.003 mm. The concentric rings are less apparent in the barley than in the wheat.

*Rye Starch* (Fig. 148, Pl. VII) has also two sizes of granules, but the larger vary from 0.025 mm. to over 0.05 mm. in diameter, and are considerably larger than the corresponding wheat granules. The smaller granules average about 0.004 mm. in diameter. As in the case of wheat and barley, the larger granules are lenticular, while the smaller are rounded or polygonal. The concentric rings are usually indistinct in the large granules, and many of these show cross-shaped rifts in the center.

*Corn Starch* (Fig. 133, Pl. IV).—This starch is a common adulterant of spices, cocoa, and other foods. It is placed in a series of four cereal starches whose granules are polygonal, and all of which show more or less tendency to arrange themselves in close contact side by side in masses suggestive of a tessellated or mosaic floor. Arranged in order of the size of their grains, these starches are: Corn, buckwheat, oats, and rice. Corn starch granules tend toward the hexagonal in shape, varying from 0.007 mm. to 0.035 mm. in diameter, and having very marked rifted hila. They are most readily recognized in any mixture, and from their size are readily distinguishable from the other polygonal starches, which never reach 0.017 mm. in diameter.

*Buckwheat Starch* (Fig. 128, Pl. II, and Fig. 129, Pl. III).—This is a very common adulterant of many spices, especially pepper, which, as shown in Fig. 256, Pl. XXXIV, it much resembles in the manner in which its masses of granules group themselves, conforming to the shape of the cells. The individual granules are commonly 0.006 mm. to 0.012 mm. in diameter. Curious rod-shaped aggregates of two to four individuals are of frequent occurrence.



*Oat Starch* (Fig. 139, Pl. V).—The granules of this starch vary from 0.002 mm. to 0.012 mm. in diameter, and are polygonal, or less often rounded or spindle-shaped in form. They have no rings or hila, and arrange themselves in rounded aggregates of from two to many granules that at first sight might be mistaken for large grains; careful examination, however, shows the dividing lines.

*Rice Starch* (Fig. 143, Pl. VI).—The granules of rice starch resemble closely those of oats both in form and size, but spindle-shaped forms are not present. As in the case of oats, the granules are often united to form rounded aggregates.

*Starches of the Pea and Bean*.—The starches of these legumes much resemble each other, and are with difficulty distinguished one from the other (see Fig. 164, Pl. XI, and Fig. 154, Pl. IX). The granules are more nearly oval than most other starches, and have both concentric rings and elongated hila. The granules of the pea show a less distinct hilum than those of the bean, and some of them are irregularly swollen. Both peas and beans roasted are commonly used as adulterants of coffee.

*Arrowroot*.—There are many varieties of arrowroot, including Jamaica, Bermuda, East Indian, Australian, and others, all having certain variations in form and size, but resembling each other in a general way. Fig. 167, Pl. XII, shows the Bermuda arrowroot, the granules of which are somewhat egg-shaped, being usually smaller at one end than the other, and having rifted hila near the small end.

*Potato Starch* (Fig. 165, Pl. XII).—This starch has large, irregularly oval granules, with very apparent hila situated eccentrically near one end, and with rings around the hilum. The granules are about 0.07 mm. in large diameter. Fig. 134, Pl. IV, and Fig. 166, Pl. XII, show corn and potato starch when viewed with polarized light with crossed Nicol prisms, the specimens being mounted in Canada balsam.

*Tapioca Starch*.—The granules of this starch, as shown in Fig. 168, Pl. XII, are more uniform in size throughout than those already described, averaging about 0.018 mm. in diameter, and being quite smoothly circular, without concentric rings, but having a distinctly dotted hilum in the center. Many of the grains are cup-shaped, as if a segment of the circle had been removed.

*Sago Starch* (Fig. 172, Pl. XIII).—The granules of sago starch vary much in size, and might be called irregularly ellipsoidal in shape with one or more truncated surfaces. Some of them have indistinct concentric



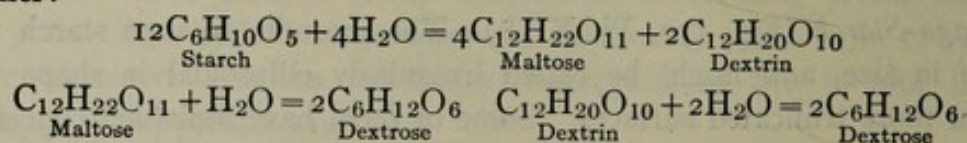
rings, and in some, but not all, a hilum is apparent, usually near one end of the granule.

**Microscopical Appearances of Starches with Polarized Light.**—With polarized light starch granules show dark crosses, the point of intersection being at the hilum (Fig. 166, Pl. XII). These crosses vary in distinctness with the variety. Certain of the starches show a play of colors with polarized light and a selenite plate, especially those whose granules have some sort of hilum. This is particularly striking in such starches as corn, tapioca, potato, and arrowroot. Blyth has made the phenomenon a means of classification of the starches, but the writer considers their appearance with ordinary light sufficient for identification. Canada balsam is the best mountant for examination in polarized light.

**Estimation of Starch.**—*Direct Acid Conversion.*—By this method the hemicellulose, if present, or such of the carbohydrates as are capable of being converted to sugar, are reckoned in with the starch. Where little or none of the insoluble carbohydrates other than starch are present, as for instance in the case of commercial starches, this method is sufficiently accurate.

Exhaust 3 grams of the finely divided substance on a fine but rapidly acting filter with ether by washing with 5 successive portions of 10 cc. each, and wash the residue first with 150 cc. of 10% alcohol and then with a little strong alcohol. Transfer by washing to a flask with 200 cc. of water and 20 cc. of hydrochloric acid (specific gravity 1.125), connect with a reflux condenser, and heat the flask in boiling water for 2½ hours. Cool, and carefully neutralize with sodium hydroxide, clarifying if necessary with alumina cream. Mix well, make up the volume to 500 cc., filter, and determine the dextrose in an aliquot part of the filtrate by any of the methods for dextrose. Convert dextrose to starch by the factor 0.9.

*Diastase Method.*—By this method the hemicellulose is not converted, only the starch being acted upon. Hence for exact work in the presence of other insoluble carbohydrates this method is to be recommended. Under the action of diastase, starch is first converted into maltose and dextrin, and finally into dextrose, in somewhat the following manner:





Exhaust 3 grams of the material, ground to an impalpable powder, with ether and alcohol as in the acid conversion method, wash the residue into a beaker with 50 cc. of water and immerse in a boiling water-bath. Keep in the bath for 15 minutes or until completely gelatinized, stirring constantly, and cool to 55° C. Add 20 cc. of malt extract and digest at 55° C. one hour. Heat again to boiling, boil for 15 minutes, replacing the water lost by evaporation, cool to 55° C., and digest as before with 20 cc. of malt extract for one hour or until the residue treated with iodine solution shows no starch under the microscope. Cool, make up to 250 cc., filter, pipette 200 cc. into a flask, add 20 cc. of hydrochloric acid (specific gravity 1.125) and proceed as in the acid conversion method. Correct for the copper-reducing power of the malt extract as below.

*Preparation of Malt Extract.*—Digest at room temperature 10 grams of freshly pulverized malt with 200 cc. of water for 2 to 3 hours with occasional shaking and filter. Determine the amount of dextrose in a given volume of the extract after heating with acid, etc., as in the actual analysis and make the proper correction.

*Use of "Animal Diastase."*—Pancreatin and similar powdered preparations, such as "vera diastase" and "panase," obtained from the pancreas of cattle and hogs, are preferable to diastase as starch-converting reagents, since, as a rule, they have no copper-reducing power, thus obviating a correction.

Use instead of the malt extract the same amount, viz., 20 cc., of a 0.5% aqueous solution of powdered U. S. P. pancreatin in starch determinations as above described.

*Determination of Sugars in Grain and Cereal Products.*—*Method of Bryan, Given and Straughn.\**—Place 12 grams of the material in a 300-cc. graduated flask (if acid add 1-3 grams of precipitated calcium carbonate), add 150 cc. of 50% (by vol.) alcohol (carefully neutralized), mix and boil on a steam bath under a reflux condenser for one hour. Cool, and if desired allow to stand overnight. Make up to volume with neutral 95% alcohol, mix, allow to settle, pipette 200 cc. into a beaker, and evaporate on the steam bath to 20-30 cc. Transfer to a 100-cc. graduated flask with water, add enough saturated normal lead acetate solution to produce a flocculent precipitate, and allow to stand 15 minutes or if desired overnight. Make up to the mark with water and pass through a folded filter, saving all the filtrate. Precipitate the lead with anhydrous sodium

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\* U. S. Dept. of Agric., Bur. of Chem., Circ. 71.



carbonate, allow to stand 15 minutes and pour onto an ashless filter. Dilute 25 cc. of this clear filtrate with 25 cc. of water and determine reducing sugars by the Munson and Walker method (p. 622).

In a 100 cc. graduated flask place 50 cc. of the same filtrate, neutralize to litmus paper with acetic acid, add 5 cc. of concentrated hydrochloric acid, and let stand overnight (or if desired 48 hours) for inversion. Neutralize in a large beaker with anhydrous sodium carbonate; return to the 100 cc. flask and make up to the mark. Filter and determine total sugars as invert in 50 cc. of the filtrate by the Munson and Walker method.

Multiply the difference between the percentages of invert sugar before and after inversion by 0.95, thus obtaining the per cent of sucrose. Correct the percentages of both sucrose and reducing sugars for the volume of the alcohol precipitate by multiplying by 0.97.

**Cellulose** forms the framework of all vegetable organisms, being next to water, the most abundant substance in the vegetable kingdom. Pure cellulose is white, translucent, and of fibrous or silky texture. It is insoluble in water, alcohol, and ether, but dissolves readily in an ammoniacal solution of cupric hydroxide known as Schweitzer's Reagent or "cuprammonia," (p. 80). Cellulose turns violet when treated with chloriodide of zinc, and blue when treated with sulphuric acid and iodine in potassium iodide (p. 78).

The "crude fiber," as determined in foods, being the portion that resists the action of hot dilute acid and alkali, is composed largely of cellulose.

**The Pentosans** are amorphous, insoluble in water, but soluble in dilute alkali, and are converted by boiling with dilute acids into so-called pentose sugars, the best known of which are xylose and arabinose, corresponding to the pentosans xylan and araban respectively. Hemicellulose is the more appropriate generic term for the insoluble carbohydrates capable of hydrolysis by acids to sugars, inasmuch as there are insoluble bodies besides the pentosans that may thus be converted into sugar, such as the hexosans, hydrolyzed by acid to hexose sugars, mannose, galactose, etc. Since the greater portion of these insoluble hydrolyzable carbohydrates are pentosans, it is simpler to calculate them all as such.

**Determination of Pentosans.**—Pentosans are determined either by hydrolyzing to reducing sugar, and estimating the latter as described on



page 304 (Sone's method) or by calculation from the furfural\* yielded on distillation with hydrochloric acid, as carried out in the provisional method of the A. O. A. C.† as follows:

Place 3 grams of the material in a flask together with 100 cc. of 12% hydrochloric acid (specific gravity 1.06) and several pieces of recently heated pumice stone, connect with a condenser and heat on a wire gauze, rather gently at first, using a gauze top to distribute the flame so as to distil over 30 cc. in about ten minutes and passing the distillate through a small filter. Replace the 30 cc. driven over with a like amount of the 12% acid added through a separatory funnel in such a manner as to wash down the particles on the sides of the flask and continue the process until the distillate amounts to 360 cc. To the distillate add gradually a quantity of phloroglucinol (free from diresorcin) dissolved in 12% hydrochloric acid, about double that of the furfural expected. The solution first turns yellow, then green, and soon an amorphous greenish precipitate appears, which rapidly grows darker, finally becoming almost black. Make the solution up to 400 cc. with 12% hydrochloric acid and allow to stand overnight.

Filter the amorphous black precipitate on a Gooch crucible, wash with 150 cc. of water, keeping the precipitate covered with the liquid until the last portion has run through, dry for four hours at the temperature of boiling water, cool in a weighing bottle and weigh. Calculate by Kröber's formulæ as follows:

(a) For weight of phloroglucide "a" under 0.03 gram:

$$\text{Furfural} = (a + 0.0052) \times 0.5170.$$

$$\text{Pentoses} = (a + 0.0052) \times 1.0170.$$

$$\text{Pentosans} = (a + 0.0052) \times 0.8949.$$

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\* Furfural or furfuraldehyde ( $\text{C}_5\text{H}_4\text{O}_2$ ) is the aldehyde of pyromucic acid. It is a colorless liquid, having an odor suggestive of cassia. Its boiling-point is  $162^\circ$  and its specific gravity 1.164. It is sparingly soluble in water and readily soluble in alcohol. Nearly half the tissue of ordinary bran, exclusive of proteins and starch, yields furfural on distillation with acid.

† U. S. Dept. of Agric., Bur. of Chem., Bul. 107 (rev.), p. 54.



(b) For weight of phloroglucide "a" over 0.300 gram.

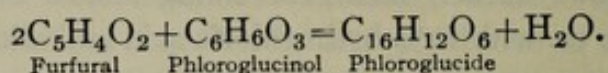
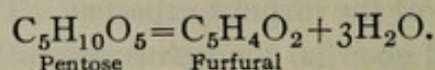
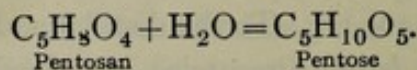
$$\text{Furfural} = (a + 0.0052) \times 0.5180.$$

$$\text{Pentoses} = (a + 0.0052) \times 1.0026.$$

$$\text{Pentosans} = (a + 0.0052) \times 0.8824.$$

For weight of phloroglucide "a" from 0.03 to 0.300 gram use Kröber's table (pp. 297-303) to calculate the weight of pentoses (arabinose, xylose), and pentosans (araban, xylan).

The reactions that take place are thought to be somewhat as follows:



The theoretical yield of phloroglucide should be 2.22 parts to one of furfural, but in practice this is never obtained. The varying factors for calculation as above given are based on experiment.

The phloroglucinol used should be free from diresorcin. To test for the latter, dissolve the reagent in acetic anhydride, heat nearly to boiling, and add a few drops of concentrated sulphuric acid. If more than a faint violet color is produced, the phloroglucinol should be purified as follows:

Heat in a beaker about 300 cc. of hydrochloric acid (sp. gr. 1.06) and 11 grams of commercial phloroglucinol, added in small quantities at a time, stirring constantly until it has almost dissolved. Some impurities may resist solution, but it is unnecessary to dissolve them. Pour the hot solution into a sufficient quantity of the same hydrochloric acid (cold) to make the volume 1500 cc. Allow it to stand at least overnight—better several days—to allow the diresorcin to crystallize out, and filter immediately before using. The solution may turn yellow, but this does not interfere with its usefulness. In using it, add the volume containing the required amount to the distillate.



KRÖBER'S TABLE FOR DETERMINATION OF PENTOSEs AND PENTOSANS  
FROM PHLOROGLUCID.

<sup>1</sup> Phloroglucid	<sup>2</sup> Furfural.	<sup>3</sup> Arabinose.	<sup>4</sup> Araban.	<sup>5</sup> Xylose.	<sup>6</sup> Xylan.	<sup>7</sup> Pentose.	<sup>8</sup> Pentosan.
0.030	0.0182	0.0391	0.0344	0.0324	0.0285	0.0358	0.0315
.031	.0188	.0402	.0354	.0333	.0293	.0368	.0324
.032	.0193	.0413	.0363	.0342	.0301	.0378	.0333
.033	.0198	.0424	.0373	.0352	.0309	.0388	.0341
.034	.0203	.0435	.0383	.0361	.0317	.0398	.0350
.035	.0209	.0446	.0393	.0370	.0326	.0408	.0359
.036	.0214	.0457	.0402	.0379	.0334	.0418	.0368
.037	.0219	.0468	.0412	.0388	.0342	.0428	.0377
.038	.0224	.0479	.0422	.0398	.0350	.0439	.0386
.039	.0229	.0490	.0431	.0407	.0358	.0449	.0395
.040	.0235	.0501	.0441	.0416	.0366	.0459	.0404
.041	.0240	.0512	.0451	.0425	.0374	.0469	.0413
.042	.0245	.0523	.0460	.0434	.0382	.0479	.0422
.043	.0250	.0534	.0470	.0443	.0390	.0489	.0431
.044	.0255	.0545	.0480	.0452	.0398	.0499	.0440
.045	.0260	.0556	.0490	.0462	.0406	.0509	.0448
.046	.0266	.0567	.0499	.0471	.0414	.0519	.0457
.047	.0271	.0578	.0509	.0480	.0422	.0529	.0466
.048	.0276	.0589	.0519	.0489	.0430	.0539	.0475
.049	.0281	.0600	.0528	.0498	.0438	.0549	.0484
.050	.0286	.0611	.0538	.0507	.0446	.0559	.0492
.051	.0292	.0622	.0548	.0516	.0454	.0569	.0501
.052	.0297	.0633	.0557	.0525	.0462	.0579	.0510
.053	.0302	.0644	.0567	.0534	.0470	.0589	.0519
.054	.0307	.0655	.0576	.0543	.0478	.0599	.0528
.055	.0312	.0666	.0586	.0553	.0486	.0610	.0537
.056	.0318	.0677	.0596	.0562	.0494	.0620	.0546
.057	.0323	.0688	.0605	.0571	.0502	.0630	.0555
.058	.0328	.0699	.0615	.0580	.0510	.0640	.0564
.059	.0333	.0710	.0624	.0589	.0518	.0650	.0573
.060	.0338	.0721	.0634	.0598	.0526	.0660	.0581
.061	.0344	.0732	.0644	.0607	.0534	.0670	.0590
.062	.0349	.0743	.0653	.0616	.0542	.0680	.0599
.063	.0354	.0754	.0663	.0626	.0550	.0690	.0608
.064	.0359	.0765	.0673	.0635	.0558	.0700	.0617
.065	.0364	.0776	.0683	.0644	.0567	.0710	.0625
.066	.0370	.0787	.0692	.0653	.0575	.0720	.0634
.067	.0375	.0798	.0702	.0662	.0583	.0730	.0643
.068	.0380	.0809	.0712	.0672	.0591	.0741	.0652
.069	.0385	.0820	.0721	.0681	.0599	.0751	.0661



KRÖBER'S TABLE FOR DETERMINATION OF PENTOSE AND PENTOSANS  
 FROM PHLOROGLUCID—*Continued.*

<sup>1</sup> Phloroglucid	<sup>2</sup> Furfural.	<sup>3</sup> Arabinose.	<sup>4</sup> Araban.	<sup>5</sup> Xylose.	<sup>6</sup> Xylan.	<sup>7</sup> Pentose.	<sup>8</sup> Pentosan.
.070	.0390	.0831	.0731	.0690	.0607	.0761	.0670
.071	.0396	.0842	.0741	.0699	.0615	.0771	.0679
.072	.0401	.0853	.0750	.0708	.0623	.0781	.0688
.073	.0406	.0864	.0760	.0717	.0631	.0791	.0697
.074	.0411	.0875	.0770	.0726	.0639	.0801	.0706
.075	.0416	.0886	.0780	.0736	.0647	.0811	.0714
.076	.0422	.0897	.0789	.0745	.0655	.0821	.0722
.077	.0427	.0908	.0799	.0754	.0663	.0831	.0731
.078	.0432	.0919	.0809	.0763	.0671	.0841	.0740
.079	.0437	.0930	.0818	.0772	.0679	.0851	.0749
.080	.0442	.0941	.0828	.0781	.0687	.0861	.0758
.081	.0448	.0952	.0838	.0790	.0695	.0871	.0767
.082	.0453	.0963	.0847	.0799	.0703	.0881	.0776
.083	.0458	.0974	.0857	.0808	.0711	.0891	.0785
.084	.0463	.0985	.0867	.0817	.0719	.0901	.0794
.085	.0468	.0996	.0877	.0827	.0727	.0912	.0803
.086	.0474	.1007	.0886	.0836	.0735	.0922	.0812
.087	.0479	.1018	.0896	.0845	.0743	.0932	.0821
.088	.0484	.1029	.0906	.0854	.0751	.0942	.0830
.089	.0489	.1040	.0915	.0863	.0759	.0952	.0838
.090	.0494	.1051	.0925	.0872	.0767	.0962	.0847
.091	.0499	.1062	.0935	.0881	.0775	.0972	.0856
.092	.0505	.1073	.0944	.0890	.0783	.0982	.0865
.093	.0510	.1084	.0954	.0900	.0791	.0992	.0874
.094	.0515	.1095	.0964	.0909	.0800	.1002	.0883
.095	.0520	.1106	.0974	.0918	.0808	.1012	.0891
.096	.0525	.1117	.0983	.0927	.0816	.1022	.0899
.097	.0531	.1128	.0993	.0936	.0824	.1032	.0908
.098	.0536	.1139	.1003	.0946	.0832	.1043	.0917
.099	.0541	.1150	.1012	.0955	.0840	.1053	.0926
.100	.0546	.1161	.1022	.0964	.0848	.1063	.0935
.101	.0551	.1171	.1032	.0973	.0856	.1073	.0944
.102	.0557	.1182	.1041	.0982	.0864	.1083	.0953
.103	.0562	.1193	.1051	.0991	.0872	.1093	.0962
.104	.0567	.1204	.1060	.1000	.0880	.1103	.0971
.105	.0572	.1215	.1070	.1010	.0888	.1113	.0976
.106	.0577	.1226	.1080	.1019	.0896	.1123	.0988
.107	.0582	.1237	.1089	.1028	.0904	.1133	.0997
.108	.0588	.1248	.1099	.1037	.0912	.1143	.1006
.109	.0593	.1259	.1108	.1046	.0920	.1153	.1015



KRÖBER'S TABLE FOR DETERMINATION OF PENTOSE AND PENTOSANS  
 FROM PHLOROGLUCID—(Continued).

<sup>1</sup> Phloroglucid	<sup>2</sup> Furfural.	<sup>3</sup> Arabinose.	<sup>4</sup> Araban.	<sup>5</sup> Xylose.	<sup>6</sup> Xylan.	<sup>7</sup> Pentose.	<sup>8</sup> Pentosan.
0.110	0.0598	0.1270	0.1118	0.1055	0.0928	0.1163	0.1023
.111	.0603	.1281	.1128	.1064	.0936	.1173	.1032
.112	.0608	.1292	.1137	.1073	.0944	.1183	.1041
.113	.0614	.1303	.1147	.1082	.0952	.1193	.1050
.114	.0619	.1314	.1156	.1091	.0960	.1203	.1059
.115	.0624	.1325	.1166	.1101	.0968	.1213	.1067
.116	.0629	.1336	.1176	.1110	.0976	.1223	.1076
.117	.0634	.1347	.1185	.1119	.0984	.1233	.1085
.118	.0640	.1358	.1195	.1128	.0992	.1243	.1094
.119	.0645	.1369	.1204	.1137	.1000	.1253	.1103
.120	.0650	.1380	.1214	.1146	.1008	.1263	.1111
.121	.0655	.1391	.1224	.1155	.1016	.1273	.1120
.122	.0660	.1402	.1233	.1164	.1024	.1283	.1129
.123	.0665	.1413	.1243	.1173	.1032	.1293	.1138
.124	.0671	.1424	.1253	.1182	.1040	.1303	.1147
.125	.0676	.1435	.1263	.1192	.1049	.1314	.1156
.126	.0681	.1446	.1272	.1201	.1057	.1324	.1165
.127	.0686	.1457	.1282	.1210	.1065	.1334	.1174
.128	.0691	.1468	.1292	.1219	.1073	.1344	.1183
.129	.0697	.1479	.1301	.1228	.1081	.1354	.1192
.130	.0702	.1490	.1311	.1237	.1089	.1364	.1201
.131	.0707	.1501	.1321	.1246	.1097	.1374	.1210
.132	.0712	.1512	.1330	.1255	.1105	.1384	.1219
.133	.0717	.1523	.1340	.1264	.1113	.1394	.1227
.134	.0723	.1534	.1350	.1273	.1121	.1404	.1236
.135	.0728	.1545	.1360	.1283	.1129	.1414	.1244
.136	.0733	.1556	.1369	.1292	.1137	.1424	.1253
.137	.0738	.1567	.1379	.1301	.1145	.1434	.1262
.138	.0743	.1578	.1389	.1310	.1153	.1444	.1271
.139	.0748	.1589	.1398	.1319	.1161	.1454	.1280
.140	.0754	.1600	.1408	.1328	.1169	.1464	.1288
.141	.0759	.1611	.1418	.1337	.1177	.1474	.1297
.142	.0764	.1622	.1427	.1346	.1185	.1484	.1306
.143	.0769	.1633	.1437	.1355	.1193	.1494	.1315
.144	.0774	.1644	.1447	.1364	.1201	.1504	.1324
.145	.0780	.1655	.1457	.1374	.1209	.1515	.1333
.146	.0785	.1666	.1466	.1383	.1217	.1525	.1342
.147	.0790	.1677	.1476	.1392	.1225	.1535	.1351
.148	.0795	.1688	.1486	.1401	.1233	.1545	.1360
.149	.0800	.1699	.1495	.1410	.1241	.1555	.1369



KRÖBER'S TABLE FOR DETERMINATION OF PENTOSE AND PENTOSANS  
 FROM PHLOROGLUCID—(Continued).

<sup>1</sup> Phloroglucid	<sup>2</sup> Furfural.	<sup>3</sup> Arabinose.	<sup>4</sup> Araban.	<sup>5</sup> Xylose.	<sup>6</sup> Xylan.	<sup>7</sup> Pentose.	<sup>8</sup> Pentosan.
0.150	0.0805	0.1710	0.1505	0.1419	0.1249	0.1565	0.1377
.151	.0811	.1721	.1515	.1428	.1257	.1575	.1386
.152	.0816	.1732	.1524	.1437	.1265	.1585	.1395
.153	.0821	.1743	.1534	.1446	.1273	.1595	.1404
.154	.0826	.1754	.1544	.1455	.1281	.1605	.1413
.155	.0831	.1765	.1554	.1465	.1289	.1615	.1421
.156	.0837	.1776	.1563	.1474	.1297	.1625	.1430
.157	.0842	.1787	.1573	.1483	.1305	.1635	.1439
.158	.0847	.1798	.1583	.1492	.1313	.1645	.1448
.159	.0852	.1809	.1592	.1501	.1321	.1655	.1457
.160	.0857	.1820	.1602	.1510	.1329	.1665	.1465
.161	.0863	.1831	.1612	.1519	.1337	.1675	.1474
.162	.0868	.1842	.1621	.1528	.1345	.1685	.1483
.163	.0873	.1853	.1631	.1537	.1353	.1695	.1492
.164	.0878	.1864	.1640	.1546	.1361	.1705	.1501
.165	.0883	.1875	.1650	.1556	.1369	.1716	.1510
.166	.0888	.1886	.1660	.1565	.1377	.1726	.1519
.167	.0894	.1897	.1669	.1574	.1385	.1736	.1528
.168	.0899	.1908	.1679	.1583	.1393	.1746	.1537
.169	.0904	.1919	.1688	.1592	.1401	.1756	.1546
.170	.0909	.1930	.1698	.1601	.1409	.1766	.1554
.171	.0914	.1941	.1708	.1610	.1417	.1776	.1563
.172	.0920	.1952	.1717	.1619	.1425	.1786	.1572
.173	.0925	.1963	.1727	.1628	.1433	.1796	.1581
.174	.0930	.1974	.1736	.1637	.1441	.1806	.1590
.175	.0935	.1985	.1746	.1647	.1449	.1816	.1598
.176	.0940	.1996	.1756	.1656	.1457	.1826	.1607
.177	.0946	.2007	.1765	.1665	.1465	.1836	.1616
.178	.0951	.2018	.1775	.1674	.1473	.1846	.1625
.179	.0956	.2029	.1784	.1683	.1481	.1856	.1634
.180	.0961	.2039	.1794	.1692	.1489	.1866	.1642
.181	.0966	.2050	.1804	.1701	.1497	.1876	.1651
.182	.0971	.2061	.1813	.1710	.1505	.1886	.1660
.183	.0977	.2072	.1823	.1719	.1513	.1896	.1669
.184	.0982	.2082	.1832	.1728	.1521	.1906	.1678
.185	.0987	.2093	.1842	.1738	.1529	.1916	.1686
.186	.0992	.2104	.1851	.1747	.1537	.1926	.1695
.187	.0997	.2115	.1861	.1756	.1545	.1936	.1704
.188	.1003	.2126	.1870	.1765	.1553	.1946	.1712
.189	.1008	.2136	.1880	.1774	.1561	.1955	.1721



KRÖBER'S TABLE FOR DETERMINATION OF PENTOSE AND PENTOSANS  
 FROM PHLOROGLUCID—(Continued).

1 Phloroglucid	2 Furfural	3 Arabinose	4 Araban.	5 Xylose	6 Xylan.	7 Pentose	8 Pentosan.
.190	0.1013	0.2147	0.1889	0.1783	0.1569	0.1965	0.1729
.191	.1018	.2158	.1899	.1792	.1577	.1975	.1738
.192	.1023	.2168	.1908	.1801	.1585	.1985	.1747
.193	.1028	.2179	.1918	.1810	.1593	.1995	.1756
.194	.1034	.2190	.1927	.1819	.1601	.2005	.1764
.195	.1039	.2201	.1937	.1829	.1609	.2015	.1773
.196	.1044	.2212	.1946	.1838	.1617	.2025	.1782
.197	.1049	.2222	.1956	.1847	.1625	.2035	.1791
.198	.1054	.2233	.1965	.1856	.1633	.2045	.1800
.199	.1059	.2244	.1975	.1865	.1641	.2055	.1808
.200	.1065	.2255	.1984	.1874	.1649	.2065	.1817
.201	.1070	.2266	.1994	.1883	.1657	.2075	.1826
.202	.1075	.2276	.2003	.1892	.1665	.2085	.1835
.203	.1080	.2287	.2013	.1901	.1673	.2095	.1844
.204	.1085	.2298	.2022	.1910	.1681	.2105	.1853
.205	.1090	.2309	.2032	.1920	.1689	.2115	.1861
.206	.1096	.2320	.2041	.1929	.1697	.2125	.1869
.207	.1101	.2330	.2051	.1938	.1705	.2134	.1878
.208	.1106	.2341	.2060	.1947	.1713	.2144	.1887
.209	.1111	.2352	.2069	.1956	.1721	.2154	.1896
.210	.1116	.2363	.2079	.1965	.1729	.2164	.1904
.211	.1121	.2374	.2089	.1975	.1737	.2174	.1913
.212	.1127	.2384	.2098	.1984	.1745	.2184	.1922
.213	.1132	.2395	.2108	.1993	.1753	.2194	.1931
.214	.1137	.2406	.2117	.2002	.1761	.2204	.1940
.215	.1142	.2417	.2127	.2011	.1770	.2214	.1948
.216	.1147	.2428	.2136	.2020	.1778	.2224	.1957
.217	.1152	.2438	.2146	.2029	.1786	.2234	.1966
.218	.1158	.2449	.2155	.2038	.1794	.2244	.1974
.219	.1163	.2460	.2165	.2047	.1802	.2254	.1983
.220	.1168	.2471	.2174	.2057	.1810	.2264	.1992
.221	.1173	.2482	.2184	.2066	.1818	.2274	.2001
.222	.1178	.2492	.2193	.2075	.1826	.2284	.2010
.223	.1183	.2503	.2203	.2084	.1834	.2294	.2019
.224	.1189	.2514	.2212	.2093	.1842	.2304	.2028
.225	.1194	.2525	.2222	.2102	.1850	.2314	.2037
.226	.1199	.2536	.2232	.2111	.1858	.2324	.2046
.227	.1204	.2546	.2241	.2121	.1866	.2334	.2054
.228	.1209	.2557	.2251	.2130	.1874	.2344	.2063
.229	.1214	.2568	.2260	.2139	.1882	.2354	.2072



KRÖBER'S TABLE FOR DETERMINATION OF PENTOSE AND PENTOSANS  
 FROM PHLOROGLUCID—(Continued).

<sup>1</sup> Phloroglucid	<sup>2</sup> Furfural.	<sup>3</sup> Arabinose.	<sup>4</sup> Araban.	<sup>5</sup> Xylose.	<sup>6</sup> Xylan.	<sup>7</sup> Pentose.	<sup>8</sup> Pentosan.
0.230	0.1220	0.2579	0.2270	0.2148	0.1890	0.2364	0.2081
.231	.1225	.2590	.2280	.2157	.1898	.2374	.2089
.232	.1230	.2600	.2289	.2166	.1906	.2383	.2097
.233	.1235	.2611	.2299	.2175	.1914	.2393	.2106
.234	.1240	.2622	.2308	.2184	.1922	.2403	.2115
.235	.1245	.2633	.2318	.2193	.1930	.2413	.2124
.236	.1251	.2644	.2327	.2202	.1938	.2423	.2132
.237	.1256	.2654	.2337	.2211	.1946	.2433	.2141
.238	.1261	.2665	.2346	.2220	.1954	.2443	.2150
.239	.1266	.2676	.2356	.2229	.1962	.2453	.2159
.240	.1271	.2687	.2365	.2239	.1970	.2463	.2168
.241	.1276	.2698	.2375	.2248	.1978	.2473	.2176
.242	.1281	.2708	.2384	.2257	.1986	.2483	.2185
.243	.1287	.2719	.2394	.2266	.1994	.2493	.2194
.244	.1292	.2730	.2403	.2275	.2002	.2503	.2203
.245	.1297	.2741	.2413	.2284	.2010	.2513	.2212
.246	.1302	.2752	.2422	.2293	.2018	.2523	.2220
.247	.1307	.2762	.2432	.2302	.2026	.2533	.2229
.248	.1312	.2773	.2441	.2311	.2034	.2543	.2238
.249	.1318	.2784	.2451	.2320	.2042	.2553	.2247
.250	.1323	.2795	.2460	.2330	.2050	.2563	.2256
.251	.1328	.2806	.2470	.2339	.2058	.2573	.2264
.252	.1333	.2816	.2479	.2348	.2066	.2582	.2272
.253	.1338	.2827	.2489	.2357	.2074	.2592	.2281
.254	.1343	.2838	.2498	.2366	.2082	.2602	.2290
.255	.1349	.2849	.2508	.2375	.2090	.2612	.2299
.256	.1354	.2860	.2517	.2384	.2098	.2622	.2307
.257	.1359	.2870	.2526	.2393	.2106	.2632	.2316
.258	.1364	.2881	.2536	.2402	.2114	.2642	.2325
.259	.1369	.2892	.2545	.2411	.2122	.2652	.2334
.260	.1374	.2903	.2555	.2420	.2130	.2662	.2343
.261	.1380	.2914	.2565	.2429	.2138	.2672	.2351
.262	.1385	.2924	.2574	.2438	.2146	.2681	.2359
.263	.1390	.2935	.2584	.2447	.2154	.2691	.2368
.264	.1395	.2946	.2593	.2456	.2162	.2701	.2377
.265	.1400	.2957	.2603	.2465	.2170	.2711	.2385
.266	.1405	.2968	.2612	.2474	.2178	.2721	.2394
.267	.1411	.2978	.2622	.2483	.2186	.2731	.2403
.268	.1416	.2989	.2631	.2492	.2194	.2741	.2412
.269	.1421	.3000	.2641	.2502	.2202	.2751	.2421



KRÖBER'S TABLE FOR DETERMINATION OF PENTOSE AND PENTOSANS  
FROM PHLOROGLUCID—(Concluded).

1 Phloroglucid	2 Furfural.	3 Arabinose.	4 Araban.	5 Xylose.	6 Xylan.	7 Pentose.	8 Pentosan.
0.270	0.1426	0.3011	0.2650	0.2511	0.2210	0.2761	0.2429
.271	.1431	.3022	.2660	.2520	.2218	.2771	.2438
.272	.1436	.3032	.2669	.2529	.2226	.2781	.2447
.273	.1442	.3043	.2679	.2538	.2234	.2791	.2456
.274	.1447	.3054	.2688	.2547	.2242	.2801	.2465
.275	.1452	.3065	.2698	.2556	.2250	.2811	.2473
.276	.1457	.3076	.2707	.2565	.2258	.2821	.2482
.277	.1462	.3086	.2717	.2574	.2266	.2830	.2490
.278	.1467	.3097	.2726	.2583	.2274	.2840	.2499
.279	.1473	.3108	.2736	.2592	.2282	.2850	.2508
.280	.1478	.3119	.2745	.2602	.2290	.2861	.2517
.281	.1483	.3130	.2755	.2611	.2298	.2871	.2526
.282	.1488	.3140	.2764	.2620	.2306	.2880	.2534
.283	.1493	.3151	.2774	.2629	.2314	.2890	.2543
.284	.1498	.3162	.2783	.2638	.2322	.2900	.2552
.285	.1504	.3173	.2793	.2647	.2330	.2910	.2561
.286	.1509	.3184	.2802	.2656	.2338	.2920	.2570
.287	.1514	.3194	.2812	.2665	.2346	.2930	.2578
.288	.1519	.3205	.2821	.2674	.2354	.2940	.2587
.289	.1524	.3216	.2831	.2683	.2362	.2950	.2596
.290	.1529	.3227	.2840	.2693	.2370	.2960	.2605
.291	.1535	.3238	.2850	.2702	.2378	.2970	.2614
.292	.1540	.3248	.2859	.2711	.2386	.2980	.2622
.293	.1545	.3259	.2868	.2720	.2394	.2990	.2631
.294	.1550	.3270	.2878	.2729	.2402	.3000	.2640
.295	.1555	.3281	.2887	.2738	.2410	.3010	.2649
.296	.1560	.3292	.2897	.2747	.2418	.3020	.2658
.297	.1566	.3302	.2906	.2756	.2426	.3030	.2666
.298	.1571	.3313	.2916	.2765	.2434	.3040	.2675
.299	.1576	.3324	.2925	.2774	.2442	.3050	.2684
.300	.1581	.3335	.2935	.2784	.2450	.3060	.2693



SEPARATION AND DETERMINATION OF THE VARIOUS CARBOHYDRATES  
OF CEREALS, ETC. STONE'S METHOD.

Stone has thus tabulated the results of a series of analyses of various samples of wheat, flour, corn, and bread, in which he has separated the principal carbohydrates.\*

PERCENTAGES OF VARIOUS CARBOHYDRATES IN CERTAIN FOODSTUFFS.

	Sucrose.	Invert Sugar.	Dextrin.	Soluble Starch.	Pentose.	Crude Fiber.
Whole wheat, I. ....	0.52	0.08	0.27	0.00	4.54	2.68
Whole wheat, II. ....	0.72	0.00	0.41	0.00	4.37	2.51
Wheat flour, I. ....	0.18	0.00	0.90	0.00	0.00	0.25
Wheat flour, II. ....	0.20	0.00	1.06	0.00	0.00	0.25
Corn. ....	9.27	0.00	0.32	0.00	5.14	1.99
Sugar-beet. ....	8.38	0.07	0.35	0.00	4.89	1.00
Bread (wheat, I). ....	0.05	0.32	0.68	1.37	4.16	2.70
Bread (wheat, II). ....	0.06	0.37	0.23	2.36	4.34	2.02
Bread (flour, I). ....	0.01	0.10	0.27	1.99	0.00	0.34
Bread (flour, II). ....	0.15	0.38	0.91	1.74	0.00	0.17
Corn cake (maize). ....	0.16	0.19	0.00	2.80	3.54	2.22

**Determination of Cane Sugar.**—100 grams of the finely ground material are extracted by boiling under a reflux condenser with 500 cc. of 95% alcohol for three hours, the alcoholic extract is filtered, evaporated nearly to dryness, and then taken up with a small amount of water, to separate the sugar from the oils and waxes dissolved by the alcohol. This aqueous solution is invariably dextro-rotary, and seldom contains any reducing sugar. If the latter is present, it is determined in an aliquot part of the aqueous solution with Fehling's solution, the result being calculated to dextrose. The remainder of the aqueous sugar solution, or the whole of it, if, as is almost always the case, dextrose is absent, is then inverted by heating with hydrochloric acid in the usual manner (page 611) and the sugar is estimated with Fehling's solution, calculating the result to sucrose (page 642).

**Determination of Dextrin.**—Digest the residue from the above alcoholic extraction from eighteen to twenty-four hours with 500 cc. of cold distilled water, shaking frequently. On filtering, a clear solution is ob-

\* Jour. Am. Chem. Soc., 19, 1897, p. 183, and U. S. Dept. of Agric., Off. of Exp. Sta., Bul. 34. The percentages of normal starch found by Stone are obviously erroneous, and are for this reason excluded from the table as here given.



tained, which should be tested with iodine for soluble starch. If the latter is not found (which is nearly always the case), the solution is concentrated to a small volume, avoiding a temperature higher than 80° to 90°, and this is boiled under a reflux condenser for two hours with one-tenth its volume of hydrochloric acid (specific gravity 1.125). Determine the dextrose by Fehling's solution and calculate to dextrin by the factor 0.9. Or, instead of submitting the concentrated aqueous extract to hydrolysis as above, the dextrin may be roughly determined gravimetrically therein by treating with several volumes of strong alcohol until no further precipitation is produced. The flocculent precipitate thus obtained is collected, dried, and weighed.

**Determination of Starch.**—Dry in an oven the residue from the preceding treatment and determine its quantitative relation to the original sample; 2 grams are then accurately weighed and subjected to the diastase method of starch determination (page 292).

**Determination of Pentosans and Hemicelluloses.**—The washed residue, left after filtering off the starch-containing solution from the process of heating with malt extract in the preceding starch determination, is boiled for an hour with 100 cc. of 1% hydrochloric acid, which converts all the pentosans into sugar. Filter, and wash the residue thoroughly, make up the solution to 200 cc., and determine the sugar with Fehling's solution, calculating the results for xylan, assuming that the chief sugar formed is xylose. The reducing power of xylose is assumed to be 4.61 milligrams for each cubic centimeter of Fehling's solution. If the volumetric Fehling method is used, 10 cc. of Fehling's solution are thus equivalent to 0.046 gram xylose.  $\text{Xylose} \times 0.88 = \text{xylan}$ .

**Crude Fiber (Cellulose, etc.).**—The residue from the last dilute acid hydrolysis is boiled with 200 cc. of 1.25% solution of sodium hydroxide for half an hour, filtered, dried, and weighed. It is then ignited, and the weight of the ash deducted from the first weight.

#### PROTEINS OF CEREALS AND VEGETABLES.

Different cereal and vegetable foods present considerable variations in the character and extent of their protein constituents, and by no means all of the common vegetable foods have been studied in detail.

Osborne, in connection with Voorhees and Chittenden, has made a careful study of the proteins of many of the cereals, of potatoes, and of peas. A brief outline only will be given in what follows of methods



for separation of the vegetable proteins. For fuller details the reader is referred to the work of Osborne *et al.* in the American Chemical Journal, Vols. 12, 14, and 15, and to the Journal of the American Chemical Society, Vols. 17, 18, 19, and 20.

**Proteins Soluble in Water and Dilute Salt Solution.**—By the action of various solvents it is possible to separate the different classes of proteins for examination or analysis. Thus water at first applied extracts certain of the soluble proteins, as does a weak salt solution. Osborne and Voorhees recommend the use of a 10% solution of sodium chloride as the first solvent to apply for separating vegetable proteins, shaking the finely ground material with twice its weight of the salt solution. The salt solution, after filtering, is then subjected to dialysis, the protein matter thus separated out being a globulin, while that not precipitated on dialysis is assumed as the protein matter of the substance soluble in water. Two albumins and a proteose are found in wheat to be thus soluble in water.

If the proteins soluble in salt solution are to have their total nitrogen determined, they are completely precipitated from the solution by saturating with zinc or ammonium sulphate.

There are thus two classes of proteins soluble in 10% salt solution: (a) globulins, insoluble in water alone, and (b) albumins and proteoses, which are soluble in water.

**Separation of Albumins, Proteoses, and Globulins.**—Starting with the aqueous solution containing the albumins and proteoses, if present, the former are best separated according to Osborne and Voorhees by fractional coagulation, effected by heating at different temperatures, those that precipitate out at a temperature under 65° being first filtered out, and the filtrate submitted to a higher temperature not exceeding 85°. The two portions thus separated may be collected in filters, and their nitrogen separately determined.

The proteose may be precipitated from the filtrate by saturating with ground salt, or by adding, first salt to the extent of 20%, and finally acetic acid.

The globulins, precipitated in the original 10% salt solution by the process of dialysis as described, may themselves be separated by employing salt solution of varying strength as solvents.\*

**Proteins Soluble in Dilute Alcohol, but Insoluble in Water.**—The residue from the treatment with 10% sodium chloride is digested with 75% alcohol at about 46° C. for some time and filtered. The residue is further

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\* Am. Chem. Jour., 13, p. 464.



digested at about 60° with 75% alcohol three separate times. The evaporated filtrates contain the alcohol-soluble proteins. In this class are the hordein of barley, the gliadin of wheat and rye, and the zein of corn.

**Proteins Insoluble in Water, Salt Solution, and Dilute Alcohol.**—It is customary to determine the nitrogen in the final residue without further attempt to separate the remaining protein matter. It is, however, possible to further extract with alkaline and acid solvents, if desired, which process, however, changes the nature of the proteins from that in which they originally exist in the substance.

**Character and Amount of Proteins in Wheat.\***—The proteins of wheat, according to Osborne, are five in number, as follows:

	Amount Present, Per Cent.
Soluble in water:	{ Albumin (leucosin) . . . . . 0.3 to 0.4
	{ Proteose . . . . . 0.3
Soluble in 10 per cent NaCl:	Globulin (edestin) . . . . . 0.6 to 0.7
Soluble in dilute alcohol:	Gliadin . . . . . 4.25
Insoluble in above:	Glutenin . . . . . 4.00 to 4.5

The term *gluten* is applied to the protein content of wheat flour insoluble in water, the value of flour for baking bread depending on the amount present. Gluten contains the two definite proteins, *gliadin* and *glutenin*. Crude gluten, as obtained by washing the dough in the analytical process (page 331), is a complex mixture of many bodies, containing, besides the two proteins above named, small quantities of cellulose, mineral matter, lecithin, and starch.

**Separation and Determination of Wheat Proteins.**—*Teller's Method.*†—**Non-gluten Nitrogen.**—Two grams of the finely divided sample are mixed with about 15 cc. of 1% salt solution in a 250-cc. flask. The flask is shaken at intervals of ten minutes during one hour, after which it is filled to the mark with the salt solution and allowed to stand two hours. The supernatant liquid is then filtered through a dry filter into a dry flask, leaving most of the solid material in the flask, passing the first part through twice, if necessary, for a clear filtrate. With a pipette, exactly 50 cc. of clear filtrate are run into a 500-cc. Kjeldahl digestion-flask, 20 cc. of the usual reagent sulphuric acid for the Gunning process (p. 58) are added, and the contents of the flask brought to a gentle boil. After the water has

\* Am. Chem. Jour. XV, 392-471; XVI, 524.

† Ark. Exp. Sta. Bul. 42, p. 96.



been driven off and the acid has stopped foaming, the potassium sulphate is added and the digestion completed. From the per cent of nitrogen thus obtained 0.27% is deducted, this figure corresponding to the amount of gliadin soluble in 1% salt solution under the above conditions. The remainder is the percentage of non-gluten nitrogen.

*Gluten Nitrogen.*—This is obtained by difference between the total nitrogen and the non-gluten nitrogen as above obtained, or by deducting the combined nitrogen of the edestin, leucosin, and the amido-nitrogen from the total nitrogen.

*Edestin and Leucosin.*—Edestin is a globulin belonging to the vegetable vitellins, and is precipitated from salt solutions by dilution, or by saturation with magnesium or ammonium sulphate, but not by saturating with sodium chloride. It is not coagulated below 100° C., but is partly precipitated by boiling. Leucosin is an albumin, coagulating at 52°, but precipitates from salt solution by saturating with sodium chloride or magnesium sulphate.

To 50 cc. of the clear salt extract, obtained as described under non-gluten nitrogen, 250 cc. of pure 94% alcohol are added in a Kjeldahl 500-cc. digestion-flask, the contents thoroughly mixed, and allowed to stand over night. The precipitate is collected in a 10-cm. filter, which is returned to the flask and the nitrogen determined. This represents the nitrogen of the combined edestin and leucosin. These proteins may, however, be separated by coagulating the leucosin at 60°, and precipitating the edestin by adding alcohol to 50 cc. of the clear filtrate, determining the nitrogen separately in each precipitate.

*Amido-nitrogen.*—Allantoin, asparagin, cholin, and betaine are nitrogenous bases present in wheat.

Ten cc. of a 10% solution of pure phosphotungstic acid are added to 100 cc. of the clear salt extract as above obtained, thus precipitating all the proteins, which are allowed to settle preferably over night. Filter, and determine the nitrogen in the clear filtrate. The filtrate should be tested with a little of the phosphotungstic acid reagent to make sure that all the proteins have been separated. In some cases, as in bran for instance, more than 10 cc. of the reagent are necessary.

*Gliadin* is dissolved most readily from flour by hot dilute alcohol, but is entirely insoluble in absolute alcohol. One gram of the material is extracted with 100 cc. of hot 75% alcohol, by shaking the mixture thoroughly in a flask, and heating for an hour at a temperature just below



the boiling-point of alcohol, with occasional shaking. After standing for an hour, the hot liquid is decanted upon a 10-cm. filter, and 25 cc. of the hot alcohol are added to the residue and shaken, after which the residue is again allowed to settle, and the liquid decanted. This is repeated six times. The remainder of the alcohol is then driven off by evaporation, and the nitrogen determined in the residue. The difference between the total nitrogen and the nitrogen thus obtained, gives the nitrogen of the alcoholic extract, which includes the amides. Subtracting the latter, or amido-nitrogen, the remainder is the gliadin nitrogen.

*Glutenin Nitrogen.*—This is the difference between the gluten nitrogen and the gliadin nitrogen.

The factor by which the nitrogen should be multiplied in determining the various proteins, according to Osborne and Voorhees, is 5.7 for wheat.

**Proteins of the Common Cereals and Vegetables.**—Osborne and his coworkers have made a detailed study of the protein constituents not only of wheat as above outlined, but of other common grains and vegetables, and the results of these investigations may be thus briefly summarized:

**Proteins of rye:\***

	Per Cent.
Insoluble in salt solution .....	2.44
Soluble in alcohol, gliadin .....	4.00
Soluble in water, leucosin .....	0.43
Soluble in salt solution: { Edestin } { Proteose } .....	1.76
	<hr/> 8.63

**Proteins of barley:†**

	Per Cent.
Soluble in water: { Leucosin } { Proteose } .....	0.3
Soluble in salt solution, edestin .....	1.95
Soluble in dilute alcohol, hordein .....	4.00
Insoluble in water, salt solution, and alcohol .....	4.50

**Proteins of corn:‡**

Soluble in water: Proteose .....	0.06
Soluble in salt solution: { Very soluble globulin .....	0.04
{ Maysin .....	0.25
{ Edestin .....	1.10
Soluble in dilute alcohol: Zein .....	5.00
Insoluble in above, but soluble in two-tenths per cent potash solution .....	3.15

**Protein of pea:§**

Soluble in salt solution: Globulins { Legumin .....	7.00
{ Vicilin .....	3.00
Soluble in water: Albumin, legumelin, proteose .....	2.03

\* Jour. Am. Chem. Soc., 17, page 429.

† Ibid., 17, p. 539.

‡ Ibid., 19, p. 525.

§ Ibid., 18, p. 583; 20, pp. 348 and 410.



## MINERAL CONSTITUENTS OF CEREALS AND VEGETABLES.

The food analyst often finds the determination of one or more of the mineral constituents of a food product of value as a means of detecting adulteration, since the addition of foreign material may alter materially the composition of the ash.

The following table \* shows the composition of the pure ash of common cereals.

## COMPOSITION OF ASH OF CEREALS.

	K <sub>2</sub> O.	Na <sub>2</sub> O.	CaO.	MgO.	Fe <sub>2</sub> O <sub>3</sub> .	P <sub>2</sub> O <sub>5</sub> .	SO <sub>3</sub> .	Cl.	SiO <sub>2</sub> .
Wheat (Canada).....	24.03	9.55	3.50	13.24	0.52	46.87	0.01	0.00	2.28
Rye (Minnesota).....	27.60	4.64	5.56	11.73	5.23	41.81	0.52	0.58	2.45
Barley (U. S.).....	24.15	6.42	2.44	8.23	0.33	35.47	0.22	0.56	22.30
Oats (U. S.).....	15.91	4.38	4.09	7.18	0.20	24.34	0.48	1.02	42.64
Corn (U. S.).....	33.92	7.72	3.18	17.99	0.50	35.25	0.44	0.00	1.00
Rice, polished (Guatemala)...	20.84	13.98	4.48	9.60	0.89	43.21	0.24	0.80	6.14
Buckwheat (U. S.).....	35.15	2.26	6.62	20.55	1.68	24.09	3.59	0.67	5.54

Snyder † obtained the following average results in the analysis of the ash of 12 samples of wheat:

Potash (K <sub>2</sub> O).....	30.2%
Soda (Na <sub>2</sub> O).....	0.7
Lime (CaO).....	3.5
Magnesia (MgO).....	13.2
Ferric oxide (Fe <sub>2</sub> O <sub>3</sub> ).....	0.6
Phosphoric acid (P <sub>2</sub> O <sub>5</sub> ).....	47.9
Sulphuric acid (SO <sub>3</sub> ).....	0.1
Silica (SiO <sub>2</sub> ).....	0.7
Chlorine (Cl).....	0.2

The average amount of ash in the grain was 2.0%.

König gives the following analyses of the ash of various leguminous and other vegetables:

\* U. S. Dept. of Agric., Bur. of Chem., Bul. 13, part 9, p. 1212.

† Minn. Agric. Exp. Sta., Bul. 29, 1893, p. 149.



	Number of Analyses.	Ash in Dry Substance.	Potash.	Soda.	Lime.	Magnesia.	Iron Oxide.	Phosphoric Acid.	Sulphuric Acid.	Silica.	Chlorine.
Beans.....	15	3.57	42.49	1.34	4.73	7.08	0.57	38.74	2.53	0.73	1.57
Peas.....	29	2.73	41.79	0.96	4.99	7.96	0.86	36.43	3.49	0.86	1.54
Potatoes.....	53	3.77	60.37	2.62	2.57	4.69	1.18	17.33	6.49	2.13	3.11
Beets.....	15	6.44	54.02	15.90	4.12	4.54	0.82	8.45	3.17	2.38	8.40
Carrots.....	11	5.58	35.21	22.07	11.42	4.73	1.03	12.46	6.72	2.47	5.13
Turnips.....	32	8.01	45.40	9.84	10.60	3.69	0.81	12.71	11.19	1.87	5.01

**Scheme for Complete Ash Analysis.**—The following scheme in essential details was suggested by the later Prof. S. L. Penfield of Yale University for use at the Connecticut Agricultural Experiment Station.

*Preparation of Ash.*—The amount of material which should be reduced to ash depends on the percentage of total ash present and the amount of material available. Usually 100 grams is a suitable amount; if, however, the material (e.g., tobacco) is rich in ash, 50 grams is sufficient, while if it contains but a small amount of ash, 200 grams or even more may be required. About 5 grams of ash is a liberal amount for a complete analysis, but in case of necessity 1 gram will suffice if care is taken to so adapt the scheme as to make as many determinations as possible on one weighed portion.

The ashing is carried on in a platinum dish heated below redness by a Bunsen burner. In order to distribute the heat and prevent overheating, a piece of asbestos paper is introduced between the dish and the flame. The material first chars, then begins to glow just below the surface, and the combustion gradually extends downward until it reaches the bottom of the dish. Then, and not until then, the unburned carbon on the surface should be stirred in with the ash to facilitate burning. Care should be taken not to heat higher than dull redness, thus avoiding the loss of alkali chlorides and the fusion of alkali phosphates about the particles of carbon. A muffle furnace may be used to complete the burning.

Substances rich in starch or sugar are most difficult of combustion, as the charcoal forms a hard mass, while substances rich in fibrous or woody matter burn quite readily without losing their powdered condition. A certain amount of unburned carbon is no disadvantage, as it is determined in the course of the analysis.



Finally cool the ash, grind to a powder, mix without loss, and weigh, thus determining the percentage of crude ash.

*Determination of Water.*—Heat 1 gram of the ash in a platinum crucible well below redness to constant weight.

*Determination of Carbonic Acid.*—Determine carbonic acid as described on p. 353 using the portion dried for the determination of water.

*Determination of Charcoal and Sand.*—Weigh 1 gram of the ash, or transfer the solution and residue from the determination of carbonic acid, into a beaker, add 25 cc. of water and 25 cc. of 10 per cent hydrochloric acid, and boil gently for 10 minutes. Filter on a Gooch crucible, and wash thoroughly with hot water. Reserve the filtrate for determination of silica, iron oxide, alumina, lime, and magnesia. Wash the residue on the crucible once with alcohol and once with ether, and dry to constant weight at 100° C. Ignite and weigh again. The loss on ignition is the charcoal, the residue is sand.

*Determination of Silica, Iron Oxide, Alumina, Lime and Magnesia.*—Evaporate to dryness in a platinum dish the filtrate from the determination of charcoal and sand, heat for some hours on the water bath, and dry at 130° C. until all hydrochloric acid is removed. Moisten the residue thoroughly with concentrated hydrochloric acid, add hot water, stir, and decant the solution on an ashless filter. Treat the residue again with acid and hot water, and repeat the treatment until nothing but silica remains undissolved. Finally collect the silica on the paper, wash with hot water, ignite in a platinum crucible, and weigh.

To the filtrate add ammonia until a precipitate forms which remains on stirring, and then add sufficient hydrochloric acid to just dissolve the precipitate. Heat to 50° C. and add an excess of ammonium acetate solution and 4 cc. of 80 per cent acetic acid. Digest at 50° C. until the mixed phosphates of iron and alumina have settled, filter, wash with hot water, ignite in a platinum crucible, and weigh. As the precipitate is usually slight and consists almost entirely of iron phosphate, the iron oxide may be calculated with reasonable accuracy using the factor 0.53. If, however, greater accuracy is desired fuse the weighed precipitate with 10 parts of sodium carbonate, dissolve in dilute sulphuric acid, reduce with hydrogen sulphide, determine iron by the volumetric permanganate method, and in the same solution determine phosphoric acid by the molybdic method. The alumina is obtained by difference, subtracting the sum of the weights of the oxide of iron and phosphoric acid from the total weight of the precipitate.



To the filtrate from the mixed phosphates add an excess of ammonium oxalate, allow to stand in a warm place over night, filter, ignite the precipitate in a platinum crucible over a Bunsen burner, and finally to constant weight over a blast lamp, thus obtaining the calcium oxide.

Precipitate the magnesia in the filtrate from the lime by adding ammonia to alkaline reaction, then an excess of sodium phosphate solution with constant stirring, and finally sufficient concentrated ammonia to form one-tenth the final volume. Let stand over night, collect the magnesium ammonium phosphate on a Gooch crucible, ignite to magnesium pyrophosphate, and weigh.

*Determination of Sulphuric Acid, Potash, and Soda.*—Boil 1 gram of the ash with dilute hydrochloric acid, and remove charcoal, sand, and silica, as described in the preceding section. Evaporate nearly to dryness to remove the excess of acid. Dilute to 100 cc., heat to boiling, and add barium chloride solution drop by drop until the sulphuric acid is precipitated. Allow to stand over night, filter, ignite, and weigh as  $\text{BaSO}_4$ .

Heat the filtrate to boiling, add enough barium hydroxide to make the solution strongly alkaline, filter, and proceed with the determination of potash and soda, as described on p. 361.

*Determination of Phosphoric Acid.*—Dissolve 0.5 gram of the ash in hydrochloric acid, filter, and wash. Neutralize with ammonia, clear with nitric acid, and proceed as described on p. 362.

*Determination of Chlorine.*—Dissolve 1 gram of the ash in cold, very dilute nitric acid, filter, and wash. To the filtrate add an excess of silver nitrate, and heat nearly to boiling with constant stirring. Filter on a Gooch crucible, wash with hot water, dry the precipitate at a low heat, and heat cautiously at dull redness until the silver chloride has partially melted.

If desired the chlorine may be determined volumetrically by Volhard's method, as follows: To the nitric acid solution add a known volume of decinormal silver nitrate solution sufficient to precipitate the chlorine, and 5 cc. of saturated solution of ferric alum. Titrate with decinormal ammonium thiocyanate solution until a permanent brown color is formed. Subtract the volume required from the volume of decinormal silver nitrate added, and calculate the chlorine.

**Determination of Sulphur in Vegetable Materials.\***—Place from 1.5 to 2.5 grams of material in a nickel crucible of about 100 cc. capacity

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\*A. O. A. C. Method, U. S. Dept. of Agric., Bur. of Chem., Bul. 107 (rev.), pp. 23, 24.



and moisten with approximately 2 cc. of water. Mix thoroughly, using a nickel or platinum rod. Add 5 grams of pure anhydrous sodium carbonate, and mix. Add pure sodium peroxide, small amounts (approximately 0.50 gram) at a time, thoroughly mixing the charge after each addition. Continue adding the peroxide until the mixture becomes nearly dry and quite granular, requiring usually about 5 grams of peroxide. Place the crucible over a low alcohol flame (or other flame free from sulphur), and carefully heat with occasional stirring until contents are fused. (Should the material ignite the determination is worthless.) After fusion, remove the crucible, allow to cool somewhat, and cover the hardened mass with peroxide to a depth of about 0.5 cm. Heat gradually, and finally with full flame until complete fusion takes place, rotating the crucible from time to time in order to bring any particles adhering to the sides into contact with the oxidizing material. Allow to remain over the lamp for ten minutes after fusion is complete. Cool somewhat. Place warm crucible and contents in a 600 cc. beaker, and carefully add about 100 cc. of water. After violent action has ceased, wash material out of crucible, make slightly acid with hydrochloric acid (adding small portions at a time), transfer to a 500 cc. flask, cool, and make to volume. Filter, and take a 200 cc. aliquot for determination of sulphates by precipitating with barium chloride in the usual manner.

**Determination of Chlorine in Vegetable Substances.\*** — Impregnate 5 grams of substance in a platinum dish with 20 cc. of a 5 per cent solution of sodium carbonate, evaporate to dryness, and ignite as thoroughly as possible. Extract the residue with hot water, filter, and wash. Return to the platinum dish, ignite to an ash, dissolve in nitric acid, and determine chlorine by the Volhard method (p. 313).

#### MICROSCOPY OF CEREAL PRODUCTS.

The histology of the cereals is more fully considered in the works on food microscopy, the brief descriptions here given should, however, enable the food chemist to identify the commoner products. The tissues of the various cereals are quite distinctive, serving usually to determine the particular grain from which a given product is made. In the case of flour the tissues are largely removed in milling, the fragments remaining being small and few in number. Such products are identified either by

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\* A. O. A. C. Method, U. S. Dept. of Agric., Bur. of Chem., Bul. 107 (rev.), pp. 23, 24.



the character of the starch grains—as in the detection of wheat or corn flour in buckwheat flour—or else, if the starch is not sufficiently characteristic—as in the detection of wheat flour in rye flour—by examining the tissues from a considerable amount of the material.

The most convenient method of accumulating the tissues from flour is to mix thoroughly 2 grams of the material with 200 cc. of water and 3 cc.

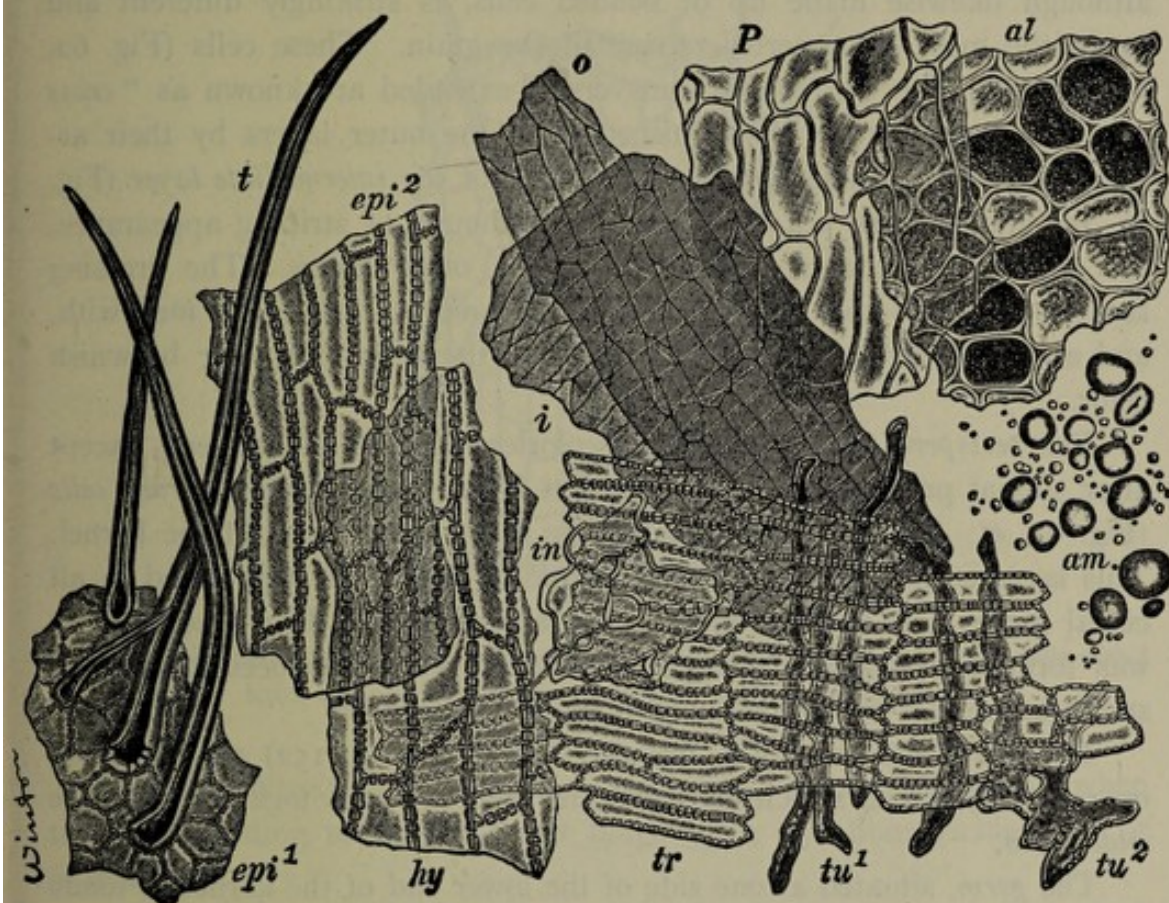


FIG. 62.—Wheat. Elements in Surface View.  $\times 160$ . (WINTON.)

*epi¹* epicarp at end of grain, with *t* hairs; *epi²* epicarp on body of grain; *hy* hypoderm (first layer of mesocarp); *in* intermediate cells; *tr* cross cells; *tu¹* typical tube cells; *tu²* tube cells passing into spongy parenchyma; *o* outer layer of spermoderm; *i* inner layer of spermoderm; *P* perisperm; *al* aleurone cells; *am* starch grains.

of sulphuric acid, bring to a boil, allow to settle, and carefully decant off the liquid from the deposit of tissues. The tissues are mounted for examination in very dilute sodium hydroxide solution.

**Wheat Products.**—Fig. 62 and Pl. VIII show the principal elements of the wheat kernel.

The outer layer or *epicarp* (Fig. 62, *epi¹* and *epi²*) consists of beaded



cells, which on the body of the kernel are elongated, but at the end are polygonal. From this layer at the end of the kernel arise the hairs (Fig. 62, *t*; Pl. VIII, Fig. 151) which form a beard clearly visible under a lens. Some of these hairs become detached in milling, and pass endwise through the bolts, hence their presence in even the highest grade of flour. The second layer or *hypoderm* (*hy*) resembles the first, while the third, although likewise made up of beaded cells, is strikingly different and forms the most characteristic tissue of the grain. These cells (Fig. 62, *tr*; Pl. VIII, Fig. 150) being transversely extended are known as "*cross cells*," and are further distinguished from the outer layers by their arrangement side by side in rows. The cells of the *intermediate layer* (Fig. 62, *in*) and the *tube cells* (*tu*<sup>1</sup> and *tu*<sup>2</sup>), although of striking appearance, are not of as frequent occurrence as the other layers. The crossing layers of the *seed coat* or *spermoderm* (*i* and *o*), are often met with, and are characterized by the thin walls of the cells and their brownish color.

The *perisperm* (*P*), consisting of colorless cells, is seldom seen, except after special preparation, while the next layer, made up of *aleurone cells* (Fig. 62, *al*; Pl. VIII, Fig. 150), is the most conspicuous of the kernel. This layer is not, however, characteristic of wheat, as it is found in all cereal grains and in buckwheat. The aleurone cells do not contain, as was formerly supposed, the gluten of the grain; this occurs with the starch in the thin-walled cells within the aleurone layer.

The *starch granules* (Fig. 62, *am*; Pl. VIII, Fig. 152) are described on page 289. The starch cells and the aleurone cells together form the *endosperm*.

The *germ*, situated at one side of the lower end of the kernel, is made up of very small cells containing fat and protein, but no starch.

**Rye Products.**—The structure of rye (Fig. 63; Pl. VII) resembles closely that of wheat. The number and general characters of the cell layers are the same in both, and the starch granules are very much alike. There are, however, certain points of difference which serve to distinguish the products of the two cereals, and even to detect the presence of a wheat product in a rye product, and *vice versa*:

First. The breadth of the cavities of wheat hairs is usually less than the thickness of the walls, whereas in rye hairs the reverse is often true (Figs. 62 and 63, *t*).

Second. The cross cells of wheat have rather thick, distinctly beaded side walls, and thin, pointed end walls; the cross cells of rye have rather



thin, indistinctly beaded side walls, and usually swollen, rounded end walls (Figs. 62 and 63, *tr*; Figs. 150 and 146).

Third. The large starch granules of wheat seldom reach 0.050 mm. in diameter, while those of rye frequently exceed that limit. Radiating clefts often occur in the starch granules of rye (Pl. VII, Fig. 148).

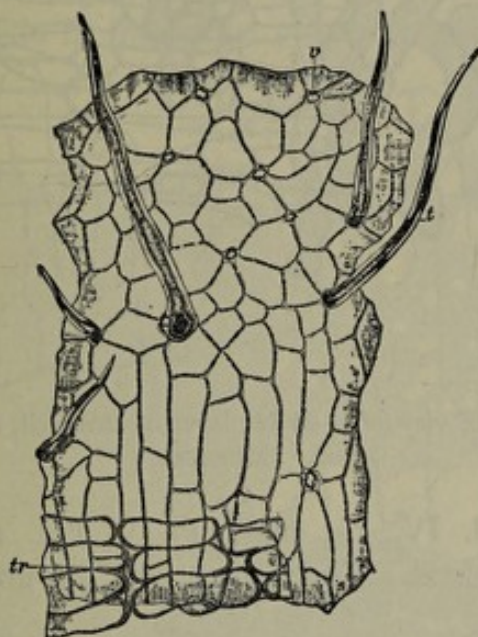


FIG. 63.—Rye, Outer Bran Layers in Surface View. Epicarp consists of porous cells with *t* hairs, and *v* hair scars; *tr* cross cells.  $\times 160$ . (MOELLER.)

Fourth. Wheat flour yields a considerable amount of gluten when treated according to Bamihl's test (page 336); rye flour yields none or only a trace.

**Barley Products.**—The common varieties of barley are "chaffy," that is, the grain after threshing is still closely invested by the chaff (Pl. I, Fig. 123). The grain within the chaff is analogous in structure to wheat and rye, but differs from these in that the cross cells are not beaded and form a double layer (Fig. 64, *tr*), and the starch granules seldom exceed 0.035 mm. in diameter (Pl. I, Fig. 124). The starch is more fully described on page 290.

**Corn Products.**—The most characteristic element of corn is the starch (page 290; Pl. IV). Polygonal starch granules 0.017 to 0.030 mm. in diameter occur in no other vegetable product of economic importance, excepting the seeds of Kaffir corn and other grains belonging to the genus *Sorghum*, which are used chiefly for cattle or poultry foods.



**Oat Products.**—The oat kernel resembles barley in appearance, but is not ribbed. In the preparation of oat meal and other breakfast

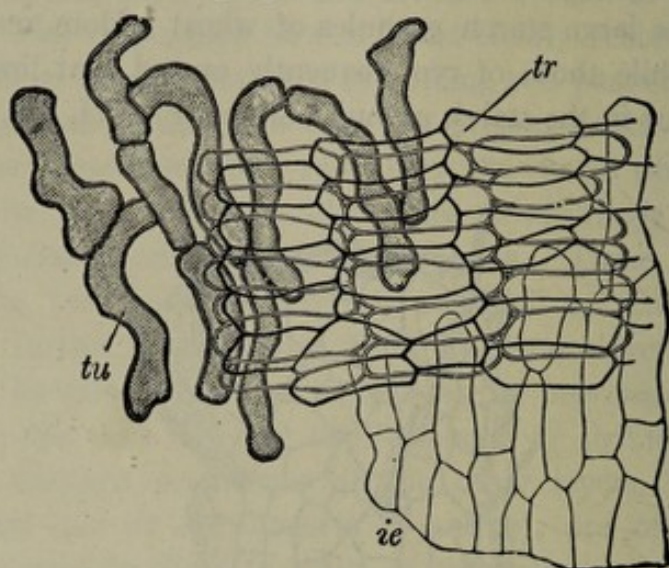


FIG. 64.—Barley. Surface view of *tr* double layer of cross-cell; *tu* tube cells; *ie* seed coat.  $\times 300$ . (MOELLER.)

foods, the chaff (Pl. IV, Fig. 135; Pl. V, Fig. 137) is removed and utilized as a cattle food. The elements of the grain of chief value in

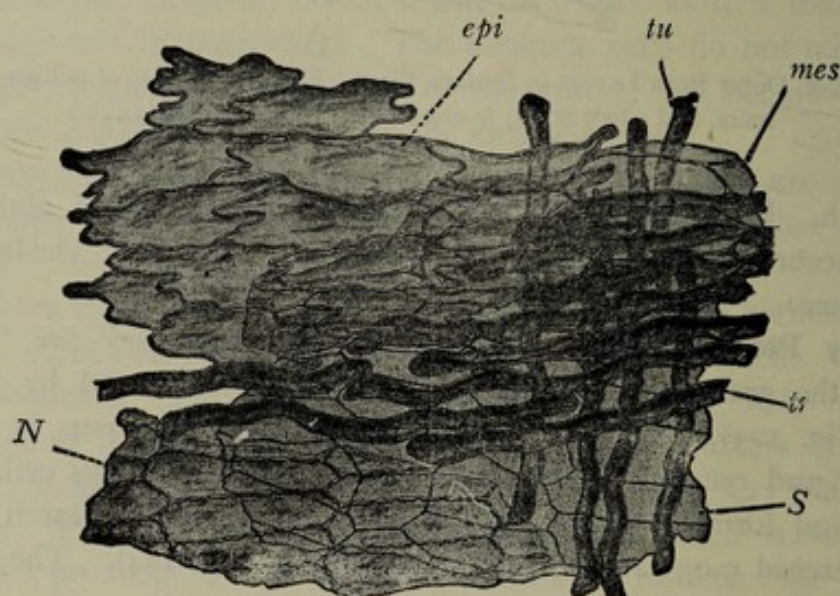


FIG. 65.—Rice. Bran coats in surface view. *epi* epicarp; *mes* mesocarp; *tr* cross cells; *tu* tube cells; *S* seed coat; *N* perisperm.  $\times 300$ . (WINTON.)

identification are the hairs and the starch granules. The hairs (Pl. V, Fig. 138) are much longer than those of wheat, rye, and barley, often reaching 1 mm. They taper toward both ends, so that when detached



they often appear to be pointed at the base as well as at the apex. The starch granules are small, of the polygonal type, and often occur in egg-shaped aggregates (page 291; Pl. V, Fig. 139).

**Rice Products.**—The chaff which envelops this grain is rough and silicious, and after removal from the inner kernel is not suited even for cattle food. Its appearance under the microscope is shown in Plate VI, Fig. 142. The thin skin of the kernel proper is largely but not entirely

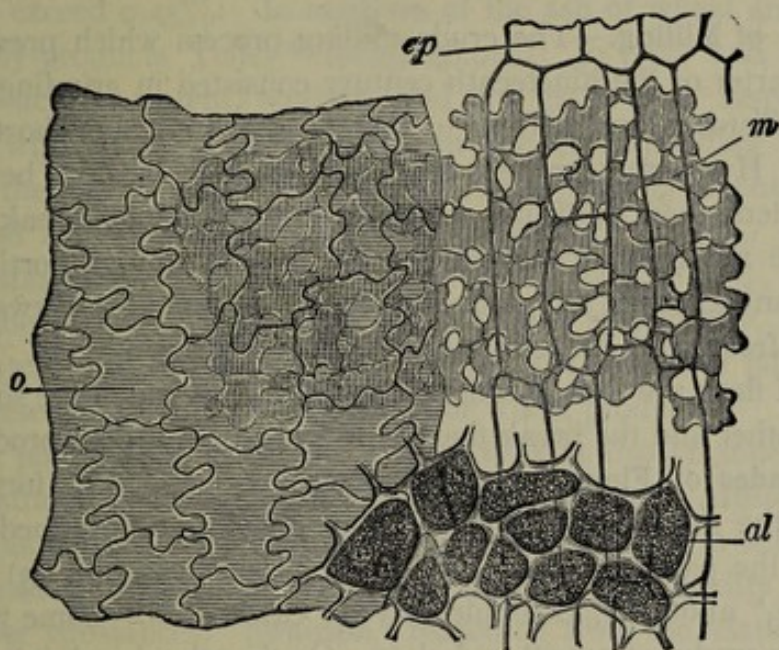


FIG. 66.—Buckwheat. Bran coats in surface view. Seed coat consists of *o* outer epidermis, *m* spongy parenchyma, and *ep* inner epidermis; *al* aleurone cell.  $\times 300$ . (MOELLER.)

removed in the preparation of rice for the market. The elements of this skin are shown in Fig. 65, the outer layer (*epi*) being the most characteristic. Unlike wheat, rye, and barley, it has no beard. Rice starch (Pl. VI, Fig. 143) is hardly distinguishable from oat starch. It is described on page 291.

**Buckwheat Products.**—In the preparation of buckwheat flour the black outer hulls and the inner skin or bran are largely, but not completely, removed. The bran elements are characteristic constituents of the flour, and are rendered especially distinct by adding a drop of dilute potassium hydroxide solution to a water mount (Fig. 66). The cells with wavy walls (*o*) and the spongy parenchyma (*m*) are especially noticeable. The starch of buckwheat resembles that of oats, but the individual granules are somewhat larger and occur in rod-shaped, not egg-shaped, aggregates (page 290; Pl. II, Fig. 128). Masses of starch



granules (Pl. III, Fig. 129) conforming to the shape of the cells, occur in abundance in the flour.

#### FLOUR.

Flour is the term applied to the finely ground and bolted substance of wheat and other grains, though, unless otherwise qualified, by the term "flour" is generally understood that of wheat.

Graham flour is an unbolted meal prepared from the whole wheat kernel.

**Process of Milling.**—The crude milling process which prevailed until the last quarter of the nineteenth century consisted in grinding the wheat between millstones and bolting to remove the bran and shorts. In the modern or Hungarian process the wheat is first crushed between corrugated rollers, then by sifting separated into middlings, break flour, and bran. The middlings, consisting of the hard glutinous portions of the grain in granular form, are gradually reduced to fine flour between smooth rollers and freed from impurities by means of a series of bolts. A number of grades of flour are thus produced, the streams of which are so combined with each other and the break flour as to form the finished products.

**The Grades of Flour** commonly made are (1) *patent*, forming 85% or less of the flour output; (2) *clear* or *bakers'*, an intermediate grade inferior to the patent in color and rising properties; and (3) *low grade* or "*red dog*," about 5%, suitable only for cattle food. Some mills make two or more grades of patent and clear. On the other hand, it is a frequent practice to combine all the flour streams other than of low grade to form a *straight*.

**The By-products** are (1) *bran*, the outer coatings of the grain in flakes, (2) *shorts*, the finer offal containing both starchy matter and bran elements, and (3) *germ*, rich in oil, often run in with the bran.

**Composition of Wheat Flour and By-products.**—The following analyses by Clifford Richardson \* represent the products of the same milling:

	Mois- ture.	Protein N × 5.7	Moist Gluten.	Dry Gluten.	Ether Ex- tract (Fat).	Nitro- gen-free Extract (Starch, etc).	Crude Fiber.	Ash.	Phos- phoric Acid.	Fuel Value per Pound. Cal.
Wheat (Spring)...	9.07	12.93	....	....	2.74	71.77	1.70	1.79	0.82	1723
Patent flour.....	11.48	11.81	36.14	10.85	1.45	74.69	0.18	0.39	0.18	1673
Clear flour.....	12.18	13.57	51.51	16.97	2.00	71.30	0.33	0.62	0.31	1669
Low-grade flour..	12.01	16.37	10.01	4.26	3.86	64.84	0.93	1.99	1.16	1691
Shorts.....	10.94	15.32	....	....	4.67	61.76	3.90	3.41	1.62	1703
Bran.....	10.91	14.84	....	....	5.03	57.65	5.98	5.59	2.78	1672

\* U. S. Dept. of Agric., Bul. 4, 1884, p. 38.



From the above it appears that the fat, fiber, ash, and phosphoric acid increase through the series, being least in the patent flour and greatest in the bran, while the nitrogen-free extract decreases. Considering only the flour, the protein is least in the patent and greatest in the low grade, but the gluten, although greater in the clear than in the patent, drops down sharply in the low grade.

The *ash* of flour is a valuable index of grade. In a true patent it should not exceed 0.45%. In analyses of the ash of wheat and its products and by-products Teller\* found 48.05%, 49.32%, and 53.10% of phosphoric acid in patent, straight, and low grade flour respectively, as compared with 52.14% and 52.18% in the corresponding wheat and bran. In the ash of patent flour Teller found 38.50% of potash, 5.59% of lime, and 4.39% of magnesia, as compared with 29.70%, 3.10%, and 13.23% respectively in the wheat. He found 0.24%, 0.27%, and 0.04% of zinc oxide in wheat, bran, and straight flour respectively, but reports none in patent and low grade flour. Other analysts have failed to confirm the presence of zinc oxide in wheat or wheat products.

As first pointed out by Snyder,† the *acidity* is also an index of grade. This is illustrated by the figures in the table on page 322. Although usually calculated as lactic acid, which acid is doubtless present in slight amount on aging and in larger amount after spoilage, the acidity is largely due to other substances. Swanson,† reasoning from the relation between acidity, amino compounds, and phosphorus and the apparent relation between acidity and ash, believes the acidity to be due in part to monopotassium phosphate and amino compounds.

*Hard Wheats*, such as the Spring varieties of the Northwest and Turkey red Winter wheat of Kansas, yield a "strong" flour rich in protein and gluten, the latter being of good tenacity, while *Soft Wheats*, such as are grown in states adjoining the Ohio River, yield a white starchy flour, the gluten being smaller in amount and lacking in tenacity.

Analyses of typical hard and soft wheat flour freshly ground are given in the table on top of page 322. The percentages are of the total flour, excluding the low grade. Color values are given on page 326.

**Color of Flour.**—The time-honored test for grade is by the color. A patent is practically free from bran specks while a clear contains such specks in noticeable amount. Both grades have a yellowish tint due to the color associated with the fat, which is more or less pronounced,

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\* Ark. Agric. Exp. Sta., Bul. 42.

† Jour. Ind. Eng. Chem., 4, 1912, p. 274.



	Minnesota. Hard Spring.		Nebraska. Hard Winter.		Michigan. Soft Winter.		Missouri. Soft Winter.	
	78% Patent.	22% Clear.	80% Patent.	20% Clear.	80% Patent.	20% Clear.	40% Patent.	60% Clear.
Moisture.....	13.74	13.26	13.33	12.85	13.22	12.62	12.27	12.02
Ash.....	0.44	0.85	0.39	0.67	0.42	0.89	0.39	0.50
Crude fiber.....	0.06	0.26	0.18	0.24	0.19	0.27	0.34	0.38
Protein (N×5.7).....	10.60	11.74	11.09	11.86	8.66	12.26	9.01	10.72
Alcohol sol. protein....	5.84	6.21	5.79	6.55	5.24	5.53	5.04	6.21
Salt sol. protein.....	1.62	2.19	1.48	2.02	1.45	2.19	1.25	1.43
Moist gluten.....	36.93	38.76	30.48	42.50	20.23	31.24	17.90	33.21
Dry gluten.....	12.48	13.41	9.85	13.08	6.97	10.55	5.90	10.22
Nitrogen-free extract...	74.07	71.91	75.16	73.06	76.40	72.19	77.12	75.23
Fat.....	1.09	1.98	0.85	1.32	1.11	1.77	0.87	1.15
Acidity as lactic.....	0.108	0.230	0.081	0.158	0.110	0.250	0.063	0.095

according to the kind of wheat, but is not proportional to the per cent of fat. This is measured by the gasoline color value (pp. 326 and 327).

**Graham and Whole Wheat Flour.**—Graham flour, so named because of its early advocate, Dr. Graham, is the meal obtained by grinding the whole wheat kernel. Entire wheat flour, etomologically, should be a synonym for Graham flour, but by trade usage has come to mean the ground wheat after removal of the bran. The following analyses by Snyder\* show the composition of a sample of Oklahoma wheat, also the Graham, entire wheat, and straight flour made therefrom:

	Water.	Protein N × 5.7	Fat.	Total Carbohy- drates.	Ash.	Fuel Value, per Pound, Calories.
Grain.....	8.65	15.33	1.83	72.87	1.32	4160
Graham flour.....	7.73	15.33	1.79	73.83	1.32	4196
Entire wheat flour.....	7.46	15.16	1.64	74.52	1.22	4201
Straight grade flour.....	9.93	13.74	0.92	74.89	0.52	4065

So-called Graham and whole wheat flour are too often various mixtures of the by-products of milling.

In distinguishing imitation from Graham flour, valuable indications are furnished by comparing the analysis with that of whole wheat. After removal of the flour from the grain by the ordinary process of milling, no combination of the by-products can have the same analysis as that of the wheat.

\* U. S. Dept. of Agric., Off. of Exp. Sta., Bul. 156, 1905.



Le Clerc and Jacobs\* have attempted to distinguish genuine from imitation Graham flour by determining the percentages of flour, fine middlings, coarse middlings, shorts, and bran obtained by sifting and by determining in each portion total and alcohol-soluble nitrogen, and gliadin ratio, also in many cases by determining in addition ash, fiber, and pentosans (but not fat and nitrogen-free extract) in the unseparated sample and the flour.

**Flour of Other Cereals.**—The following analyses are from Bulletin 13, Part 9, of the Bureau of Chemistry:

	No. of Analyses.	Moisture.	Protein N X 6.25.	Ether Extract (Fat).	Nitrogen- free Ex- tract (Starch, etc.).	Crude Fiber.	Ash.	Calcu- lated Calories of Com- bustion.
Corn flour.....	3	12.57	7.13	1.33	78.36	0.87	0.61	38.37
Rye flour.....	1	11.41	13.56	1.97	73.37	1.86	1.55	
Barley flour.....	1	10.92	7.50	0.89	80.50	0.67	0.86	
Buckwheat flour	1	11.89	8.75	1.58	75.41	0.52	1.85	38.54

**Damaged Flour.**—Grain is often damaged by the growth of smuts, rusts, and ergot. Both grain and flour are also liable to attacks of molds, yeasts, algæ, and bacteria.

Various insects and other forms of animal life frequently infest grain or flour. Among these are weevils and various other beetles, flour moths, mites, and the wheat worm, a nematode related to trichina.

Grain may also be damaged by sprouting, the diastase thus formed partially dissolving the starch granules with the formation of fissures and branching channels, readily seen under the microscope. Flour thus damaged is high in cold-water extract (p. 333).

Experiments by Olsen † and Swanson, Fitz, and Dunton, ‡ indicate that the injury to the flour resulting from sprouting of the grain is not so great as was formerly supposed. The former found that while loaves made entirely from the flour of sprouted wheat were sticky, sweet, and spongy, addition of 10 % of sprouted to sound flour gave larger and better loaves, and the latter investigators found that the addition of flour from sprouted grain to sound flour produced little or no injurious effects.

**Ergot.**—Ergot, a fungous growth containing a poisonous alkaloid, sometimes develops in rye and, less often, in wheat. Under the micro-

\* U. S. Dept. of Agric., Bur. of Chem., Bul. 164, 1913.

† Amer. Food Jour., 6, 1911, p. 36.

‡ Kan. Agric. Exp. Sta., Tech. Bul. 1, 1916.



scope it appears as a fine network of mostly colorless parenchyma cells, containing globules of fat (Fig. 67). Some of the cells are circular, others considerably elongated, and some contain a deep-brown coloring matter, which, with ammonia, become violet-red, changing to red with acid. Occasionally the cell walls appear of a dark color. If flour containing ergot be treated with a very dilute solution of anilin violet, the stain will be practically absorbed by the damaged particles of the grain, and resisted by the normal granules. A hot, alcoholic extract of flour containing ergot is colored red when treated with dilute sulphuric acid.

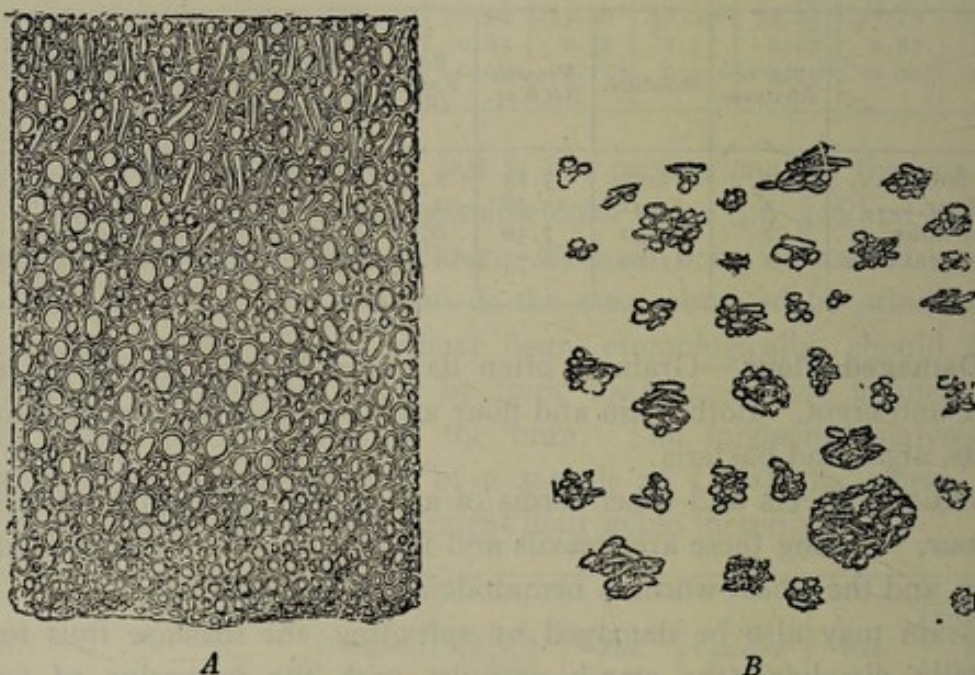


FIG. 67.—A, Transverse Section of the Ergot of Wheat under the Microscope; B, Powdered Wheat Ergot. (After Villiers and Collin.)

**Adulteration of Flour.**—Besides the substitution of cheaper or inferior grades for those of higher quality, the fraudulent admixture of corn flour to wheat flour was at one time extensively practiced. This adulterant is best detected by the microscope (p. 317).

Rye flour has been adulterated with cheap grades of wheat flour or middlings. These admixtures are detected by the Bamihl test (p. 336) and by microscopic examination of the residue after boiling with dilute acid (page 315), noting especially the cross cells.

Much of the so-called buckwheat flour formerly contained large amounts of wheat or corn flour, or both. Rice flour is also used in pancake flours, although probably not to cheapen the product. Self-raising pancake



flours are usually mixtures of two or more flours with leavening material. The microscopic characteristics of the starch grains and tissues serve to identify the different flours present in such mixtures.

In England so-called "improvers," notably calcium acid phosphate and other soluble phosphates and potassium persulphate, have come into use to correct certain defects of flour made from native wheats. The addition of phosphorus trichloride and pentachloride and various phosphorus and sulphur compounds has also been proposed.

**Alum in Flour.**—Alum was formerly used in Europe, both by miller and baker, to improve the appearance of inferior or slightly damaged flour, but now is rarely if ever employed, and the presence of notable quantities of aluminum compounds in flour or bread is usually due to alum baking powder.

**Bleaching of Flour.**—In 1908 about 80% of the flour produced in the United States was bleached by nitrogen peroxide, but as a result of the enforcement of the federal law the practice has been largely discontinued. The gas is generated by electrical, chemical, or electro-chemical means, and is diluted with air before treatment of the flour. In the Alsop process, which is most commonly employed, it is formed by a flaming discharge of electricity, which causes the nitrogen and oxygen of the air to combine.

Nitrogen peroxide destroys almost immediately the yellow color which is associated with the fat of the flour, thus increasing the whiteness of the product. It also forms with the moisture of the flour nitrous and nitric acids, the former (free or combined), being easy of detection. A considerable part of the nitrous nitrogen remains in yeast bread after baking and nearly all of it in soda biscuit. Bleaching also diminishes the iodine number of the fat, affects the quality of the gluten, and injures the flavor of the bread.

Recently bleaching with chlorine has come into use.

**Aging versus Bleaching.**—Storage under proper conditions slowly whitens flour, improves its baking properties, increases its organic acidity, diminishes its water-content and brings about other changes not well understood. Nitrogen peroxide immediately whitens flour but does not improve its baking properties, increase its organic acidity nor appreciably affect its water-content. It does, however, introduce nitrous and nitric acids. Often 2 parts of nitrous nitrogen per million are recoverable and sometimes 6 or 7 parts, but this gradually disappears so that after some months hardly a trace remains.



The extent to which typical flours are whitened by aging and by bleaching so as to contain 2 parts of nitrous nitrogen per million is apparent from the gasoline color values in the following table by Winton:

	Minnesota, Hard Spring.		Nebraska, Hard Winter.		Michigan, Soft Winter.		Missouri, Soft Winter.	
	78% Patent.	22% Clear.	80% Patent.	20% Clear.	80% Patent.	20% Clear.	40% Patent.	60% Clear.
Gasoline color value of								
Unbleached:								
New.....	2.00	2.00	2.63	2.50	1.43	1.61	1.47	1.60
Aged 10 weeks....	1.78	1.82	2.12	2.17	1.22	1.49	1.22	1.33
Aged 20 " ....	1.20	1.34	1.36	1.68	0.80	1.20	0.68	0.88
Aged 30 " ....	0.72	0.88	0.70	0.82	0.56	0.72	0.48	0.52
Bleached:								
New.....	0.60	0.66	0.80	0.80	0.40	0.50	0.32	0.40
Aged 10 weeks....	0.44	0.54	0.46	0.48	0.26	0.38	0.22	0.26
Aged 20 " ....	0.30	0.50	0.34	0.40	0.20	0.36	0.18	0.24
Aged 30 " ....	0.30	0.50	0.24	0.36	0.18	0.40	0.14	0.16

#### INSPECTION AND ANALYSIS OF FLOUR.

In some of the larger cities, authorized inspectors are appointed by boards of trade to pass upon the quality of flour. To such inspectors dealers submit samples, which are gauged as to color, soundness, weight, etc., comparing them usually with a series of graded samples, and stamping or branding them officially with the date as well as the grade. Market quotations also are based on the standard terms adopted. The names of the various grades differ with the locality. In St. Louis, the following names are adopted in order of the quality, viz., Patent, Extra Fancy, Fancy, Choice, and Family.

The grade or quality of flour is determined largely by its color, fineness, odor, absorption, and dough-making properties. Baking tests are also relied on to a considerable extent by millers and buyers.

Of the chemical methods those for ash, protein, gluten, acidity, fat, and fiber are of chief importance.

**Fineness.**—The granulation is determined by rubbing the flour between the thumb and fingers. A gritty flour is one that feels rough and granular, due to aggregates of cells with contents intact. Smooth flour, on the other hand, feels soft and slippery. It is so finely ground that the cells are isolated and often ruptured, thus liberating the contents.

**Pekar Color-test.**—Place 10–15 grams of the flour on a rectangular glass plate, about 12 cm. long and 8 cm. wide, and pack on one side in a



straight line by means of a flour trier. Treat the same amount of the standard flour used for comparison in the same manner, so that the straight edges of the two flours are adjacent. Carefully move one of the portions so as to be in contact with the other, and "slick" both with one stroke of the trier, in such a manner that the thickness of the layer diminishes from about 0.5 cm. on the middle of the plate to a thin film at the edge, and the line of demarcation between the two flours is distinct. Cut off the edges of the layer with the trier, so as to form a rectangle, and compare the color of the two flours. The difference in color becomes more apparent after carefully immersing the plate with the flour in water, and still more apparent after drying.

**Gasoline Color Value.**—*Winton Method.*—Place 20 grams of the flour in a wide-mouthed glass-stoppered bottle of about 120 cc. capacity and add 100 cc. of colorless gasoline. Stopper tightly and shake vigorously for five minutes. After standing sixteen hours, shake again for a few seconds until the flour has been loosened from the bottom of the bottle and thoroughly mixed with the gasoline, then filter immediately through a dry 11-cm. paper, previously fitted to the funnel with water and thoroughly dried, into a flask, keeping the funnel covered with a watch-glass to prevent evaporation. In order to secure a clear filtrate, a certain quantity of the flour should be allowed to pass over on to the paper and the first portion of the filtrate passed through a second time.

Determine the color value of the clear gasoline solution in a Schreiner colorimeter, using for comparison a 0.005% water solution of potassium chromate. This solution corresponds to a gasoline number of 1.0 and may be prepared by making 10 cc. of a 0.5% solution up to one liter. The colorimeter tube containing the gasoline solution should first be adjusted so as to read 50 mm., then the tube containing the standard chromate solution raised or lowered until the shades in both tubes match. The reading of the chromate solution, divided by the reading of the gasoline solution, gives the gasoline color value.

**Absorption and Dough Test.**—Stir 30 grams of the flour in a heavy coffee cup with 15 cc. of water by means of a spatula until a smooth ball of dough has been formed. If after standing two minutes, the amount of water proves insufficient to thoroughly dough up the flour, repeat the operation, using 15.5 cc. of water, and, if necessary, continue to repeat until the quantity is found that will yield a stiff, but thoroughly elastic dough. From the results of this test, calculate the absorption of 1000 grams of flour in terms of cc. of water.



The physical characters of the dough, such as color and elasticity, furnish valuable indications of the quality or grade of the flour.

**Expansion of Dough.**—Rub to a smooth paste 3.5 grams of granulated sugar, 1.2 grams of salt, and 3 grams of compressed yeast, and thoroughly mix with 60 cc. of water at 35° C. Warm 100 grams of the flour in a shallow pan to 35° C., add to it the yeast mixture, mix with a spatula, and knead with the fingers until a smooth ball of dough has been formed. Drop the dough into a graduated, 500-cc. cylinder, pat down so as to force out the air, and note the volume of the mass. Place in a raising closet kept at 35° C. Read the volume at the end of the first hour and every half hour thereafter until the maximum is reached.

**Baking Tests.**—These should be carried out in such a manner as to produce the best results with the kind of flour used and the purpose for which it is intended—whether for yeast bread, soda-biscuit, pastry, or crackers. Only tests for bread flours are here taken up, although methods for flours designed for other purposes may be easily devised to suit the conditions.

In judging the loaf the chief points are volume, shape, flavor, odor, texture, and color.

Two types of bread baking tests are in general use, (1) the long fermentation, or more accurately the short sponge, straight dough method, and (2) the straight dough method, best known in the form described by John Koelner of Milwaukee who also supplies a special kneader and other apparatus.

*Apparatus.*—1. *The Proofing or Raising Closet* is a kind of incubator with double doors, provided with one or more electric lamps or other heating device to keep the temperature at about 32° C.

2. *Kneader.*—Hand kneading is recommended for the long fermentation process. The combined mixer and kneader supplied by Koelner is used with his process.

3. *Baking Tins* for the long fermentation method are 16.8×8.8 cm. at the top, 14.9×6.9 cm. at the bottom, and 13.9 cm. deep (inside measurements) with aprons on both sides to prevent the falling over of the loaf. Those for the Koelner method are 27×6.3 cm. at the top, 25.4×5 cm. at the bottom, and 8.8 cm. deep (inside measurements) and do not have aprons.

4. *Oven.*—The best form is an electric oven provided with three heats (low, medium, and full); a gas or kerosene oil oven may also be used.



A thermometer passes through a hole in the top so that the bulb is on a level with the baking tins and the portion of the graduation showing the desired temperature is outside of the oven.

5. *Volume Measure*.—For determining the size of the loaf a tight box is provided of such a size as to hold the largest loaf obtained by the method with which it is used, but no larger. The box is first filled with flaxseed without jarring, the excess struck off by means of a straight edge, and the seed weighed. The capacity of the box (in cc. or cu. in.) is then divided by the weight of the seed (in grams or pounds) thus obtaining a constant showing the value of the unit in weight in terms of volume. In actual test the loaf is introduced into the box, flaxseed added until full, the excess struck off, and the weight of the seed determined. This weight multiplied by the constant obtained above gives the volume of the seed which, subtracted from the total volume of the box, gives the volume of the loaf.

*Long Fermentation Method*.—The process in general is as follows: Weigh out 340 grams of flour and 6 grams of shortening into a mixing bowl and place in the proofing closet together with a thorough mixture of yeast, sugar, salt, and water sufficient to make a moderately stiff dough as calculated from the absorption. After 30 minutes prepare a dough from the flour, shortening, and yeast mixture and place in the proofing closet, where it is kept for 50 minutes. Pull, knead, return to the proofing closet, and allow to raise for about 40 minutes. Knead and pull a second time, place in tins, keep for 30–50 minutes in the proofing closet, and finally bake at 200–205° C.

While the proportions for the yeast mixture must be varied to suit the conditions the following will serve as a guide: fresh compressed yeast 9 grams, salt 3.5 grams, sugar 12 grams, water as calculated from the absorption for 340 grams of flour.

Snyder, who has had a wide experience in scientific and commercial flour testing, observes:

“In baking tests the method of procedure must be appreciably varied to correspond with the individual characteristics of the flour that is being tested. Uniform and inflexible methods of making baking tests that are alike applicable to all kinds of flours cannot be given, as a method that would give the best baking results with one type or kind of flour often gives very poor results when applied, unmodified, to another kind or type of flour. Each flour tested is entitled to the quantity and kinds of ingredients and method of manipulation as may be necessary to produce



the best loaf of bread from a physical point of view, that the flour is capable of making.

"Some flours require more and some less of the sponge to make the right kind of a dough, Then, too, it may be necessary to slightly vary the amounts of yeast, salt, sugar, etc., in order to get the best baking results from flours made from different kinds of wheat.

"A standard flour of known baking character is usually put through the process at the same time the flour in question is being tested. Sometimes baking tests are defective because the yeast is old or of poor quality. The technical skill of the individual who makes a baking test is a matter of very great importance in assigning a proper value to the results."

*Straight Dough or Koelner Method.*—This process is useful in distinguishing certain grades of flour, such as patents and clears by the volume of the loaf, and in testing for flavor, but is limited in its value because the manipulation is not varied to suit the conditions.

Warm 220 grams of the flour, in a shallow pan in a raising closet kept at 35° C., transfer to a Koelner dough kneader, warmed to 35° C. by means of water placed in the special compartment, add 12 grams of sugar, 5 grams of salt, and 10 grams of compressed yeast, rubbed smooth in a cup with 100 cc. of water at 35° C. Rinse the cup with sufficient water to make the total quantity required, (usually about 87 cc.) as calculated from the absorption test. Adjust the blades of the kneader for mixing, and turn 90 revolutions per minute for 10 minutes. Adjust the blades for kneading, add 120 grams of flour at 35° C., and turn 60 revolutions per minute for 10 minutes. Remove the dough immediately to a warmed plate, cut into two equal parts, mould the two separately, and place end to end in a warmed, greased, and tared baking tin. Weigh the tin with dough, place a tin gauge across the top, and set the whole in the raising closet. After the dough has risen to the gauge bake at 200 to 205° C. until 30 grams of water have been removed, which usually requires from 30 to 35 minutes. Break the loaf in two, and note the odor when hot and again when cold, also the flavor when cold.

**Determination of Water, Fat, Fiber, and Protein.**—Employ the methods described on pages 285 to 287. The crude fiber should be collected and weighed on a Gooch crucible.

**Determination of Ash.**—Char 3-5 grams of the flour in a flat-bottomed platinum dish heated on a piece of thin asbestos board over a Bunsen burner. Complete the burning at dull redness, preferably in a muffle furnace. If the ash is black or dark gray add a few drops of nitric acid, evaporate



to dryness on a water-bath and again heat at dull redness, repeating the treatment if necessary.

**Determination of Moist and Dry Gluten.\***—Place 25 grams of the flour in a coffee cup, add 15 cc. of water at a temperature not to exceed 15°, and work the mass into a ball with a spatula, taking care that none of it adheres to the dish. Allow the dough to stand one hour, then knead in a stream of cold water over a piece of bolting cloth held in place by two embroidery hoops, until the starch and soluble matters are removed. Place the gluten thus obtained in cold water, and allow to remain for one hour, after which press as dry as possible between the hands, roll into a ball, place in a tared flat-bottomed dish, and weigh as moist gluten. Spread the gluten out in the dish, dry for 24 hours, in a boiling water-oven, and weigh again, thus obtaining the dry gluten.

Because of the inaccuracies of determining gluten by washing many cereal chemists employ only determinations of nitrogen calculating the protein therefrom by the factor 5.70.

**Determination of Alcohol-soluble Protein (Crude Gliadin).—Chamberlain Method.**—Digest 2.5 grams of the sample with 125 cc. of 70% (by vol.) alcohol for 24 hours, shaking every half hour during the first 8 hours. Filter through a dry paper, determine nitrogen in 100 cc. of the filtrate, and multiply the result by 5.7.

**Determination of Gliadin.—Snyder Method.†**—Place 15.97 grams of the flour and 100 cc. of alcohol (sp. gr. 0.90) in a 300-cc. flask, shake at intervals for 3 hours, and let stand over night. Filter through a dry filter and polarize in a 220-mm. tube. Precipitate the proteins in 50 cc. of the filtrate with 5 cc. of Millon's reagent, filter, and polarize in a 220-mm. tube, increasing the reading by 50%. Deduct the sum from the first polarization and multiply the difference by 0.2. The product is the per cent of nitrogen as gliadin, which, multiplied by 5.7, gives the per cent of gliadin.

**Olson Method.‡**—Digest 4 grams of the sample with 200 cc. of 50% (by vol.) alcohol for 2 hours, shaking at 5-minute intervals, let stand over night, filter, and determine nitrogen in 25 cc. of the clear filtrate. Evaporate slowly 50 cc. of the filtrate to within 5 cc., add 50 cc. of water, heat nearly to boiling, and evaporate to about 10 cc. Add another portion

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 81, 1904, p. 118.

† Jour. Amer. Chem. Soc., 27, 1905, p. 1068.

‡ Jour. Ind. Eng. Chem., 5, 1913, p. 917.



of 50 cc. of water and evaporate to 35 cc. or less., let cool to room temperature, filter, returning to the filter until clear, determine nitrogen in the filtrate, and subtract from the total alcohol-soluble nitrogen. The difference is gliadin nitrogen.

*Gliadin Ratio.*—Snyder considers the percentage of gliadin in the total protein (gliadin ratio) more significant than the gliadin-glutenin ratio of Fleurant.\*

**Determination of Salt-soluble Protein.**—*Chamberlain Method.*†—Digest 10 grams of the flour with 250 cc. of 5% potassium sulphate solution, as described under Alcohol-soluble Protein. Determine nitrogen in 50 cc. of the filtrate, and multiply the result by 5.7.

**Determination of Albumin, Globulin, and Amides.**—*Olson Method.*‡—Digest 10 grams of the sample with 500 cc. of 1% sodium chloride solution for 2 hours, shaking at 5-minute intervals, let stand over night in a cool place, and filter clear. Boil down 200 cc. of the filtrate to 20 cc. or less, and evaporate slowly to dryness on a hot plate. Digest the solid mass with 100-cc. portions of 55% (by vol.) alcohol, filter, and wash with 55% alcohol. Determine nitrogen in the precipitate and correct for blank, thus obtaining the albumin (leucosin) nitrogen.

Evaporate the alcoholic filtrate to within 10 cc., add 50 cc. of water, boil down to 35 cc., add 15 cc. of water, let cool to room temperature, filter, and wash with cold water. Treat the filtrate with phosphotungstic acid, filter, wash with water containing phosphotungstic acid, determine nitrogen in the precipitate, and correct for blank. This gives the globulin (edestin?) nitrogen.

Determine nitrogen in the filtrate from the phosphotungstic acid precipitate and correct for blank. The corrected result is regarded as amide nitrogen.

Olson calculates only the nitrogen in the various forms; the corresponding proteins may be obtained, using the appropriate factors. See page 286.

**Determination of Glutenin.**—Subtract the sum of the gliadin, albumin, globulin, and amide nitrogen from the total nitrogen and multiply the difference by 5.7.

**Determination of Water-soluble Nitrogen.**—*Rousseaux and Sirot*

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\* Manuel l'analyse chimique, 1898, p. 308.

† Loc. cit.

‡ Jour. Ind. Eng. Chem., 6, 1914, p. 211.



*Method.\**—Digest 10 grams of the sample in a 200-cc. graduated flask for 2–5 minutes in a boiling water-bath with 150 cc. of water, shaking frequently. Cool, make up to the mark, filter, and determine nitrogen in 50 cc. of the filtrate.

**Determination of Acidity of Flour.**—*Mitchell Method.†*—Weigh out 20 grams of the flour into an Erlenmeyer flask, add 200 cc. of distilled water at 40° C., free from carbon dioxide, shake vigorously for 5 minutes, and digest at 40° for one hour, shaking every 10 minutes. Filter and titrate 100 cc. of the filtrate with N/10 sodium hydroxide solution, using phenolphthalein as indicator. Calculate the acidity as per cent of lactic acid, (1 cc. N/10 NaOH = 0.009 gram).

If the distilled water used contains an appreciable amount of carbon dioxide, it should previously be boiled in a Jena flask until neutral, but not long enough to dissolve alkali from the glass. Two hundred cc. of the boiled water should remain colorless on addition of phenolphthalein, but should take on a distinct pink color when mixed with a single drop of tenth-normal alkali.

**Determination of Cold-water Extract.**—*Wanklyn Method.*—Mix 100 grams with distilled water in a graduated liter flask, shake frequently during 6 or 8 hours and allow to stand over night. Decant on a filter, rejecting the first portions that run through, and evaporate 50 cc. of the clear filtrate to dryness in a tared metal dish on a water-bath. The weight of the dried residue multiplied by 20 gives the cold-water extract which, according to Wanklyn, should not exceed 5%.

**Determination of Iodine Number of the Fat.**—Dry over sulphuric acid for three days sufficient flour to yield 0.2 to 0.25 gram of fat and extract for 16 hours in a Johnson extractor with 25 cc. of absolute ether, into a tared 35-cc. flask. Drive off the ether and dry at 100° C. for 15-minute periods to constant weight, passing a current of dry hydrogen through the flask. Proceed according to the Hanus method, adding the chloroform and iodine solution directly to the flask, and breaking the flask within a wide-mouthed glass-stoppered bottle for the final dilution and titration.

**Detection of Improvers.**—Phosphorus and sulphur compounds may be determined in the ash after burning with sufficient sodium carbonate to form sodium phosphate and sulphate, thus preventing loss by volatiliza-

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\* Compt. rend., 156, 1913, p. 723.

† U. S. Dept. Agric., Bur. of Chem., Bul. 122, 1909, p. 54.



tion. The well-known method of burning with magnesium nitrate is useful in fixing phosphorus compounds. Ready-formed sulphates may be determined in the solution obtained without burning by boiling with 2% hydrochloric acid. The dextrose formed by the hydrolysis of the starch does not interfere. See [page 362. Microchemical methods should also prove useful. The mechanical separation of certain inorganic salts by the chloroform method, page 336, is recommended. Because of the great variety of improvers which may be added, definite instructions are not possible.

**Detection of Alum.**—*Logwood Test.*—Mix 10 grams of the sample with 10 cc. of water and stir in 1 cc. of logwood tincture (5 grams logwood digested in 100 cc. alcohol) and 1 cc. of a saturated solution of ammonium carbonate. If the sample is pure, the color will be a faint brown or pink, but if alum is present, a distinct lavender-blue color is produced, which should remain after heating for 2 hours in the water-oven.

*Lenz Hematoxylin Test.*\*—Mix in a test tube 2 grams of the flour with 3 cc. of water and 1 cc. of 1% hematoxylin in 50% alcohol, then shake vigorously with 10 cc. of saturated salt solution. With pure flour the deposit which settles is flesh color, while with flour containing alum it is bluish or slate color.

*Borghesi Test.*†—Shake 10 grams of the flour 5–10 minutes with 100 cc. of water, filter, and precipitate the proteins in the filtrate with concentrated tannin solution. Filter again and add 2 drops of cochineal tincture or 1% alcoholic alizarin solution. If alum is present the solution takes on a crimson color with cochineal or orange-red with alizarin. The test is accurate to 0.2% of alum.

**Detection of Peroxide Bleaching in Flour.**—Place on the “slicked” surface of the flour a drop or two of a mixture of equal parts of solutions (a) and (b), described below. If the flour is unbleached and has not been stored under conditions permitting absorption of nitrous acid, the liquid, which does not immediately soak into the flour, will remain colorless or nearly so, while if it is bleached it soon takes on a marked pink or crimson color, varying in degree with the extent of bleaching. A positive test should be supplemented by determinations of nitrous nitrogen and gasoline color value, making suitable allowance if the flour has been aged.

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\* Apoth. Ztg., 26, 1911, p. 687.

† Giorn. farm. chim., 59, 1910, p. 49.



**Determination of Nitrous Nitrogen.**—*Griess-Ilosvay Method.*\*—This method, commonly employed for the determination of nitrites in water, is well adapted for the estimation of the extent to which flour has been bleached by nitrogen peroxide or nitrosyl chloride.

1. *Reagents.*—(a) *Sulphanilic Acid Solution.*—Dissolve 0.5 gram of sulphanilic acid in 150 cc. of 20% acetic acid.

(b) *Alpha-naphthylamine Hydrochloride Solution.*—Dissolve 0.2 gram of the salt in 150 cc. of 20% acetic acid with the aid of heat.

(c) *Standard Sodium Nitrite Solution.*—Dissolve 0.1097 gram of dry C.P. silver nitrite in about 20 cc. of hot water, add 0.05 gram of C.P. sodium chloride, shake until the silver chloride flocks and make up to 1000 cc. Draw off 10 cc. of the clear solution and dilute to 1 liter. One cc. of this solution contains 0.0001 mg. of nitrogen as nitrite.

Suitable silver nitrite is on the market; it may also be prepared as follows: mix a warm concentrated solution of 8 parts of sodium nitrite with a warm concentrated solution of 16 parts of silver nitrate. When cool collect the precipitate on a Büchner funnel and wash with cold water. Dry quickly on a water-bath with as little exposure to light as possible. Long continued drying at 100° C. causes it slowly to decompose.

2. *Determination.*—Weigh out 20 grams of the flour into an Erlenmeyer flask, add 200 cc. of water free from nitrites, previously heated to 40° C., close the flask with a rubber stopper, shake vigorously for 5 minutes, digest 1 hour at 40°, shaking every 10 minutes, and filter on a dry folded filter free from nitrites. As the first portion of the filtrate is usually turbid, it should be returned to the filter and the operation repeated until a clear liquid is secured. Dilute 50 cc. of the filtrate and also 50 cc. of the standard nitrite solution each with 50 cc. of water, add 2 cc. each of solutions (a) and (b); shake and allow to stand one hour to bring out the color. Compare the two solutions in a colorimeter. Divide the height of the column of the standard solution by that of the solution of the sample, thus obtaining the parts of nitrogen as nitrous acid (free or combined) per million of flour.

**Detection of Chlorine Bleaching in Flour.**—Determine the gasoline color value (page 327), the iodine number of the fat (page 333), and the chlorine in the fat expressed in terms of mgs. per kilo. If the bleaching is slight, the iodine number will not be reduced sufficiently to detect the bleaching; when, however, the bleaching is considerable the iodine number

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\* Bull. chim. [2], 2, p. 317.



may be reduced ten points or even more. Whenever possible comparisons should be made with unbleached flour of the source and grade of the suspected sample.

**Determination of Chlorine in the Fat.**—Dry at 100° C. for a few hours a portion of the flour sufficient to yield about 0.2 gram of fat and extract the nearly dry residue with absolute ether until most of the fat is removed. Evaporate the ether extract in a platinum dish with sodium carbonate free from chlorine and ignite at dull redness. Take up in cold water, add a slight excess of nitric acid, filter, wash, and determine chlorine by Volhard's method, using diluted standard solutions in order to insure greater accuracy. Precaution must be taken to use chlorine-free reagents and filter paper, washing the latter if necessary, and avoid possible contamination from the air and other sources.

**Bamihl Test for Gluten** (*Modified by Winton* \*).—This test serves to detect wheat flour mixed with rye and other flours.

Place a very small quantity of the flour (about 1.5 milligrams) on a microscope slide, add a drop of water containing 0.2 gram of water-soluble eosin in 1000 cc., and mix by means of a cover glass, holding the latter at first in such a manner that it is raised slightly above the slide, and taking care that none of the flour escapes from beneath it. Finally allow the cover glass to rest on the slide, and rub it back and forth until the gluten has collected into rolls. The operation should be carried out on a white paper so that the formation of gluten rolls can be noted.

Wheat flour or other flours containing it yields by this treatment a copious amount of gluten, which absorbs the eosin with avidity, taking on a carmine color. Rye and corn flour yield only a trace of gluten, and buckwheat flour no appreciable amount. The preparations are best examined with the naked eye, thus gaining an idea of the amount of gluten present. Under the microscope traces of gluten, such as are formed in rye flour, are so magnified as to be misleading.

In case the flour is coarse, or contains a considerable amount of bran elements, as is true of buckwheat flour and low grade wheat flour, the test should be made after bolting, as the bran particles and coarse lumps interfere with the formation of gluten rolls.

**Chloroform Test.**—This serves in separating mineral impurities and improvers; also aids in distinguishing wheat and rye flour.

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 122, p. 217.



Beneke \* shakes vigorously 1 part of the flour with 3-4 parts of chloroform, then adds 1-2 parts more of chloroform, shakes again, and examines the deposit after 24 hours. Dirt and added mineral matter first settle, then organic matter, chiefly aleurone cells, gradually deposits. The aleurone cells of rye are blue or olive green, those of wheat yellow-brown. Chemical tests should be made of any mineral deposit other than dirt and microscopic examination of the organic deposit.

The sedimentation apparatus devised by Spaeth will be found convenient. The settlings collect in the half-bored stop-cock, which when closed permits the pouring off of the supernatant liquid.

**Microscopic Examination.**—General instructions are given on page 314. In distinguishing rye flour from wheat flour whether occurring singly or in mixture, the difference in the cross cells (pages 315-317) should be especially noted; these, however, are present in considerable amount only in the cheaper grades of wheat flour. The Bamihl and chloroform tests furnish supplementary information.

#### CORN (MAIZE) MEAL.

**Process of Manufacture.**—Although the production of Indian corn is greatest in the "corn belt" of the Middle West, the consumption of corn meal and grits as human food is greatest in the Southern States of the Union where in certain sections it forms the chief article of diet. The Southern mills follow the old-time stone process and the meal consists of the kernel ground entire with or more often without the removal of a portion of the bran. In the Northern mills, however, the roller system is employed, the germ and other offal being commonly removed by special machinery preliminary to grinding. The milling and bolting processes are usually so conducted as to secure in addition to meal a large yield of grits or hominy used both in brewing and as a table cereal, also a certain amount of corn flour which is utilized in pancake mixtures and as a filler for sausage. Drying is usually essential to prevent spoilage.

Stone-ground whole-kernel meal is preferred by many in the South because of its characteristic oily flavor, but degerminated and bolted meal, such as is obtained by the roller process, has better keeping qualities owing to the smaller amount of oil.

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\* Land. Vers. Stat., 36, 1889, p. 337.



**Composition of Corn Meal.**—The composition of whole kernel meal is the same as of the corn from which it is ground except for loss of a portion of the moisture if the corn was excessively moist. The range in composition of degerminated bolted meal from white and yellow corn as reported by Winton, Burnet, and Bornman \* is given below:

	Water.	Protein.	Fiber.	Nitrogen-free Extract.	Fat.	Ash.	Acidity.
White corn meal:							
Max.....	18.28	8.84	0.96	78.40	2.31	0.81	23.0
Min.....	11.97	5.81	0.58	72.41	0.70	0.35	10.6
Yellow corn meal:							
Max.....	17.85	8.63	0.76	76.64	1.81	0.65	19.7
Min.....	13.01	6.63	0.46	71.98	0.33	0.24	14.0

**Spoilage of Meal.**—Although the theory that the disease pellagra is caused by spoiled meal has been quite generally abandoned, it behooves the miller to grind sound corn and dry the meal and the dealer to market the product before it has become sour, moldy, or rancid.

Various Italian and Austrian authors lay stress on the determination of acidity and certain qualitative tests in detecting spoilage. Corn or meal with an acidity of more than 30 as determined by the following method is regarded by Schindler as unfit for consumption.

**Determination of Acidity.**—*Schindler Method.*†—Weigh 10 grams of the meal (which should pass a sieve of 20 meshes to the inch) into a 50-cc. glass-stoppered graduated flask, fill to the mark with 85% by volume neutral alcohol (prepared by distilling 95% alcohol with quicklime) and allow to stand 24 hours with occasional shaking. Decant the clear liquid onto a folded filter and pipette 25 cc. of the filtrate into a beaker. Add 100–150 cc. of well-boiled distilled water, a few drops of phenolphthalein solution and titrate with twentieth normal sodium or potassium hydroxide solution. Express the results in terms of cc. of normal alkali per kilo of meal.

#### BREAD AND CAKE.

Bread is a term broadly applied to all baked cereal products. Unleavened bread, pilot or ship biscuit, and corn pone or hoe cake, which

\* U. S. Dept. of Agric., Bul. 215, 1915.

† Anleitung zur Beurteilung des Maises und seiner Mahlprodukte mit Rücksicht auf ihre Eignung als Nahrungsmittel. Innsbruck, 1909, p. 37.



are the simplest forms of bread, are made entirely of cereal flour or meal, water and salt, the aeration being effected by the formation of steam and the expansion of inclosed air during heating in the oven. Air and steam also serve as the leavening agents for various dry biscuits or crackers, pie crust, puff paste and beaten biscuit, but in these more or less fat is used as shortening.

In a narrower sense bread is understood to mean the raised product rendered light and porous by a gas generated before or during baking. This gas is commonly carbon dioxide generated either by the fermentive action of yeast on the sugar of the dough, alcohol also being formed in the reaction, by the action of bacteria as in salt-rising bread, or by the action of acid ingredients on sodium bicarbonate. Leavening may also be brought about by ammonium carbonate from which ammonia gas and carbon dioxide are liberated during baking.

In making ordinary bread the flour is kneaded up with water or milk, salt, shortening, and yeast. Malt extract is also used by some bakers because of its diastatic action and flavor, and yeast foods to save yeast and conserve the carbohydrates. The dough is allowed to rise in a warm place, the gas formed during the fermentation converting the mass into a light sponge.

During the subsequent process of baking, which should take place at a temperature between  $230^{\circ}$  and  $260^{\circ}$  C., further expansion ensues, much of the water is driven off, and the porous mass sets to form the loaf, the outside of which is converted into a brown crust, due to the caramelizing of the dextrin and sugar into which the starch of the outer layers is converted. Among other changes that take place in the interior or "crumb" during baking are (1) the partial breaking up of the starch grains, which however, largely retain their identity, though in some degree distorted in shape; (2) somewhat obscure changes in the character of the proteins; and (3) partial oxidation of the oil or fat.

The well-made loaf should possess an agreeable odor, and a sweet, nutty flavor, entirely free from mustiness. It should not be tough or soggy on the one hand (due to under-raising), nor extremely dry and spongy on the other (indicative of over-raising). Over-raising, moreover, produces sourness, due to advanced lactic fermentation.

**Composition of Bread.**—The following table gives the average composition of bread from Bul. 13, part 9, of the Bureau of Chemistry:



Kind of Bread.	No. of Samples.	Moisture	Protein, N $\times 6.25$ .	Ether Extract.	Crude Fiber.	Salt.	Ash.	Carbohydrates, Excluding Fiber.	Calculated Calories of Combustion.
Vienna.....	10	38.71	8.87	1.06	0.62	0.57	1.19	53.72	4435
Home-made..	2	33.02	7.94	1.95	0.24	0.56	1.05	56.75	4467
Graham.....	9	34.80	8.93	2.03	1.13	0.69	1.59	53.40	4473
Rye.....	7	33.42	8.63	0.66	0.62	1.00	1.84	56.21	4338
Miscellaneous.	9	34.41	7.60	1.48	0.30	0.49	1.00	56.18	4429
Biscuits.....	48	7.13	10.34	8.67	0.47	0.99	1.57	73.17	4755
Rolls.....	11	27.98	8.20	3.41	0.60	0.69	1.31	59.82	4538

The table which follows is a summary of analyses of bread from cheaper bakeries made in the author's laboratory:

Kind of Bread.	No. of Analyses.	Weight of Loaf in Grams.	Water, Per Cent.	Per Cent Ash in Terms of Solids.	Acidity.*
White.....	44				
Maximum.....	..	653	45.20	1.83	6.2
Minimum.....	..	126	33.00	0.60	1.3
Mean.....	..	430	40.72	0.85	2.6
Graham.....	7				
Maximum.....	..	500	45.20	1.55	4.2
Minimum.....	..	367	40.10	0.96	2.1
Mean.....	..	420	41.50	1.26	3.5
Whole wheat.....	1	507	45.10	1.20	
Diabetic.....	1	445	47.00	2.20	
Muffins.....	1	194	48.20	1.15	1.7
Rye.....	1	1291	47.15	2.13	10.0
"Black".....	1	550	47.00	2.20	
German with seeds.....	1	417	42.30	0.95	
Brown.....	1	500	48.10	3.50	
"Knackerbrod".....	1	110	8.00	1.94	

\* Cubic centimeters of tenth-normal soda required to neutralize 10 grams of the fresh bread.

The physical characteristics of bread, its color, taste, odor, porosity, etc., together with determination of moisture, ash, and acidity will usually enable the analyst to pass judgment on its quality.

**Water in Bread.**—The amount of water is of considerable importance, and, in the best bread, varies from 33 to 40 per cent. A larger content of water than 40% should be considered objectionable in a white bread, both on the ground of acting as a make weight, and because a large excess of moisture tends to cause the growth of mold.

**Acidity of Bread.**—The degree of sourness of a sample of bread, if made from sound flour, indicates the extent to which the fermentation of the dough has been carried. To neutralize the acidity of 10 grams of the normally sweet loaf of wheat bread, an average of 2 cc. of the standard



alkali solution is required, corresponding to 0.72 gram of lactic acid per loaf of an average weight of 400 grams. The loaf of rye bread exhibiting the maximum sourness or acidity in the above table required 10 cc. of standard alkali per 10 grams of bread, corresponding to 11.61 grams lactic acid in the loaf of 1291 grams. Rye bread, particularly the coarse variety known as "*Pumpernickel*," is often so prepared as to be sour to the taste.

Congdon obtained an average acidity equivalent 2.19 cc. N/10 alkali per 10 grams in the crust of bakers' bread and 1.78 cc. in the crumb. In home-made bread he found 1.6–2.1 cc. for the crust and 1.25–1.45 cc. for the crumb. He believes the acidity to be due partly to lactic acid (about 0.03%) produced during fermentation which reacts to form lacto acid phosphate and phosphoric acid, and partly to hydrochloric acid (about 0.10%) formed by the action of phosphoric acid on sodium chloride. White, also Barnard and Bishop (page 342) have found that the acidity does not increase on keeping the bread.

**Fat in Bread.**—It is well known that the amounts of fat or ether extract as obtained by the ordinary method and expressed in most bread analyses are too low, being considerably less than the combined fat of the materials entering into its composition. This is probably due to the partial oxidation of the fat and its incrustation with insoluble matter.

**Yeast Foods.**—Kohman, Hoffman, Godfrey, Ashe, and Blake,\* in extensive investigations carried out at the Mellon Institute under fellowships of the Ward Baking Co. found that in different regions, owing to variations in the composition of the water, marked differences in the action of yeast, and consequently in the character of the bread were obtained. When, however, they added to the quantity of flour (100 lbs.) required for making 160 lbs. of bread, 2 ounces of calcium sulphate, 1 ounce of ammonium chloride, and 0.02 ounce of potassium bromate, a saving of 50–65% of the yeast and 2% of fermentable carbohydrates (calculated in terms of the flour) was effected, the quality of the dough was conserved, the control of the process was facilitated, and better and more uniform bread was secured. The calcium and ammonium salts act as yeast foods while the bromate exerts an oxidizing influence, aging and maturing the dough and preventing so-called "rottenness" which reduces the

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\* Jour. Ind. Eng. Chem., 8, 1916, p. 781.



gas-retaining qualities. The composition of the bread was not appreciably different from bread made without the salts, except that the lime was slightly higher.

**Alum** was formerly added to flour by the baker as well as the miller to cover defects or spoilage, influencing the quality of the gluten. Its use in the United States, at least in recent years, has not been reported. In examining flour or oven products for alum due regard must be given its possible presence in the form of baking powder.

**Copper Sulphate**, said to have been used many years ago on the continent, does not appear to have ever found a place in English or American bakeries.

**The Wrapping of Bread** is in accord with modern ideas of cleanliness and sanitation. Winton has seen the baker's delivery man drop an unwrapped loaf in the filth of a city alley and restore it to its place in his wagon. Had the loaf been wrapped, possible consequences would have been mitigated. The bread is commonly allowed to cool before wrapping.

An exhaustive investigation of the effects of wrapping on the character and composition of the bread has been conducted by Barnard and Bishop.\* They concluded (1) that wrapping either in semi-porous waxed or paraffined paper retards the escape of moisture and tends to preserve the colloidal condition and physico-chemical equilibrium and thus prevent staling, also, contrary to common belief, the loss of moisture of the crumb is accompanied by a closely parallel loss of the crust, (2) lactic acidity does not develop either in wrapped or unwrapped bread, thus confirming the results of White,† and (3) wrapping does not injure the quality of the loaf after the third day and up to that time improves its condition, flavor, and odor.

**Cake and Similar Preparations.**—These differ from bread chiefly by the addition of considerable sugar, butter, spices, and other flavoring materials. In gingerbread, molasses is used as an important ingredient besides ginger. The adulterants of molasses, such as glucose, salts of tin, etc., would thus sometimes occur in gingerbread. In fact stannous chloride has been found in ginger cakes.‡

The following analyses of a few typical varieties of cakes are selected from Bulletin 13 of the Bureau of Chemistry:

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\* Jour. Ind. Eng. Chem., 6, 1914, p. 736.

† N. Dak. Agric. Exp. Sta., Spec. Bul. 26, 1910, p. 214.

‡ See U. S. Dept. of Agric., Bur. of Chem., Bul. 13, p. 1369.



Kind of Cake.	Moisture	Protein. N X 6.25.	Ether Extract.	Crude Fiber.	Ash.	Salt.	Sugar.	Other Carbohy- drates.	Cal- culated Calories.
Doughnuts...	21.61	6.73	19.33	0.60	0.40	0.03	1.28	50.64	5529
Ginger snaps...	4.86	6.06	15.44	0.79	1.82	0.47	28.66	24.90	4971
Fruit cake...	24.47	4.56	12.35						
Gingerbread...	21.49	6.25	8.42	0.90	1.55	0.28	9.48	52.46	4757
Cup cakes...	14.81	5.24	15.56	0.27	1.21	0.34	32.48	30.89	5073
Macaroons...	8.06	6.67	12.97	1.41	0.82	0.07	58.77	10.89	4835
Jumbles.....	13.34	7.62	14.79	1.04	0.97	0.39	16.60	46.31	5133

## METHODS OF ANALYSIS.

**Preparation of the Sample.**—Weigh a whole loaf, divide into quarters by cutting through the center at right angles to the sides and ends, weigh one of the quarters, break up into pieces, dry at a moderate heat, grind without loss, and weigh, and calculate the loss of moisture. A more representative sample is secured by using quarters of several loaves. If desired, crust and crumb may be prepared separately.

**Determination of Moisture.**—Proceed as with flour, taking into account the moisture driven off in preparing the sample.

**Determination of Fat.**—Owing to the presence of gelatinized starch, which incloses the fat globules, direct extraction yields results much too low. Doubtless some, if not all the results for fat given in the tables, page 340 and above, were obtained by faulty methods.

*The Polenske-Grujič Method*\* gives the full amount of fat: Heat on a boiling water-bath for 90 minutes 5 grams of the sample in a 200-cc. flask with 50 cc. of water and 2 cc. of 25% hydrochloric acid (sp. gr. 1.125), cool, add 1 cc. of 0.04% methyl orange solution, neutralize with concentrated sodium hydroxide solution, and acidify with a drop of dilute hydrochloric acid. Filter, wash with hot water, dry filter and contents on a watch-glass at 105° C., and extract with ether in the usual manner.

**Determination of Fiber, Starch, Sugars, Protein, and Ash.**—See general methods (pages 285 and 286), also pages 292 and 293.

## YEAST.

The yeast plant is a fungus of the genus *Saccharomyces*, widely distributed through the vegetable kingdom and in the air. It is capable of rapid growth by the multiplication of its cells when present in a

\* Eighth Int. Cong. App. Chem., 26, 1912, p. 1.



favorable medium, such as malt wort, and with propitious conditions of temperature, moisture, etc. Under such conditions, it forms a yellowish, viscous, frothy substance, the chief value of which, in the liquor industry, is the production of alcohol, while for bread-making, as a result of the same kind of fermentation, the end desired is the leavening of the doughy mass by the carbon dioxide liberated.

A vigorous, pure yeast which will "raise" quickly is a great preventive against sour bread, for not only is it comparatively free from the germs and products of lactic acid fermentation, but by doing its work quickly it enables the baker to check the fermentation or raising process before the lactic acid or sour decomposition is far advanced.

Yeast most commonly used in bread-making is of the so-called "compressed" variety. The use of compressed yeast is almost universal for domestic purposes, and is more or less common in bakeries. A small amount of brewers' yeast in liquid form from beer wort is used, especially in the immediate neighborhood of breweries, and dry yeasts are used to some extent in localities so remote that fresh compressed yeast cannot readily be obtained.

**Compressed Yeast** is a product of distilleries where the wort from malt and raw grain is fermented for the manufacture of whiskey, gin, and other distilled liquors, as well as distilled vinegar. Little, if any, of the commercial compressed yeast is made from beer wort yeast.

In the manufacture of compressed yeast, the yeast floating on the top of the wort is separated by skimming, while that settling to the bottom is removed by running the wort into shallow settling trays. Top yeast is considered more desirable than bottom yeast for bread-making. The separated yeast is washed in cold water, and impurities are removed, either by sieving through silk or wire sieves, or by fractional precipitation while washing. The yeast, with or without the addition of starch, is finally pressed in bags in hydraulic presses, after which it is cut into cakes packed in tin-foil, and kept in cold storage till distributed for use.

Such yeast should be used when fresh, as it readily decomposes and soon becomes stale. When fresh, it should have a creamy, white color, uniform throughout, and should possess a fine, even texture; it should be moist without being slimy. It should quickly melt in the mouth without an acid taste. Its odor is characteristic, and should be somewhat suggestive of the apple. It should never be "cheesy," such an odor indicating incipient decomposition, as does a dark or streaked color.

**Dry Yeast** is prepared by mixing fresh yeast with starch or meal,



molding into a stiff dough, and drying, either in the sun or at a moderate temperature under reduced pressure. Such yeast, when dry, is cut into cakes and put in packages. It will keep almost indefinitely. During the drying process, many of the yeast cells are rendered torpid and temporarily inert, and for this reason the dried yeast does not act so promptly in leavening as does compressed or brewers' yeast, but when once it begins to act it is quite as efficacious.

**Composition of Yeast.**—The following is the result of the analysis of under-fermentation yeast, after drying, by Nägele and Loew:

Cellulose and Mucilage . . . . .	37
Albuminoids (Mycroprotein, etc.) . . . . .	36
Albuminoids (Soluble in Alcohol) . . . . .	9
Peptones (Precipitable by Subacetate of Lead). . . . .	2
Fat . . . . .	5
Extractive Matters (Leucin, Glycerin, etc.) . . . . .	4
Ash. . . . .	7
	<hr/>
	100

Lintner gives the following average analyses of the ash of three samples of yeast, analyzed by him:

Silica . . . . .	1.34
Iron ( $\text{Fe}_2\text{O}_3$ ) . . . . .	0.50
Lime ( $\text{CaO}$ ) . . . . .	5.47
Sulphuric anhydride ( $\text{SO}_3$ ) . . . . .	0.56
Magnesia ( $\text{MgO}$ ) . . . . .	6.12
Phosphoric Anhydride ( $\text{P}_2\text{O}_5$ ) . . . . .	50.60
Potash ( $\text{K}_2\text{O}$ ) and a little Soda . . . . .	33.49
	<hr/>
	98.08

Matthews and Scott give the following as the ash composition of yeast:

Potassium Phosphate . . . . .	78.5
Magnesium Phosphate . . . . .	13.3
Calcium Phosphate . . . . .	6.8
Silica, Alumina, etc . . . . .	1.4
	<hr/>
	100.0



**Starch in Compressed Yeast.**—Potato, corn, or tapioca starch has long been added to yeast before pressing, on the ground that the starch acts as a drier, producing a much cleaner product, and one that can be more readily and intimately mingled with the materials of the bread, besides enhancing the keeping qualities of the yeast, especially in warm weather. The quantities used vary from about 5% up to over 50%. Undoubtedly the larger amounts are added as a make weight. Some manufacturers use no starch whatever.

The question has frequently been raised whether, with improved methods of manufacture, whereby yeast can be produced comparatively free from slime, and thus capable of pressure without the admixture of starch, the use of the latter should not be considered as an adulterant.

Briant claims that the admixture of starch up to 5% increases rather than decreases the actual content of yeast, in that the starch abstracts moisture from the yeast cells themselves, the proportion of water being much smaller, and that of the yeast larger in the starch-mixed substance. T. J. Bryan,\* on the other hand, finds that the addition of starch to yeast reduces the carbon dioxide value, and that the percentage reduction is greater than the percentage of starch present. His experiments further indicate that the keeping qualities of starch yeast are not greater, but actually less than that of pure yeast.

**U. S. Rulings.**†—1. The term "compressed yeast," without qualification, means distillers' yeast without admixture of starch.

2. If starch and distillers' yeast be mixed and compressed such product is misbranded if labeled or sold simply under the name "compressed yeast." Such a mixture or compound should be labeled "compressed yeast and starch."

3. It is unlawful to sell decomposed yeast under any label.

#### ANALYSIS OF YEAST.

**Microscopical Examination.**—Mix a bit of the yeast in water on the glass slip till a milky fluid is formed, and stir in a drop of a very weak anilin dye solution, such as methyl violet, eosin, or fuchsin (1 gram crys. fuchsin, 160 cc. water, 1 cc. alcohol). Put on the cover-glass and examine under the microscope. Living, active cells resist the stain, if the latter is dilute enough, and appear colorless or nearly so, while the decayed and

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 116, 1907, p. 25.

† Food Insp. Decision, 111, 1910.



lifeless cells are stained and can easily be distinguished by their color. Yeast cells are circular or oval in shape and vary from 0.007 to 0.009 mm. in diameter. They are sometimes isolated and sometimes grouped in colonies; each cell has an outer, mucilaginous coating or envelope. The interior, granular mass or substance of the cell is the protoplasm, and within the protoplasm are frequently seen one or more circular empty spaces known as vacuoles.

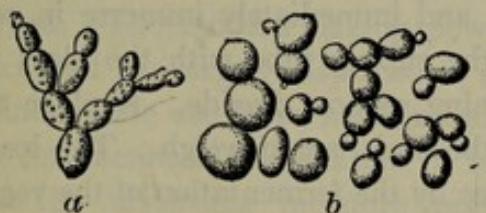


FIG. 68.—Sprouting Yeast-cells (*Saccharomyces cerevisiae*). (a, after Lürssen; b, after Hansen).

Yeast cells multiply by the process of budding. The decadence of yeast cells is marked by the increased size of vacuole and by the thickening of the cell wall.

**Determination of Leavening Power.**—The value of yeast in bread-making depends on the amount of carbon dioxide which it is capable of generating under given circumstances, hence the available carbon dioxide is the chief factor in gauging a yeast. There are various methods of determination: (1) either by measuring the volume of gas set free by the action of a weighed quantity of yeast in a sugar solution of known strength, kept for a fixed time at a fixed temperature (say 30°), or (2) by conducting the gas from such a fermenting solution through a weighed absorption bulb, containing potassium hydroxide and noting the increase in weight, or (3) by the more convenient method of Meissl as follows:

A mixture is made of 400 grams pure, concentrated sugar, 25 grams ammonium phosphate, and 25 grams potassium phosphate. A small wide-mouthed flask of about 100 cc. capacity (Fig. 69) is fitted with a doubly perforated rubber stopper, having two tubes, one of which is bent and passes nearly to the bottom of the flask, being fitted at the outer

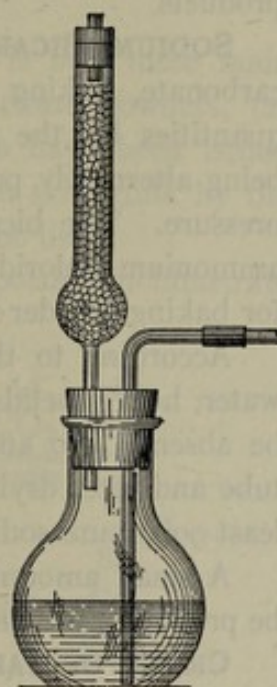


FIG. 69.—Apparatus for Determining Leavening Power of Yeast.



end with a rubber tube and glass plug, while the other is connected with a small calcium chloride tube. Measure 50 cc. of distilled water into this flask, and dissolve 4.5 grams of the above sugar phosphate mixture. Finally add 1 gram of the yeast to be tested, stir it well till there are no lumps, and cork the flask. Carefully weigh on a delicate balance the flask with its contents, and immerse in a water-bath at 30° C., keeping it at that temperature for 6 hours. At the end of this time, remove the flask from the bath, and immediately immerse in cold water to cool the contents. Remove the rubber tube with the glass plug, and by suction draw out the remaining carbon dioxide. Replace the plug, and having carefully wiped off the flask, again weigh. The loss in weight is due to carbon dioxide set free by the fermentation of the yeast.

#### CHEMICAL LEAVENING MATERIALS.

Under this heading are included the various chemicals, added separately, or mixed in the form of baking powder, for the aeration of oven products.

**SODIUM BICARBONATE.**—Sodium hydrogen carbonate, acid sodium carbonate, baking soda, or saleratus ( $\text{NaHCO}_3$ ), is produced in large quantities by the Solvay process, ammonia and carbon dioxide gases being alternately passed into concentrated sodium chloride solution under pressure. The bicarbonate, being almost insoluble in cold concentrated ammonium chloride solution, separates out. As placed on the market for baking powder it is a white powder with a disagreeable alkaline taste.

According to the United States Pharmacopœia, matter insoluble in water, heavy metals, and appreciable amounts of normal carbonate should be absent. No ammonia fumes should be evolved on heating in a test tube and after drying over sulphuric acid the powder should consist of at least 99% pure sodium bicarbonate.

A small amount of harmless impurity, such as sodium chloride, may be present. Because of its cheapness there is no incentive to adulteration.

**CREAM OF TARTAR**, or potassium bitartrate ( $\text{KH}_5\text{C}_4\text{O}_6$ ), is obtained by the recrystallization of crude argols. The lees, or argols, consist chiefly of crude potassium bitartrate, which is present in the juice of the grape, but being insoluble in alcohol is deposited during fermentation. If the wine has been plastered calcium tartrate will be found in the lees and also, if not eliminated, in the cream of tartar.

Calcium acid phosphate, gypsum, starch, and alum have been used



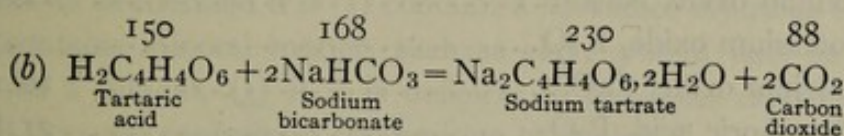
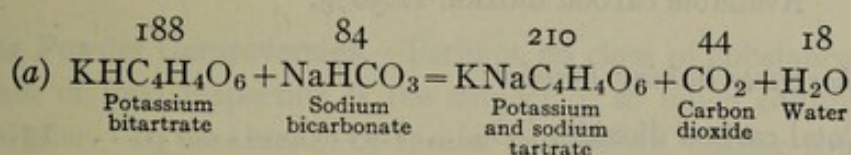
as adulterants, while small amounts of lead from the tanks in which the cream of tartar is crystallized constitute a common impurity.

Potassium bitartrate is insoluble in alcohol, sparingly soluble in cold, and readily soluble in hot water. It is usually guaranteed 99% pure.

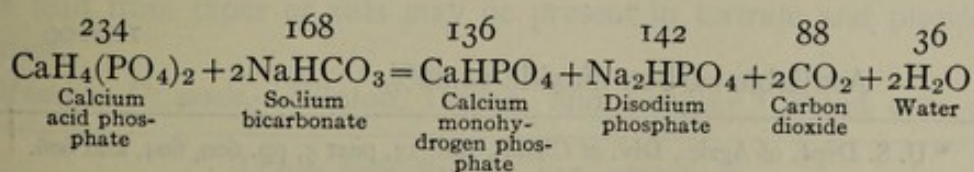
**BAKING POWDER.**—Formerly the housewife was accustomed to measure out in proper proportion an acid substance, such as sour milk, molasses, or cream of tartar, and sodium bicarbonate ("saleratus") to produce quick aeration of bread. The modern baking powder is a natural outgrowth of this practice and has largely displaced it, containing, as it does, a mixture ready for immediate use of an acid and an alkaline constituent in proper proportion for chemical combination to form the gas. A dry, inert material, which by absorbing moisture prevents the premature chemical action between the reagents, is generally considered an essential ingredient. Starch is nearly always preferred for this purpose, though sugar of milk has a limited use, and hydrogenated oil, which serves also as shortening has recently been proposed. The alkaline principle of nearly all baking powders is sodium bicarbonate, in some cases mixed with a little ammonium carbonate.

**Classification of Baking Powders.**—The division is into three main classes, with reference to the acid principle: (1) tartrate powders, (2) phosphate powders, and (3) alum powders. Patents have been issued for the use in baking powder of lactic acid made from skim milk by the action of *B. Bulgaricus*, thus adding a fourth class to the list.

(1) *Tartrate Powders.*—The acid principle is (a) potassium bitartrate or (b) tartaric acid, the reactions being as follows:

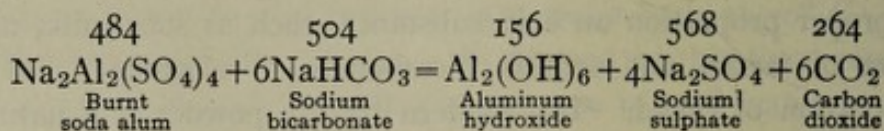


(2) *Phosphate Powders* contain calcium acid phosphate as the acid principle:





(3) "*Alum Powders.*"—In these the acidity is due to aluminum sulphate added as such or as potash-, ammonia-, or soda-alum. At present calcined or "burnt" soda alum, known commercially as S. A. S. (sodium aluminum sulphate) is almost exclusively used, the reaction being as follows:



Baking powders consisting of various mixtures of the above classes are also on the market. Alum-phosphate powders are especially popular.

**Composition of Various Baking Powders.**—Following are analyses of typical baking powders of the above classes: \*

1. *Cream of Tartar Baking Powder:*

Total carbon dioxide, CO <sub>2</sub> . . . . .	13.21
Sodium oxide, Na <sub>2</sub> O . . . . .	13.58
Potassium oxide, K <sub>2</sub> O . . . . .	14.93
Calcium oxide, CaO . . . . .	.18
Tartaric acid, C <sub>4</sub> H <sub>4</sub> O <sub>5</sub> . . . . .	41.60
Sulphuric acid, SO <sub>3</sub> . . . . .	.10
Starch . . . . .	7.42
Water of combination and association by difference..	8.98
	<hr/>
	100.00

Available carbon dioxide 12.58%.

2. *Phosphate Baking Powder:*

Total carbon dioxide, CO <sub>2</sub> . . . . .	13.47
Sodium oxide, Na <sub>2</sub> O . . . . .	12.66
Potassium oxide, K <sub>2</sub> O . . . . .	.31
Calcium oxide, CaO . . . . .	10.27
Phosphoric acid, P <sub>2</sub> O <sub>5</sub> . . . . .	21.83
Starch . . . . .	26.41
Water of combination and association by difference..	15.05
	<hr/>
	100.00

Available carbon dioxide 12.86%.

\* U. S. Dept. of Agric., Div. of Chem., Bul. 13, part 5, pp. 600, 604, and 606.



3. *Alum Baking Powder:*

Total carbon dioxide, $\text{CO}_2$ .....	9.45
Sodium oxide, $\text{Na}_2\text{O}$ .....	9.52
Aluminum oxide, $\text{Al}_2\text{O}_3$ .....	3.73
Ammonia, $\text{NH}_3$ .....	1.07
Sulphuric acid, $\text{SO}_3$ .....	10.71
Starch .....	43.25
Water of combination and association by difference..	22.27
	<hr/>
	100.00

Available carbon dioxide 8.10%.

*Mixed Powder:*

Total carbon dioxide, $\text{CO}_2$ .....	10.68
Sodium oxide, $\text{Na}_2\text{O}$ .....	14.04
Calcium oxide, $\text{CaO}$ .....	1.29
Aluminum oxide, $\text{Al}_2\text{O}_3$ .....	4.59
Ammonia, $\text{NH}_3$ .....	1.13
Phosphoric acid, $\text{P}_2\text{O}_5$ .....	3.38
Sulphuric acid, $\text{SO}_3$ .....	11.57
Starch .....	42.93
Water of combination and association by difference..	10.39
	<hr/>
	100.00

Available carbon dioxide 10.37%.

**Baking Powder Controversies.**—Perhaps no class of substances within the domain of food inspection is the subject of so much controversy as baking powder. In the absence of special regulations, baking powder can be regarded as adulterated if it (1) contains a substance injurious to health, (2) if it contains mineral matter, such as clay, ground talc, or calcium sulphate, as a diluent, (3) if it is deficient in available carbon dioxide, or (4) if it does not conform in composition to the label.

**Metallic Impurities.**—Traces of arsenic derived from the raw materials used in manufacture often occur in both alum and phosphate powders while lead from pipes or vats may be present in tartrate and phosphate powders.

**Mineral Diluents.**—Winton, Ogden, and Langley\* found over 25%

\* Conn. Agric. Exp. Sta. Rep., 1900, p. 15.



of ground rock (talc and tremolite) in a sample of baking powder and 20-30% of plaster, calculated as the anhydrous salt, in several aluminous powders. Although experiments by Patten\* indicate that calcium sulphate liberates a certain amount of carbon dioxide from sodium bicarbonate, its presence in considerable amount is generally regarded as an adulterant.

*Deficiency in Available Carbon Dioxide* is a mark of adulteration. Some powders have been found to yield only half the proper amount.

*Misbranding.*—In cases where the general nature of the powder or its constituents is declared on the label, whether or not required by state laws or rulings, any misstatement constitutes a misbranding.

**Cathartics in the Residue.**—The residue left in the bread by all classes of baking powder contains one or more cathartics, but opinions differ as to whether the amount is sufficient even after long continued use to be injurious to health. The nature of these cathartics is shown by the reactions given above and the approximate amounts may be derived from the reactions and the analyses of the baking powder.

**The "Alum Question"** involving the completeness of the decomposition into aluminum hydroxide and the injurious nature of this substance appears to be disposed of for the present by the official report of the Referee Board,† which found that aluminum compounds do not (1) injure the nutritive value of foods, (2) contribute poisonous or other deleterious effect, or (3) reduce the quality or strength of the food. The catharsis produced by very large amounts is due to the sodium sulphate formed and not the aluminum compound, although occasional colic, when very large amounts of aluminum have been ingested, is produced. Giess‡ and co-workers§ obtained somewhat different results from the Referee Board which led them to continue their investigations.

#### METHODS OF ANALYSIS OF BAKING CHEMICALS AND BAKING POWDERS.

**Titration of Sodium Bicarbonate.**—The degree of purity of sodium bicarbonate is best ascertained by titration with standard acid, using methyl orange as indicator, each cubic centimeter of tenth-normal acid being equivalent to 0.0084 gram of sodium bicarbonate.

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\* Jour. Assn. Off. Agric. Chem., 2 II, 1917, p. 214.

† U. S. Dept. of Agric., Bul. 103, 1914.

‡ Biochem. Bul. 5, 1916, p. 151.

§ Steel, Amer. Jour. Physiol., 28, 1911, p. 94; Kahn, Biochem. Bul. 1, 1911, p. 235.



**Titration of Cream of Tartar.**—The degree of purity of commercial cream of tartar is best determined by weighing out exactly 0.188 gram of the sample, dissolving in hot water, and titrating with tenth-normal sodium hydroxide, using phenolphthalein as an indicator. If the article is pure, exactly 10 cc. of the standard alkali will be required for the titration. All the above-named adulterants, with the exception of alum, are either insoluble, or sparingly soluble in hot water, and will indicate the impurity of the sample even before titration. If the adulterant be alum, the sample would go into solution in the water, but the alum would be precipitated by the sodium hydroxide, the precipitate being, however, soluble in an excess of the alkali.

**Determination of Total Carbon Dioxide.**—*Heidenhain Apparatus* \* (Fig. 70).—This is an evolution of the form devised by Mulder and improved by Kolbe, Stobe, and Fresenius.† The apparatus consists of the following parts:

A. A cylinder filled with soda lime to free the air from carbon dioxide. A thick layer of cotton prevents soda lime dust from being carried over.

B. Glass cock to regulate the air current, which finds resistance at C.

C. A capillary contraction.

D. Funnel tube of peculiar shape. The funnel is cylindrical, three-fourths of an inch wide and 4 inches long, and is reduced to half its width at the bottom, so as to make a neck for a perforated rubber stopper.

E. A glass tube tightly fitted into the perforated rubber stopper, allowing the stopper to be taken out, thus admitting the required amount of acid into the flask.

F. Evolution flask, ordinarily of 150 cc. capacity, but for foaming liquids of 300 cc. capacity.

G. Return condenser, consisting of a glass tube of one-fourth of an inch bore, around which a small lead pipe is wound. The tube following the condenser contains a few pieces of calcium chloride to retain the bulk of the moisture. It is refilled when contents are liquefied.

H. U-tube filled with coarse calcium chloride.

K. Tube filled at I with a 3-inch long column of pumice stone impregnated with copper sulphate completely dehydrated at 150° C. The remainder of the tube is filled with fine calcium chloride.

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\* Jour. Am. Chem. Soc., 18, 1896, p. 1.

† Quantitative Analysis, German Edition, 1, 449; 2, 308.



*L.* Cock to close the apparatus when not in use.

*M.* First absorption tube about one-half inch in diameter and 5 inches long, filled mainly with soda lime, with a little calcium chloride on the side at which the air current enters.

*N.* Second absorption tube of same size as *M*, filled half with soda lime and half with calcium chloride, the side containing calcium chloride being toward the end of the apparatus where the air current leaves.

*O.* Guard tube containing calcium chloride toward *N* and soda lime toward *P*.

*P.* Indicator tube trapped with glycerine.

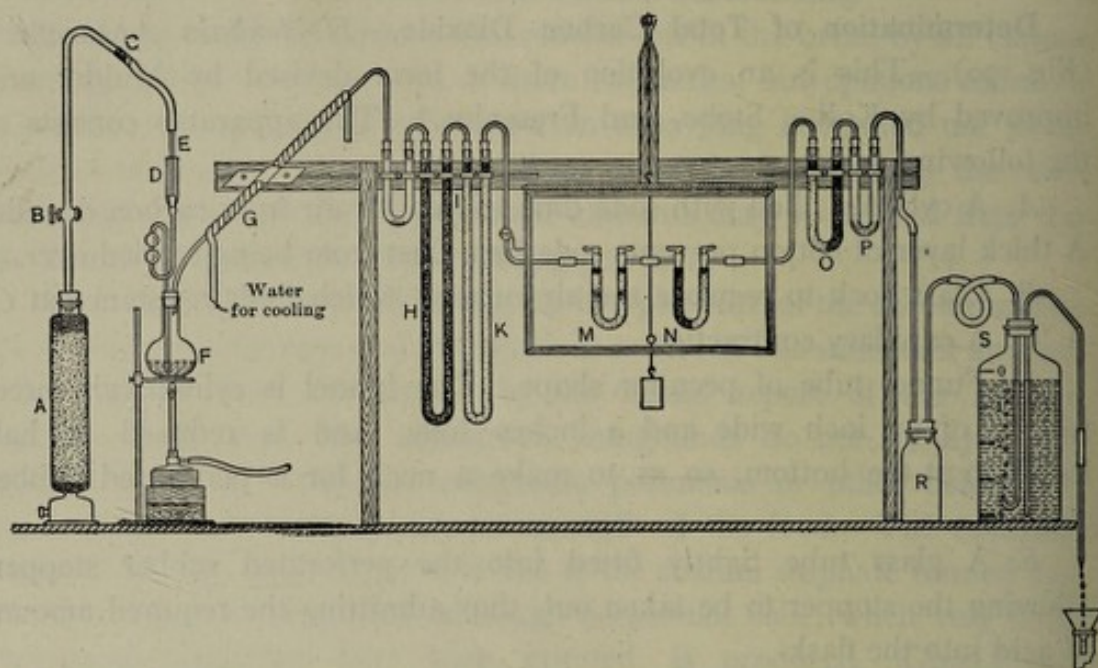


FIG. 70.—Heidenhain's Apparatus for the Determination of Carbon Dioxide. Scale, 1:18.

*R.* Safety bottle to receive water which may be sucked back from the aspirator.

*S.* The aspirator, which is a Mariette's bottle of about 4 liters capacity.

Tubes *M* and *N* should hold about 20 grams, making the capacity of *M* for carbon dioxide nearly 1 gram and of *N* for moisture 0.2 gram. They should be refilled when they have gained 0.75 and 0.1 gram respectively. Use best rubber connections lubricated with a trace of castor oil. Boil connections for the weighed tubes in dilute alkali, wash and dry. Wire or tie all joints. Before using pass carbon dioxide through *H* and *K* for several hours and exhaust.



*Reagents.*—1. *Calcium Chloride.*—This should be dehydrated at 200° C., not fused. Grind so as to pass a No. 18 mesh and reject what passes a No. 30 mesh. The tubes should be filled from the same lot so that the air leaving the apparatus shall have the same moisture content as as that which enters.

2. *Soda Lime.*\*—To a kilogram of commercial sodium hydroxide add 500 to 600 cc. of water and heat in an iron kettle to form a thin paste. While still hot add a kilogram of coarsely powdered quicklime and stir with an iron rod to mix, break up lumps and facilitate the escape of moisture. When cool grind and sift as in the case of calcium chloride, place in wide-mouthed bottles, and seal with paraffin. To give the best results the product should not be too dry.

*Process.*—Weigh tubes *M* and *N* after they have reached the room temperature, opening the cocks for a moment to insure equalization of pressure. Connect tubes with the apparatus and make sure that all joints are tight by closing *A* at the bottom, opening all cocks, starting the aspirator, and observing *P* in which the liquid must soon come to a standstill. Then disconnect the aspirator, close *B*, remove *F* and introduce the material (using about 1 gram of carbonate or 2 grams of baking powder), connect *F* and start the cooler. Introduce hydrochloric acid (sp. gr. 1.1) and carbon dioxide-free water through *D*, lifting *E* slightly. Heat to boiling and lower the flame to keep just at boiling. If no more air passes *P* start the aspirator. When water stops running, open *B* carefully and adjust the outflow of the aspirator by raising or lowering the syphon to half the safe speed.

To find the safe speed charge the apparatus as for an analysis, omitting the carbonate, aspirate at the rate of 50 cc. per minute until 2 liters of air have passed through the system, and weigh the tubes. If together they have lost weight reduce the rate until constant weights are secured. Tube *M* should lose as much as *N* gains.

After *M* has become cool, increase the current to full safe speed and aspirate altogether 3 liters, continuing boiling to the end. After the tubes have reached the room temperature open for a moment and weigh.

**Determination of Residual and Available Carbon Dioxide.**†—Weigh 2 grams of baking powder into a flask suitable for the subsequent determination of carbon dioxide, add 20 cc. of cold water, and allow to stand

\* Benedict and Tower, Jour. Am. Chem. Soc., 21, 1899, p. 396.

† Conn. Agric., Exp. Sta., Rep., 1900, p. 169.



twenty minutes. Place the flask in a metal drying cell surrounded by boiling water and heat, with occasional shaking, for twenty minutes.

To complete the reaction and drive off the last traces of gas from the semi-solid mass, heat quickly to boiling and boil for one minute. Aspirate until the air in the flask is thoroughly changed, and determine the residual carbon dioxide by absorption, as described, under total carbonic acid.

The process described, based on the methods of McGill\* and Catlin,† imitates as far as practicable the conditions encountered in baking, but in such a manner that concordant results may be readily obtained on the same sample and comparable results on different samples.

To obtain the available carbon dioxide subtract the residual from the total carbon dioxide.

**Detection of Tartaric Acid.**‡—In the absence of starch, mix a little of the dry powder in a test-tube with a bit of dry resorcin, add a few drops of concentrated sulphuric acid, and heat slowly. A rose-red color indicates tartaric acid or a tartrate, the color being discharged on dilution with water.

In case of baking powder, or a cream of tartar substitute containing starch, shake repeatedly from 3 to 5 grams of the sample with about 250 cc. of cold water in a large flask, allowing the insoluble portion to subside. Decant the solution through a filter, and evaporate the filtrate to dryness, after which test the dried residue or a portion thereof with resorcin and sulphuric acid as above described.

**Determination of Total Tartaric Acid.**—*Modified Heidenhain Method.*§—Applicable only in the absence of phosphates and salts of aluminum and calcium.

Into a shallow porcelain dish, 6 inches in diameter, weigh out 2 grams of the material and sufficient potassium carbonate to combine with all tartaric acid not in the form of potassium bitartrate. Mix thoroughly with 15 cc. of cold water, and add 5 cc. of 99% acetic acid. Stir for half a minute with a glass rod bent near the end. Add 100 cc. of 95% alcohol, stir violently for five minutes, and allow to settle at least thirty minutes. Filter on a Gooch crucible with a thin layer of paper pulp, and wash

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\* Lab. Int. Rev. Dept., Canada, Bul. 68, p. 31.

† Baking Powders: A Treatise on their Character, Methods for Determination of the Values, etc., p. 20.

‡ Wolff, Rev. chim. anal., 4, 1899, p. 2631.

§ U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 104; Bul. 107, p. 175.



with 95% alcohol until 2 cc. of the filtrate do not change the color of litmus tincture diluted with water. Place the precipitate in a small casserole, dissolve in 50 cc. of hot water, and add standard fifth-normal potassium hydroxide solution, leaving it still strongly acid. Boil for one minute. Finish the titration, using phenolphthalein as indicator, and correct the reading by adding 0.2 cc. One cc. of fifth-normal potassium hydroxide solution is equivalent to 0.026406 gram tartaric anhydride ( $C_4H_4O_6$ ), 0.03001 gram tartaric acid ( $H_2C_4H_4O_6$ ), and 0.03763 gram potassium bitartrate ( $KHC_4H_4O_6$ ).

The standard of the potassium hydroxide solution should be fixed by pure dry potassium bitartrate.

The accuracy of this method is indicated by the agreement of the percentages of potassium bitartrate in cream of tartar powders containing no free tartaric acid, obtained by calculation from the tartaric acid, with those obtained by calculation from the potassium oxide.

In presence of phosphates or of aluminum and calcium salts, the only satisfactory method of arriving at the amount of tartaric acid present is by difference, having determined or calculated the other ingredients.

*Kenrick's Polariscopic Methods.*—*Method 1. (Applicable to Cream of Tartar).*—The method is based on the fact that in the presence of excess of ammonia, the rotation of the solution is proportional to the concentration of the tartaric acid, and is independent of the other bases and acids present.

(a) *The Substance is Completely Soluble in Dilute Ammonia.*—A weighed quantity of the material containing not more than 1 gram tartaric acid is placed in a 25 cc. measuring flask, moistened with 3 or 4 cc. of water, and concentrated ammonia (sp. gr. 0.880) added in quantity sufficient to neutralize all acids that may be present, and leave about 1 cc. in excess. The actual amount of the excess is not of importance, but a greater quantity than 1 cc. of free ammonia should be avoided. The solution is then made up to 25 cc. with water, filtered, if necessary, through a dry filter, and measured in a 20 cm. tube in the polarimeter.

The amount of tartaric acid ( $C_4H_6O_6$ ) in grams ( $y$ ) in the material taken is given by the formula:

$$y = 0.00519x,$$

where  $x$  is the rotation in minutes.

(b) *The Substance is not Completely Soluble in Dilute Ammonia.*—In this case calcium tartrate is probably present, and may be determined as follows: Treat 1 gram of the substance (or an amount containing



not more than 1 gram of tartaric acid) in a small beaker with 15 cc. of water, and 10 drops of concentrated hydrochloric acid. Heat gently till both the potassium and calcium tartrates have passed into solution, and then, while still hot, add 2 cc. of concentrated ammonia (or enough to produce an ammoniacal smelling liquid), and about 0.1 gram of sodium phosphate dissolved in a little water. Transfer to a 25-cc. measuring flask, cool, make up to the mark with water, filter through a dry filter, and polarize the filtrate in a 20-cm. tube. The tartaric acid is calculated from the formula given under (a).

The precipitation of the calcium by means of sodium phosphate is not absolutely necessary, but when this is not done, in cases where the proportion of calcium in the sample is high, there is a great tendency for the calcium tartrate to crystallize out from the ammoniacal solution before the reading is made.

The tartaric acid present as bitartrate of potash may be determined by proceeding as in (a), the calcium tartrate being practically insoluble in cold ammonia solution.

The tartaric acid present as calcium tartrate is given, with sufficient accuracy for most purposes, by the difference between the results of (a) and (b). If more accurate results are required, the residue insoluble in ammonia in (a) may be dissolved in a little hydrochloric acid and treated as above with sodium phosphate and ammonia.

*Method 2. (Applicable to Baking Powder and Cream of Tartar mixed with Substitutes).*—Direct readings of rotation in ammoniacal solution are inadmissible in analyses of the substances of this class, on account of the influence of iron and aluminum on the rotation of tartaric acid, and on account of the small but unknown rotation of the trace of inverted starch.

Accurate determinations, however, may be made in the presence of excess of ammonium molybdate in neutral solution. The latter substance has the property of greatly increasing the rotation of tartaric acid, so that by its use the small rotation of the inverted starch is made insignificant. It is to be noted, however, that this increased rotation is very sensitive to the presence of alkali and acid, and is, moreover, modified by phosphates. It is therefore necessary, in the first place, to remove the phosphoric acid, and, secondly, to bring the solution to a definite state of neutrality. Both these results are attained by the following procedure, the details of which must be carefully adhered to:

(a) *Reagents.*—The following solutions must be prepared, but need not be made up very accurately:



Molybdate solution: 44 grams ammonium heptamolybdate in 250 cc.

Citric acid solution: 50 grams citric acid in 500 cc.

Magnesium sulphate solution: 60 grams  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  in 500 cc.

Ammonia solution: 80 cc. concentrated ammonia (sp. gr. 0.880) in 500 cc.

Hydrochloric acid: 60 cc. concentrated hydrochloric acid in 500 cc.

Methyl orange solution:

(b) *Process*.—An amount of material containing not more than 0.2 gram tataric acid, not more than 0.3 gram alum, and not more than 0.3 gram calcium superphosphate, is accurately weighed, and placed in a dry flask. To this, 5 cc. of citric acid and 10 cc. of molybdate solution are added, and allowed to react with the substance for 10 or 15 minutes (with an occasional shake). Next, 5 cc. of magnesium sulphate solution are added, and 15 cc. of ammonia solution stirred in. After a few minutes (not more than one hour), the solution is filtered through a dry filter, a slight turbidity of the filtrate being disregarded. To 20 cc. of the filtrate are then added a few drops of methyl orange and hydrochloric acid, from a burette, till the pink color appears (2 or 3 drops too much or too little are of no consequence). Finally, 10 cc. more of the molybdate solution are added to the pink solution, which now becomes colorless or pale yellow, and water is added to make up the volume to 50 cc. This solution, after filtering if necessary, is polarized in a 20-cm. tube.

The amount of tartaric acid in grams ( $y$ ) in the weight of substance originally taken is given by the following formula, in which  $x$  is the rotation in minutes:

$$y = 0.001086x + 0.001601\sqrt{x}.$$

But if the rotation is not less than 40', the simpler formula,

$$y = 0.0075 + 0.001168x,$$

may be employed.

The following table gives the tartaric acid in grams for every 10 minutes rotation:

Rotation in Minutes.	Grams Tartaric Acid.	Rotation in Minutes.	Grams Tartaric Acid.
10.....	0.016	90.....	0.1130
20.....	0.029	100.....	0.1246
30.....	0.0415	110.....	0.1365
40.....	0.0535	120.....	0.1479
50.....	0.0657	130.....	0.1595
60.....	0.0776	140.....	0.1710
70.....	0.0895	150.....	0.1825
80.....	0.1013		



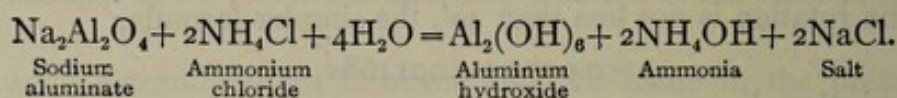
**Determination of Starch.**—*McGill's Method\** (Modified).—Digest 1 gram of the sample with 150 cc. of a cold 3% solution of hydrochloric acid during twenty-four hours, with occasional shaking. Filter through a tared Gooch crucible, wash first with water until neutral, then once with alcohol, and finally with ether. Dry at 110° C. for four hours, cool, and weigh. Burn off the starch, and again weigh. The difference in the two weights indicates the weight of the starch. The purity of the starch is insured by examination with the microscope.

*Acid Conversion Method.*†—If the sample contains lime, mix 5 grams in a 500-cc. flask with 200 cc. of 3% hydrochloric acid, and let the mixture stand an hour with frequent shaking. Filter through a wetted 11-cm. filter, wash with water, and transfer the starch by a wash-bottle from the filter-paper back into the original flask, using 200 cc. of water.

If the sample be free from lime, weigh 5 grams directly into the 500-cc. flask with 200 cc. of water. In either case add 20 cc. of hydrochloric acid (specific gravity 1.125) and heat the flask in boiling water for 2½ hours, the flask being provided with a reflux condenser. Determine the dextrose, and from this the starch in the regular manner.

**Detection of Aluminum Salts.**‡—(a) *In Baking Powder.*—Applicable in presence of phosphates. Burn to an ash about 2 grams of the sample in a platinum dish. Extract with boiling water and filter. Add to the filtrate sufficient ammonium chloride solution to produce a distinct odor of ammonia. A flocculent precipitate indicates aluminum.

In igniting, as above directed, sodium aluminate results from the more or less complete fusion. The reaction which occurs may be represented as follows:



If any phosphate of lime be present, it will be insoluble in the solution of the ash. If phosphate of sodium or potassium be present, it will go into solution, but will only precipitate out when an aluminum salt is also present on the addition of the ammonium chloride reagent.

(b) *In Cream of Tartar.*—Mix about 1 gram of the sample with an equal quantity of sodium carbonate, burn to an ash, and proceed as in the case of baking powder (a).

\* Canada Inland Rev. Bul. 68, p. 33.

† U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 105; Bul. 107 rev., p. 176.

‡ Leach, 31st An. Rep. Mass. State Board of Health, 1899, p. 638.



**Determination of Alumina.**—The above qualitative method with ammonium chloride may be made quantitative in presence of phosphates as follows: After carrying out the qualitative method as above directed, filter off the final precipitate, dissolve it in nitric acid, and test it for phosphate with ammonium molybdate. If phosphates are found absent, proceed as before with a weighed amount of the sample and wash, ignite, and weigh the residue as  $\text{Al}_2\text{O}_3$ .

If phosphate is found present in the ammonium chloride precipitate, proceed as before, igniting and weighing the total residue. Then determine the  $\text{P}_2\text{O}_5$  in the latter and subtract from the total. The difference will be the  $\text{Al}_2\text{O}_3$ .

**Determination of Lime.**—5 grams of the sample are treated in a 500-cc. graduated flask with 50 cc. of water and 25 cc. of concentrated hydrochloric acid. Add water to the mark, shake, and allow the starch to settle. Decant through a dry filter, and to 50 cc. of the filtrate add ammonia nearly to neutralization, an excess of ammonium acetate solution, and 4 cc. of 80% acetic acid, and heat at  $50^\circ \text{C}$ . Filter if necessary, and precipitate the lime with an excess of ammonium oxalate. Filter, wash, and ignite over a blast-lamp. Weigh as  $\text{CaO}$ .

**Determination of Potash and Soda.\***—Weigh out 5 grams into a platinum dish, and incinerate in a muffle at a low heat. The charred mass is well rubbed up in a mortar, then boiled fifteen minutes with about 200 cc. of water, to which has been added a little hydrochloric acid. The whole is transferred to a 500-cc. flask, and, after cooling, made up to the mark and filtered. Of the filtered liquid 100 cc., representing 1 gram of the sample, are measured out, heated to boiling, and a slight excess of barium chloride solution added; then without filtering barium hydroxide is added in slight excess, the precipitate filtered off, and washed. To the filtrate is added a little ammonium hydroxide, and ammonium carbonate solution until the barium is precipitated. This precipitate is filtered and washed, the filtrate evaporated to dryness, and carefully ignited below redness until all volatile matter is driven off. The residue is dissolved in a few cc. of water, and a few drops of ammonium carbonate solution added. The precipitate, if any, is removed by filtering and washing, and the filtrate evaporated in a small tared platinum dish, ignited below redness, and weighed.

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\* Conn. Agric. Exp. Sta. Rep., 1900, p. 178.



This gives the weight of the mixed chlorides. The residue is taken up with hot water, from 5 to 10 cc. of a 10% solution of platinic chloride added, and the whole evaporated to a sirupy consistency on the water-bath; it is then treated with 80% alcohol, the precipitate washed with 80% alcohol by decantation, transferred to a Gooch crucible, dried at 100° C., and weighed. The weight of the precipitate, multiplied by 0.19308, gives the weight of  $K_2O$ , and by 0.3056 the equivalent amount of  $KCl$ . The weight of  $KCl$  found is subtracted from the weight of the mixed chloride, the remainder being  $NaCl$ , which, multiplied by 0.5300, gives the weight of  $Na_2O$  in the sample.

**Determination of Phosphoric Acid.**—Mix 5 grams of the material with 10 cc. of magnesium nitrate solution, prepared by dissolving calcined magnesia in nitric acid, adding magnesia in excess, and filtering, dry, ignite, and dissolve in hydrochloric acid. Remove an aliquot part of the solution, corresponding to 0.25 gram, 0.50 gram, or 1 gram, neutralize with ammonia, clear with a few drops of nitric acid, and proceed according to the usual method, precipitating successively with molybdic solution and magnesia mixture.

**Determination of Sulphuric Acid.**—Boil 5 grams of the powder gently for 1½ hours with a mixture of 300 cc. of water and 15 cc. of concentrated hydrochloric acid. Dilute to 500 cc., draw off an aliquot portion of 100 cc., dilute considerably, precipitate with barium chloride, filter through a Gooch crucible, ignite, and weigh. Direct solution of the material without burning the organic matter was proposed by Crampton.\*

**Determination of Ammonia** (present in the form of ammonia alum or ammonium carbonate). Mix 5 grams of the sample with 200 cc. of water, and add an excess of sodium hydroxide. Distil into standard acid, and determine the ammonia by titration.

**Detection and Determination of Arsenic.**—Proceed according to the Marsh or Sanger-Black-Gutzeit method without preliminary treatment (page 64).

**Determination of Lead.**—*Method of the Victor Chemical Works.*—In the case of phosphate and alum phosphate powders and of acid phosphate weigh 1 to 2 grams of the material into a small beaker, add 10 to 15 cc. of water and 2 to 3 cc. of concentrated sulphuric acid. Bring to a boil and if starch is present continue the heating on a water-bath until the starch is hydrolized, replacing the water lost by evaporation. Cool,

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\* U. S. Dept. of Agric., Div. of Chem., Bul. 13, part 5, p. 596.



add 30 to 40 cc. of 95% alcohol, stir and allow to stand over night. If a precipitate of sodium aluminum sulphate appears, due to an excess of alcohol, add water in small amounts until dissolved. Filter and wash with 75 to 80 cc. of alcohol until free from acid. Dry, transfer the bulk of the precipitate to a crucible, digest with hot alkaline ammonium acetate solution (390 grams of ammonium acetate in 1800 cc. of water and 150 cc. of concentrated ammonium hydroxide), filter through the paper previously used, and wash with small portions of the hot solution. After cooling, make up to 50 cc., add 1 cc. each of 10% potassium cyanide solution, 1% gelatine solution, and colorless ammonium sulphide solution. Compare with standard lead solution prepared from a stock solution of 0.160 gram of lead nitrate (dried over sulphuric acid) in 1000 cc. of water (1 cc. = 0.0001 gram Pb) and the quantities of alkaline acetate, cyanide, gelatine and sulphide already given.

In the case of tartrate powders and cream of tartar shake 10 grams of the material with 50 cc. of water and 40 cc. of 2N ammonia water. Make up to 100 cc., mix and filter through a dry filter. Determine lead colorimetrically in 50 cc. of the filtrate as above, omitting the preliminary precipitation as sulphate. The standard lead solution should be made up using about the same amount of lead-free cream of tartar as in the solution of the material.

#### SEMOLINA AND EDIBLE PASTES.

*Semolina* is the coarse meal ground from certain varieties of hard or "durum" wheats, grown originally in Italy, Sicily, and Russia, but at present in France and certain parts of the United States and Canada. This hard wheat is high in gluten, and especially adapted for the preparation of macaroni and the various pastes. A peculiar process is employed in preparing the wheat, whereby the husk is removed by wetting, heating, grinding, and sifting, the resulting meal or *semolina*, being in the form of small, round, glazed granules.

**Italian Pastes.**—*Semolina* furnishes the basis of the Italian edible pastes, being mixed with warm water, kneaded, and molded into various forms, either by pressure through holes in an iron plate, or otherwise, and finally dried. In parts of Italy juices of carrots, onions, and other vegetables are said to be mingled with the paste, but for local consumption



only. Saffron is sometimes added to pastes for the purpose, so it is claimed, of imparting a spicy flavor, although the quantity used is often so small as to be apparent only to the eye, thus indicating that the real object of its addition is to impart a color in imitation of an egg paste.

*Macaroni* is the larger of the slender-tube or pipe-shaped products; *vermicelli* is the worm-shaped variety, produced when the holes in the plate are very small; *spaghetti* is the term applied to the cord-like paste intermediate in size between the others. A variety of Italian pastes or *pâtés* is made by rolling the kneaded semolina into thin sheets, and cutting out in shapes of animals, letters of the alphabet, etc.

The composition of some of these products is as follows:

	No. of Samples.	Water.	Protein.	Fat.	Total Carbohy- drates.	Crude Fiber.	Ash.	Fuel Value per Pound. Cal's.
Semolina *	.....	10.50	11.96	0.60	75.79	0.50	0.65	
Macaroni †	11	10.3	13.4	0.9	74.1	....	1.3	1665
Noodles †	2	10.7	11.7	1.0	75.6	0.4	1.0	1665
Spaghetti †	3	10.6	12.1	0.4	76.3	0.4	0.6	1660
Vermicelli †	15	11.0	10.9	2.0	72.0	....	4.1	1625

\*Balland.

† Atwater and Bryant.

**Noodles** are a strap-shaped form of paste made in German households as well as in factories. True, or egg-noodles, contain a certain percentage of eggs, while water-noodles are practically the same in composition as Italian pastes. The difference in composition between water-noodles and noodles made with different numbers of eggs or egg yolks per German pound of flour, is shown by the analyses of Juckenack and Pasternack\* given in the following table: †

Number of Eggs per Pound of Flour.	Composition of the Dry Matter.					Number of Egg Yolks per Pound of Flour.	Composition of the Dry Matter.				
	Ash.	Total Phos- phoric Acid.	Lecithin Phos- phoric Acid.	Ether Extract	Protein N × 6½		Ash.	Total Phos- phoric Acid.	Lecithin Phos- phoric Acid.	Ether Extract	Protein N × 6½
	%	%	%	%	%		%	%	%	%	%
0	0.460	0.2300	0.0225	0.66	12.00	0	0.460	0.2300	0.0225	0.66	12.03
1	0.565	0.2716	0.0513	1.56	12.99	1	0.488	0.2720	0.0518	1.57	12.37
2	0.664	0.3110	0.0786	2.42	13.92	2	0.516	0.3127	0.0801	2.47	12.73
3	0.758	0.3482	0.1044	3.24	14.81	3	0.542	0.3520	0.1075	3.33	13.07
*	*	*	*	*	*	*	*	*	*	*	*
12	1.426	0.6123	0.2875	7.94	21.09	12	0.745	0.6533	0.3171	8.64	15.71

\* Zeits. Unters. Nahr. Genussm., 3, 1900, p. 13; 8, 1904, p. 94.

† The German pound is 500 grams; the avoirdupois pound is 454 grams.



From these results it appears that the percentages of ash, total phosphoric acid, and protein are appreciably increased by the addition of each egg or egg yolk, while the percentages of lecithin-phosphoric acid and ether extract are more than doubled by the addition of the first egg, and are increased in corresponding proportion by the addition of two or more eggs.

The German Association of Food Chemists require that commercial egg-noodles contain at least 0.045% of lecithin-phosphoric acid, and 2.00% of ether extract, corresponding to the minimum in noodles with two eggs per half kilogram of flour.

Spaeth\* considers that if the ether extract of noodles has an iodine number over 98, it is safe to assume that they contain no eggs or only traces.

Farcy† determines the nitrogen soluble in hot and cold water. In flour or noodles made from flour alone he found about 0.3%, irrespective of whether hot or cold extraction was followed, while with pastes containing 3-5 eggs, the amount soluble in cold water was the same as for water-noodles, but that soluble in hot water was 0.45-0.48%.

In interpreting the results of analysis it should be remembered that fat may have been introduced in some form other than in eggs, and that the lecithin-phosphoric acid diminishes somewhat on long standing, although results obtained by Nochmann‡ indicate that the loss is slight when care is taken to avoid exposure to warm moist air. Allowance should also be made for the variation in composition of the eggs and flour.

Of 22 brands of American noodles examined by Winton and Bailey,§ only 5 appeared to be made with eggs; the lecithin-phosphoric acid in these ranged from 0.036 to 0.058, and the ether extract from 1.83 to 2.33 per cent, while in the other samples the lecithin-phosphoric ranged from 0.015 to 0.032 and the ether extract from 0.28 to 2.50%.

**Adulteration of Pastes.**—Rice, corn, and potato flours have been used in the preparation of the cheaper varieties of semolina, but rarely in this country. A more common form of adulteration is the substitution of water-noodles for egg-noodles, artificial colors being used to carry out the deception. Substitutions of this kind are detected by determina-

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\* Forschber. Lebensm., 3, 1896, p. 47.

† Ann. fals., 7, p. 183.

‡ Zeits. Unters. Nahr. Genussm., 25, 1913, p. 717.

§ Conn. Agric. Exp. Sta. Rep., 1904, p. 138; Jour. Amer. Chem. Soc., 37, 1905, p. 137.



tions of lecithin-phosphoric acid and ether extract, supplemented by tests for artificial colors.

#### ANALYSIS OF PASTES.

##### **Determination of Lecithin-phosphoric Acid.**—*Juckenack's Method.\**

—Extract 30 grams of the finely ground material for 10 hours with absolute alcohol in a Soxhlet extractor at a temperature inside the extractor not below 55°–60° C. The extraction flask should be provided with a small quantity of pumice stone to prevent bumping during the boiling, and the extractor enclosed by asbestos paper, if the desired temperature is not readily maintained. After the extraction is completed, add 5 cc. of alcoholic solution of potash (prepared by dissolving 40 grams of phosphorus-free caustic potash in 1000 cc. alcohol), and distil off all the alcohol. Transfer the residue to a platinum dish by means of hot water, evaporate to dryness on a water bath, and char over asbestos. Treat the charred mass with dilute nitric acid, filter, and wash with water. Return the residue with the paper to the platinum dish, and burn to a white ash. Treat again with nitric acid, filter and wash, uniting the filtrates. Determine phosphoric acid by the usual method.

**Determination of Nitrogen Soluble in Hot and Cold Water.**—*Farcy Method.†*—Heat 10 grams of the powdered sample and 150 cc. of water in a 200-cc. graduated flask in a boiling water-bath for 2 hours with occasional shaking, cool, make up to the mark, centrifuge, filter, and determine nitrogen in 50 cc. of the clear filtrate. Repeat the operation, digesting at room temperature.

**Precipitin Test for Eggs.**—Arragon and Bornand‡ proposed the antiserum method of detecting eggs or parts of eggs and Gothe§ has developed details for conducting the test. While the test is doubtless valuable in the hands of a trained serologist, the chemical methods seem more practicable for ordinary use.

**Detection of Artificial Colors in Pastes.**—The following colors have been used in noodles and other pastes: turmeric, saffron, annatto, naphthol yellow (Martius yellow), naphthol yellow S, picric acid, aurantia, Victoria yellow, tartrazine, metanil yellow, azo yellow, gold yellow, and quinoline yellow. Of these naphthol yellow, picric acid, metanil yellow,

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\* Zeits. Unters. Nahr. Genussm., 3, 1900, p. 13.

† Loc. cit.

‡ Chem. Ztg., 37, 1913, p. 1345.

§ Zeits. Unters. Nahr. Genussm., 30, 1915, p. 389.



and Victoria yellow are injurious to health, and their use is illegal in European countries as well as in the United States. Fortunately, they are rarely found in the products now on the market.

The detection of artificial colors is complicated by the presence of the natural coloring matter of the flour and the lutein of eggs. These are conveniently extracted by ether, which does not remove the artificial colors, although most of them unmixed dissolve freely in this solvent.

*Juckenack's Method.\**—Thoroughly shake two portions of the finely ground material, each of about 10 grams, in test tubes with 15 cc. of ether and 15 cc. of 70% alcohol respectively, and allow to stand 12 hours.

(a) If the ether remains uncolored or only slightly tinted and the material below it remains yellow, while the alcohol is distinctly colored and the material is decolorized, a foreign dye is indicated.

(b) If both ether and alcohol are colored, either (1) lutein (egg color) alone, or (2) this with a foreign dye is present.

1. Treat a portion of the ether solution with dilute nitrous acid, according to Weyl. If the ether is not completely decolorized, a foreign dye is present.

2. If the deposit of material below the alcohol is decolorized, while that below the ether is colored, tests should be made for foreign dyes as follows: Shake the portion previously treated with ether with three or more fresh portions of the same solvent, until no more color is extracted, and then shake the residue with 70% alcohol and allow to stand 12 hours. After filtering, concentrate the solution slightly, acidify with hydrochloric acid, boil with sensitized wool, and identify the color in the usual manner (Chapter XVII).

*Schlegel's Method.†*—Extract 100 grams of the finely powdered material with ether in a continuous extraction apparatus, and shake the residue at frequent intervals for half a day with a mixture of 140 cc. of alcohol, 5 cc. of ammonia, and 105 cc. water. Filter, evaporate to remove alcohol and ammonia, acidify slightly with hydrochloric acid, and again filter. Boil the filtrate with sensitized wool, and identify the color on the dyed fiber by the usual tests (Chapter XVII).

*Fresenius Method.‡*—Extract 20 to 40 grams of the powdered material with ether in a continuous extraction apparatus. Dry the residue to remove ether, shake for 15 minutes with 120 cc. of 60% acetone, and

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\* Zeits. Unters. Nahr. Genussm., 3, 1900, p. 1.

† Untersuchungsanstalt, Nürnberg, Ber., 1906, p. 24

‡ Zeits. Unters. Nahr. Genussm., 13, 1907, p. 132.



allow to stand 12 to 24 hours. Filter, evaporate, until the acetone is removed, and divide into two portions, a larger and a smaller. To the larger portion add sufficient acetic acid to dissolve flocks, and boil with sensitized wool. Remove natural coloring matter from the wool by boiling in dilute acetic acid. If after this treatment the wool is dyed, the presence of a foreign color is indicated, which may be identified by the usual tests.

To the smaller portion of the aqueous solution, obtained after removal of the acetone as above described, add an equal volume of alcohol, heat to dissolve flocks, divide into four portions, and apply special tests to three of these, reserving the fourth for comparison. The natural color of the flour is decolorized by hydrochloric acid, intensified by ammonia, but not affected by stannous chloride, even on heating. Saffron reacts in a similar manner, but is only slightly bleached by the acid, and is not affected by the other two reagents.

*Piutti and Bentivoglio Method.\**—This method is especially designed to detect the four colors forbidden by Italian law, and to distinguish these from naphthol yellow S.

Add 50 grams of the paste to 500 cc. of boiling water, made alkaline with 2 cc. of concentrated ammonia water, add 60 to 70 cc. of alcohol, and continue the boiling 40 minutes. After filtering, acidify the liquid with 2 to 3 cc. of dilute hydrochloric acid and boil with 5 to 6 strands of sensitized wool, each strand weighing about 0.5 gram. Wash the wool, dissolve the color in dilute ammonia, and repeat the dyeing. After dissolving a second time in ammonia, evaporate the solution of the dye to dryness, avoiding as far as possible the formation of a skin, and take up the residue in water. If a skin has formed, filter and test the insoluble matter for metanil yellow with dilute hydrochloric acid, and for picric acid with ammonium sulphide.

To 1 cc. of the filtrate add stannous chloride solution and a little sodium hydroxide, or preferably sodium ethylate. If no red color forms, nitro-colors are absent; if, also, in another portion dilute hydrochloric acid produces no violet color, thus showing the absence of metanil yellow, no further test is necessary. In the presence of these colors, acidify the remainder of the solution with acetic acid, shake violently with carbon tetrachloride, and identify the color according to the following scheme:

A. Color dissolves in carbon tetrachloride to colorless solution. Extract with very dilute ammonia, concentrate and divide into two parts.

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\* *Gaz. chim. ital.* 36, II, 1906, p. 385.



1. Acidify with hydrochloric acid, and add 1 to 2 drops of stannous chloride and ammonia in excess. A rose colored solution and precipitate form.....*Naphthol yellow*.

2. Acidify slightly with hydrochloric acid, add a little zinc dust and stir. Solution becomes rose-violet. ....*Victoria yellow*.

*B.* Color is insoluble in carbon tetrachloride. Evaporate to dryness on water-bath, take up in water and divide into three parts.

1. Hydrochloric acid produces a violet coloration.....*Metanil yellow*.

2. Ammonium sulphide produces a red brown coloration.

*Picric acid.*

3. Stir on a water-bath with zinc dust and ammonia, filter, treat with zinc dust and hydrochloric acid and again filter. (a) Potassium hydroxide produces a yellow coloration, and (b) ferric chloride an orange coloration.

*Naphthol yellow S.*

*Schmitz-Dumont Test for Tropeolins.\**—Moisten a small portion of the material with a few drops of dilute hydrochloric acid. The formation of a reddish or bluish color shows the presence of an azo color or some other coal-tar color.

*Martini Test for Saffron.†*—Treat 50 grams of the finely powdered material in the cold for 24 hours with 100 cc. of 70% alcohol, shaking occasionally, or reflux for 15 minutes. Filter the extract, concentrate to a paste on the water-bath, and extract the residue with ether, thus removing interfering colors. Heat the residue on the water-bath until all ether is removed, then add 98% alcohol and continue the heating thus gradually dissolving the saffron color. Filter the alcoholic solution, evaporate on the water-bath, and test the residue which in the presence of saffron takes on a blue color changing to violet, green, and brown with concentrated sulphuric acid and a transient green color with concentrated nitric acids.

*Test for Turmeric.*—Extract the color from the ground material by alcohol and identify by the boric acid test (Chapter XVII).

#### CEREAL BREAKFAST FOODS.

The large number and variety of these preparations now on the market testify to the fact that breakfast cereals form a most important, as well as considerable, portion of our food supply. These foods are generally prepared from wheat, oats, corn, and rice, and are, as a rule, remarkably

\* Zeits. öff. Chem., 8, 1902, p. 424.

† Bol. chim. farm., 52, p. 37.



pure and free from adulteration, though the food value of different varieties is often grossly misstated by their manufacturers. Formerly the breakfast food consisted entirely of the coarsely ground, generally decorticated, raw cereal grain, and required a long period of cooking to prepare it for use. At present many of the oat products, and to some extent also those of corn, rice, and wheat, are subjected to a more or less preliminary cooking and drying, whereby they are capable of being prepared for use in a much shorter time, and their keeping qualities are enhanced. The so-called rolled oats are prepared by softening the grains through steaming, after which they are crushed between rollers and afterwards dried. The steaming process is a typical one for various other cereals, though in some cases the heating consists in baking or kiln drying.

The effect of the preliminary cooking on the finished product varies somewhat according to whether dry or moist heat has been applied, and is chiefly noticeable in the altered character of the carbohydrates. In all cases the starch is rendered more soluble, whether by the conversion of a portion into dextrin and dextrose, or by a simple breaking down of the starch grains, as in the case of bread in baking.

In spite of the seemingly endless variety of the package cereals, they divide themselves as a matter of fact into a very few well-defined classes, the members of which differ but little from each other except in name.

First there are the raw cereal grains of the oat, wheat, and corn, prepared by simple crushing to various degrees of fineness, after decorticating; next comes the classes of partially cooked preparations of each of these grains, appearing in various forms of "flakes," "granules," "puffs," etc., and again a class known as malted cereals, in which the moist, ground grain is mixed with malted barley, and, by controlling the temperature, a portion of the starch is converted to maltose and dextrin, after which the mixture is crushed between hot rollers and dried.

In the preparation of most of the corn breakfast products, such as samp and hominy, it is customary to remove the germ, which contains the oil and fat, lest the tendency of the latter to become rancid should result in the deterioration of the food. In wheat foods the germ is less often removed, and rarely, if ever, in oat preparations. The amount of fat found in the prepared cereal food as compared with that in the whole grain is of interest in this connection.

**Composition of Some of the Common Breakfast Cereals.**—The analyses below by Baird \* will serve to typify the various classes of these prepara-

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\* N. Dak. Agric. Exp. Sta., Spec. Bul. Food Dept., 3, 1915, p. 395.



tions as they appear on the market. Where more than one sample was analyzed the samples were of different brands of the same or different manufacturers:

#### AVERAGE COMPOSITION OF BREAKFAST FOODS (BAIRD).

	No. of Anal-yses.	Water.	Protein N $\times$ 6.25	Fiber.	Nitrogen free Extract.	Fat.	Ash.	Fuel Value per Lb. Cal.
Wheat Products:								
Farina.....	3	8.10	11.98	0.22	78.01	1.11	0.58	1719
Rolled Wheat.....	1	8.97	13.26	2.26	72.14	1.78	1.59	1562
Shredded Wheat.....	1	6.30	10.33	1.91	78.44	1.40	1.62	1709
Flaked Wheat.....	1	6.85	11.56	1.45	76.32	1.17	2.65	1685
Puffed Wheat.....	1	6.99	13.50	2.12	73.93	2.06	1.40	1712
Oat Products:								
Rolled Oats.....	6	6.55	15.36	1.38	68.34	6.49	1.88	1832
Corn (Maize) Products:								
Hominy.....	1	9.48	8.19	0.41	80.97	0.56	0.39	1683
Corn Flakes.....	6	5.42	7.72	0.38	83.47	0.25	2.76	1692
Corn Puffs.....	1	6.33	9.13	0.96	82.77	0.31	0.50	1723
Miscellaneous:								
Force.....	1	6.17	7.81	0.91	80.61	1.43	3.07	1705
Grape Nuts.....	1	2.59	12.50	1.63	80.66	0.82	1.80	1769

The methods of analysis employed for these preparations are the same as for ordinary cereals (p. 285), the sample being ground fine enough to pass through a 1-mm. sieve.

#### PREPARED FOOD FOR INFANTS AND INVALIDS.

In dealing with the composition and analysis of this class of proprietary foods more than ordinary care is necessary, in view of the fact that one or another of these preparations is frequently prescribed for the exclusive diet of those whose very life may depend on the character and suitability of the food to the case in hand. Many of these foods do, as a matter of fact, honestly fulfil the claims of their manufacturers, but others fall far short of so doing, so that it is hardly safe to use them unless some intelligent idea of their composition can be gained. It is not, as a rule, within the province of the analyst to furnish an opinion regarding the adaptability of a certain food to the requirements of an infant or invalid, but rather to provide the necessary data whereon such an opinion may be intelligently based.



A simple statement of moisture, fat, protein, carbohydrates (by difference), and ash, which in the case of ordinary foods would often be sufficient, would be obviously inadequate in expressing the analysis of an infant food, since it is of much more vital importance than in other foods to know the solubility of the food itself, and, to as great an extent as possible, the character of the carbohydrates.

The chief ingredients of many of these preparations are wheat or mixed cereals high in starch. Many of the foods are, according to the directions, to be used practically without cooking, but by simply mixing with milk or water, and, in some cases, bringing to the boiling-point. Hence the degree of conversion which the raw starch has undergone in the process of manufacture of the food should, if possible, be ascertained as a prime factor in judging of its character and adaptability to the needs of the young child and of the sick. Incidentally it should be said that few if any of the infant foods, even those whose high character has long been established by continued trial, conform very closely to the composition of woman's milk, which was long accepted as the true standard on which to base their efficiency. Hence it is no easy task to pass judgment on a particular food from its chemical composition alone without trial, nor is it right to unqualifiedly condemn in all cases food high in insoluble carbohydrates, since there are undoubtedly many instances in which such foods are successfully used.

**Preparation.**—The soluble farinaceous foods are usually prepared somewhat as follows: A mixture of ground wheat and barley malt (with sometimes a little wheat bran) is mixed with water to form a paste, and a little bicarbonate of potash added. The mixture is heated at 65° C. for sufficient time to convert the starch, after which it is exhausted with warm water, the extract being strained, and the filtrate evaporated to dryness to form the food. The sugars of such foods consist largely of maltose mixed with dextrin.

The farinaceous foods, which depend for the conversion of their starch on the method of cooking or heating before serving, are usually mixtures of wheat or other cereal flour with malt or pancreatic extract.

The milk foods are variously prepared, either by the simple desiccation of cow's milk (usually previously skimmed), or, when whole milk is used, by mingling the desiccated milk with sugars or baked cereal flour. Sometimes desiccated milk is used in mixture with a dried extract of malted cereals. In fact all sorts of mixtures are found on the market,



involving, however, in nearly all cases, one modification or another of the above general processes of preparation.

**Composition.**—Few complete analyses of these classes of foods have recently been made. Among the best are those of McGill,\* from whose work the following figures have been selected, illustrating typical examples of foods on the market.

	Num- ber of Anal- yses.	Water.	Pro- tein N X 6.25.	Total Car- bohy- drates.	Cold Water Extract.	Starch etc. by Differ- ence.	Fat.	Ash.	Kind of Starch.
Robinson's Patent Barley....	7	9.8	7.0	81.6	4.9*	76.9*	0.9	0.7	Barley
Ridge's Patent Food.....	2	9.6	13.2	76.0	3.9†	74.7†	0.8	0.4	Wheat
Mellin's Infant Food.....	1	3.0	10.8	82.8	87.4	...	0.1	3.3	None
Cristie's Food.....	4	5.8	6.8	83.2	35.4*	46.9*	3.2	1.0	Wheat
Benger's Food.....	1	8.0	11.7	79.1	11.9	67.3	0.3	0.9	Wheat
Allenbury's Malted Food....	2	5.3	10.1	83.1	18.3†	65.7†	0.7	0.8	Wheat
Horlick's Malted Milk.....	6	3.1	14.8	70.9	75.3*	...	8.0	3.2	None
Nestlé's Food.....	10	1.9	11.5	80.2	64.8*	15.5*	5.3	1.1	Wheat
Allenbury's Milk Food No. 1	1	2.7	10.1	65.9	66.5	...	17.8	3.5	None
Allenbury's Milk Food No. 2	4	3.7	9.5	68.4	68.5*	0.8†	15.6	2.8	None
Lactated Food.....	4	7.2	7.8	83.6	31.0*	53.6*	0.2	1.2	Wheat
Wampole's Milk Food.....	1	5.2	12.7	72.0	76.6	...	7.9	2.2	None
Eskay's Albuminized Food...	2	2.6	6.6	87.9	52.8†	35.2†	1.4	1.5	Arrowroot

\* Two analyses.

† One Analysis.

Street † in 1907-1908 analyzed single samples found on sale in Connecticut, representing most of the products since analyzed by McGill, but those included in the following table were not apparently found on sale in Canada in 1914. The figures for water extract and starch (direct determination) are not strictly comparable with those for cold water extract, starch, etc. (by difference) as obtained by McGill.

	Water.	Pro- tein N X 6.25.	Fiber.	Nitro- gen- free Ex- tract.	Reduc- ing Sugar as Dex- trose.	Starch	Water Ex- tract.	Fat.	Ash.	Kind of Starch.
Imperial Granum.....	5.9	13.1	0.1	80.1	....	74.0	3.3	0.4	0.5	Wheat
Borden's Malted Milk.....	5.4	13.4	0.2	71.7	37.1	0.6	88.4	6.1	3.2	None
Carrick's Soluble Food....	3.1	12.3	0.5	81.0	18.8	16.2	48.9	1.1	2.0	
Carrick's Lacto-preparata	2.5	11.4	....	80.9	51.4	2.8	79.7	2.3	2.9	

**Diabetic Foods.**—Gluten flour and similar preparations are primarily intended for the use of diabetics, from whose dietary carbohydrates must be excluded.

\* Canadian Dept. of Inland Rev., Bul. 278, 1914.

† Conn. Agric. Exp. Sta. Rep., 1907-8, p. 599.



The following analyses of commercial gluten preparations were made by Woods and Merrill.\*

	Protein.	Fat.	Carbohy- drates.	Ash.
"Cooked gluten".....	16.88	3.86	76.80	2.46
Whole-wheat gluten.....	17.89	5.20	73.85	3.06
"Glutine".....	15.31	0.99	82.52	1.17
Breakfast cereal gluten.....	43.70	1.60	44.40	0.70
Plain gluten flour.....	53.60	1.20	34.50	0.60
Self-raising flour.....	31.50	1.40	53.20	3.80

Many brands of gluten flour are put on the market by dealers in so-called "health foods," and in many cases are represented to be practically free from starch. Thirteen samples of gluten flour were analyzed by the author in 1899, varying in price from 11 to 50 cents per pound. Of these, 3, the product of one manufacturer, contained less than 1% of starch, 3 contained from 10 to 20%, while 7 contained from 56 to 70% of starch, the substance which, of all others, the diabetic patient tries to avoid. Some of these preparations were little more than whole-wheat flour. An analysis of one of them, known as "Pure Vegetable Gluten," and sold for 50 cents per pound, and of two similar diabetic flours reported by Winton, follows:

	"Pure Vegetable Gluten."	"Diabetic Food."	"Diabetic Flour."
Moisture.....	10.78	12.67	9.26
Ash.....	2.20	0.43	1.30
Fat.....	3.25	0.90	2.21
Protein.....	14.25	11.37	14.25
Crude Fiber.....	.....	0.25	1.03
Sugars.....	1.70	71.51	66.63
Dextrin.....	2.55		
Starch.....	56.55		
Undetermined.....	8.72	2.87	5.32
	100.00	100.00	100.00

Analyses of other preparations have been reported by Street† and McGill.‡

\* Maine Exp. Sta. Buls. 55 and 75.

† Conn. Agric. Exp. Sta. Rep., 1912, p. 107.

‡ Lab. Int. Rev. Dept. Canada, Bul. 354, 1916.



Winton has reported the following analyses of flours and meals well suited for the preparation of diabetic biscuit, and of the biscuit made from two of these by a cook in the family of a diabetic patient:

		Moisture.	Ash.	Protein, (N $\times$ 6.25).	Crude Fiber.	Nitrogen-free Extract.	Fat.	Starch, Sugar, and Dextrin.
Gluten flour . . . .	{ In original . . . . .	10.12	0.22	85.38	0.03	3.69	0.56	4.46
	{ Calc. water-free . . . . .		0.24	95.00	0.03	4.11	0.62	4.96
Gluten biscuit . . .	{ In original . . . . .	25.58	2.35	50.91	0.64	3.18	17.34	.....
	{ Calc. water-free . . . . .		3.16	68.41	0.86	4.27	23.30	.....
Soja bean meal . .	{ In original . . . . .	7.75	4.38	39.87	3.85	25.09	19.06	8.95
	{ Calc. water-free . . . . .		4.75	43.22	4.17	27.20	20.66	9.70
Soja bean biscuit	{ In original . . . . .	27.66	5.33	16.71	1.55	12.84	35.91	.....
	{ Calc. water-free . . . . .		7.37	23.10	2.14	17.75	49.64	.....
Casoid flour . . . .	{ In original . . . . .	10.01	2.46	85.56	.....	.....	0.50	none
	{ Calc. water-free . . . . .		2.73	95.08	.....	.....	0.56	none
Almond meal . . .	{ In original . . . . .	8.51	6.42	50.62	2.86	15.96	15.63	7.18
	{ Calc. water-free . . . . .		7.02	55.32	3.12	17.45	17.09	7.85

In the analysis of diabetic foods, the determination of starch, sugar and dextrin together is of greater value than of starch alone, since all three classes of carbohydrates are about equally injurious to diabetics, the starch and dextrans being converted into sugars by the digestive fluids. The nitrogen-free extract of cereal preparations corresponds closely with the sum of the starch, sugar and dextrin, but in the case of soja bean meal, almond meal and other products of legumes and oil seeds, as well as vegetables, it is considerably greater, as it includes pentosans and other substances.

#### METHODS OF ANALYSIS.

**Preparation of the Sample.**—Grind sufficiently fine in a mortar or mill to pass through a 1-mm. sieve.

**Determination of Water, Protein, and Ash.**—Follow the regular methods for cereal products (pages 285-287).

**Determination of Fat.**—Owing to the presence of gelatinized starch, sugars, and similar incrusting constituents direct ether extraction is usually not admissable. Proceed as with bread, page 343.



**Separation of the Carbohydrates** can be effected by Stone's method (pages 304, 305), but a very satisfactory idea of the solubility of these foods, which is of chief importance, can be gained by the determination of the cold water extract and reducing sugars.

**Determination of Starch, Sugar, and Dextrin.**—Determine together in diabetic preparations by the diastase method (page 292) omitting the preliminary washing with dilute alcohol. The results thus obtained with cereal products agree fairly well with the nitrogen-free extract calculated by difference in the usual manner, but this is not true of meal or biscuit made from soy beans, almonds, etc.

**Determination of Cold-water Extract.**—*McGill Method.\**—Weigh the equivalent of 10 grams of the moisture-free substance, finely ground, into a tared flask, and add water in several portions with gentle shaking till the contents of the flask weigh 110 grams. Cork the flask, then vigorously shake at intervals during 6 or 8 hours and allow to stand over night. Decant the supernatant liquid into the large tubes of a centrifuge and whirl till the sediment settles out. Filter the comparatively clear liquid, transfer 20 cc. of the filtrate, corresponding to 2 grams of the original sample, to a tared dish, evaporate to dryness and dry to constant weight, as in the determination of the total solids.

Additional information may be gained from the specific gravity of the 10% solution of the cold-water extract, best obtained by means of a pycnometer.

**Determination of Reducing Sugars.**—Determine in an aliquot part of the above 10% solution, diluted to proper strength.

**Effects of Subsequent Heating.**—It is hardly fair in the case of those farinaceous foods which, according to directions, are to be subsequently subjected to heating, or boiling with water or milk, to condemn them as containing much insoluble matter, without comparing the figures expressing results of the analyses of the raw foods, calculated to the water-free basis, with those obtained on analyzing the food after boiling or otherwise cooking with pure distilled water, for a length of time specified in the directions, and afterwards drying. It is possible that the presence in the food of diastase, or other ferment, may be depended on to hydrolyze a whole or a portion of the starch, and only by such comparison will this be shown.

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\* Lab. Int. Rev. Dept. Canada, Bul. 59, 1898.



**Microscopical Examination** of the food is of value in determining its general character, showing especially whether or not starch is present in its original form, or has been converted in whole or in part. The particular varieties of cereal grain employed are generally evident, as well as the presence and proportion of the different tissues of the grain.



## CHAPTER XI.

### TEA, COFFEE, AND COCOA.

#### TEA.

**Nature and Classification.**—Tea consists of the prepared leaves or leaf buds of *Camellia Thea* also known as *Thea chinensis*.

The best teas are made from young leaves only, the Chinese teas being classified with reference to the age and position of the leaf on the young shoot. Thus, the very choicest Chinese tea, rarely found outside of China, is prepared from the youngest or end leaves of the shoot, which are scarcely more than buds, and form the tea known as *pekoe tip*, or *flowery pekoe*. The next leaves are the *orange pekoe* and *pekoe*, which produce a very high grade of tea, while next in order as to age, size, and grade of leaf are the *souchong 1st* and *2d*, and the *congou*, producing teas called by the same names.

More than 50% of the tea consumed in the United States comes from China, and over 40% from Japan, the remainder being derived largely from India, Ceylon, and other East Indian ports.

In the manufacture of tea the fresh leaves, which are nearly 80% water, are rolled, withered by exposure to light, heat, and air, and finally dried or "fired" by treatment with artificial heat over charcoal fires, or in properly constructed furnaces.

Teas are divided into two groups, *black* and *green*, which differ from each other, not as formerly supposed in being derived from different plants, but in their process of manufacture, the same species of plant furnishing both varieties. Genuine green tea is prepared by first steaming and afterwards drying the leaves while still fresh, thus retaining the bright color. Black tea is allowed to undergo oxidation or fermentation by exposure to the sun, which gradually turns the leaves black. Less tannin is present in black tea than in green.



**Composition of Tea.**—König gives the following composition of fully developed tea leaves, being the mean of 50 to 70 analyses:

Water.	Nitrogenous Substances.	Theine.	Essential Oil.	Fat, Chlorophyll, and Wax.	Gum, Dextrin, etc.	Tannin.	Pectin, etc.	Crude Fiber.	Ash.
9.51	24.50	3.58	0.68	6.39	6.44	15.65	16.02	11.58	5.65

Though the nitrogenous substances of tea predominate in amount over any other class of constituents, yet, with the exception of theine or



FIG. 72.—*a*, Flowery Pekoe; *b*, Orange Pekoe; *c*, Pekoe; *d*, Souchong, 1st; *e*, Souchong, 2d; *f*, Congou; *a, b* (when mixed together), Pekoe; *a, b, c, d, e* (when mixed together), Pekoe Souchong. If there be another leaf below *f*, it is termed Bohea. At base of leaves are buds 1, 2, 3, 4, from which new shoots spring. (After Money.)

caffeine, they have been little studied. Theine, tannin, and essential oil give to the infusion of tea its chief characteristics.

Zollinski\* gives the following summarized results of analyses of a number of the cheaper grades of Chinese black tea:

\* Zeits. anal. Chem., 1898, 37, 365.



	Water.	Total Nitrogen.	Albuminoid and Amido-nitrogen.	Protein, N $\times$ 6.25.	Theine.	Ash.	Soluble Ash.	Insoluble Ash.
Maximum....	11.57	4.12	3.78	23.83	2.06	6.78	31.17	61.03
Minimum....	9.96	3.76	3.37	21.06	1.14	4.79	28.13	57.74
Average.....	10.58	3.93	3.52	22.01	1.55	5.94	29.67	59.75

A very complete series of analyses of tea was made by Joseph F. Geissler in 1884,\* from which the following summaries are taken:

	Number of Analyses.	Moisture.	Half-hour Extract.	Total Extract.	Insoluble Leaf.	Tannin.	Theine.	Total Ash.	Soluble Ash.	Insoluble Ash.	Ash Insol. in Acid.
Indian: Maximum..	6	6.10	39.66	45.64	53.07	18.86	3.3	5.79	3.68	2.22	.296
Minimum..	..	5.56	37.80	41.32	48.53	13.04	1.8	5.42	3.24	1.93	.137
Average....	..	5.81	38.77	42.94	51.24	14.87	2.7	5.62	3.52	2.12	.178
Oolong: Maximum..	13	6.88	44.02	48.87	53.15	20.07	3.50	6.11	3.71	3.17	.838
Minimum..	..	5.09	34.10	40.6	44.8	11.93	1.15	5.44	2.60	1.84	.266
Average....	..	5.89	37.88	43.32	50.7	16.38	2.32	5.81	3.2	2.68	.507
Congou: Maximum..	11	9.15	32.14	37.06	63.85	13.89	2.87	6.48	3.52	3.86	1.31
Minimum..	..	7.65	23.48	27.48	54.5	8.44	1.70	5.52	2.28	1.90	.32
Average....	..	8.37	28.40	34.35	57.2	11.54	2.37	5.75	3.06	2.68	.425

Kenrick † gives the following averages of a series of analyses of tea made by him in 1891:

	Number of Analyses.	Substances Extracted by 10 Minutes' Infusion.			Moisture.	Soluble Ash.	Insoluble Ash.	Total Ash.	Ratio of Total Solids to Tannin.
		Total Solids.	Tannin.	Theine.					
Congou tea.....	10	23.37	5.18	2.65	7.60	3.55	2.28	5.83	4.51
Assam tea.....	3	38.53	7.49	2.98	5.75	3.69	2.16	5.84	3.81
Ceylon tea.....	2	27.45	7.85	2.68	6.31	3.34	1.88	5.22	3.50
Unclassed black.....	13	23.76	5.40	2.82	6.54	3.53	2.37	5.90	4.40
Japan.....	18	30.07	9.38	2.45	4.00	3.62	2.73	6.35	3.20
Gunpowder.....	2	28.55	8.00	2.39	4.72	3.36	3.70	7.06	3.57
Young Hyson.....	5	34.22	10.98	2.52	5.40	3.83	2.10	5.93	3.12

The ash of many genuine teas has been examined by Battershal ‡ with the following results:

\* Am. Grocer, Oct. 23, 1884.

† Canada Inland Rev. Dep. Bul. 24.

‡ Food Adulteration and its Detection.



	Oolong. Average of 50 Samples.	Japan.	Spent Black Tea.
Total ash . . . . .	6.04	5.55	2.52
Soluble in water . . . . .	3.44	3.60	0.28
Per cent soluble. . . . .	57.00	64.55	11.11

## COMPOSITION.

Silica . . . . .	11.30	9.30	27.75
Chlorine. . . . .	1.53	1.60	0.79
Potash. . . . .	37.46	41.63	
Soda. . . . .	1.40	1.12	
Ferric oxide. . . . .	1.80	1.12	} 16.00
Alumina. . . . .	5.13	4.26	
Manganic oxide. . . . .	2.10	1.30	
Lime . . . . .	9.43	8.18	19.66
Magnesia . . . . .	8.00	5.33	11.20
Phosphoric acid. . . . .	12.27	16.62	15.80
Sulphuric acid . . . . .	4.18	3.64	1.10
Carbonic acid. . . . .	5.40	5.90	6.70
	100.00	100.00	99.00

Kozai \* gives the following as the results of analyses made by him of Japanese teas:

	Unprepared Leaves.	Green Tea.	Black Tea.
Caffeine or theine. . . . .	3.30	3.20	3.30
Ether extract . . . . .	6.49	5.52	5.82
Hot-water extract . . . . .	50.97	53.74	47.23
Tannin (as gallotannic acid) . . . . .	12.91	10.64	4.89
Other nitrogen-free extract . . . . .	27.86	31.43	35.39
Crude protein . . . . .	37.33	37.43	38.90
Crude fiber. . . . .	10.44	10.06	10.07
Ash . . . . .	4.97	4.92	4.93
Albuminoid nitrogen. . . . .	4.11	3.94	4.11
Caffeine nitrogen. . . . .	0.96	0.93	0.96
Amido-nitrogen . . . . .	0.91	1.13	1.16
Total nitrogen . . . . .	5.97	5.99	6.22

## PROXIMATE COMPONENTS AND ANALYTICAL METHODS.

**Preparation of Sample.**—Grind the material so as to pass a sieve with holes 0.5 mm. in diameter.

**Moisture, Ether Extract, and Crude Fiber** are determined in the same weighed portion of 2 grams, by methods described under cereals (p. 285). **Acidity** determination by hydrogen electrode is described on p. 1035.

\* Bul. 7, Imperial College of Agriculture, Japan.



**Determination of Protein.**—Determine total nitrogen by the Kjeldahl or Gunning method; from this subtract the nitrogen due to caffeine (obtained by dividing by 3.464) and multiply the difference by 6.25.

**Determination of Total Ash.**—Burn 2 grams of the material to a white ash in a platinum dish at a faint red heat. The total ash of pure tea should not be less than 4 nor more than 7%.

**Determination of Soluble and Insoluble Ash.**—Transfer the total ash, as obtained above, to a beaker with 50 cc. of hot water, boil, collect the insoluble ash on a Gooch crucible, wash with hot water, dry below redness, and weigh. To obtain the soluble ash subtract the insoluble from the total ash.

**Determination of Ash Insoluble in Acid.**—Proceed as in the determination of water-insoluble ash, using, however, 25 cc. of 10% hydrochloric acid instead of water for the boiling.

**Determination of Alkalinity of Ash.\***—This is expressed in terms of cc. of tenth-normal acid required for the ash of 1 gram of sample.

**Soluble Ash.**—Cool the filtrate from the determination of insoluble ash, as described above, and titrate with tenth-normal hydrochloric acid, using methyl orange as an indicator.

**Insoluble Ash.**—Add excess of tenth-normal hydrochloric acid (usually 10 to 15 cc.) to the ignited insoluble ash as obtained above in the platinum dish, heat to the point of boiling over an asbestos plate, cool, and titrate excess of hydrochloric acid with tenth-normal sodium hydroxide, using methyl orange as an indicator.

**Determination of Essential Oils.**—Distil 100 grams of the tea with 800 cc. of water, and shake out the distillate with several portions of ether. The residue from the combined ether extracts contains the volatile oil.

**Determination of Insoluble Leaf.**—*Winton, Ogden, and Mitchell Method.*†—Boil 2 grams of the unground tea for 30 minutes with 200 cc. of water, taking care to so adjust the flame as to avoid appreciable concentration. Collect the insoluble leaf on a tared filter, dry on a watch glass until no moisture is apparent, then transfer to the weighing bottle and complete the drying in a boiling water-oven. If the amount of insoluble leaf is above 60%, the presence of spent or exhausted leaves may be suspected.

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 107 (rev.), p. 69.

† Conn. Exp. Sta. Ann. Rep., 1898, p. 132.



Doolittle and Woodruff\* boil for 1 hour, but in other respects follow the above method.

**Determination of Extract.**—By this term is meant the total amount of water-soluble matter in tea, including such compounds as tannin, caffeine, albuminous matter, dextrin, gum, certain parts of the ash, etc.

The value of a tea from a food standpoint depends obviously upon the character and amount of the extract, rather than on the composition of the dry tea. The relative composition of the extract and of the insoluble leaf, as found by Eder, is given in the following table:

	Extract.	Insoluble Leaf.
	Per Cent.	Per Cent.
Dry matter .....	40.	60.
Nitrogenous substances .....	12.	12.7
Theine .....	2.	
Tea oil .....	0.6	
Resin, chlorophyll, etc. ....	.....	7.2
Tannin .....	10.	
Extractives .....	12.	10.
Ash .....	1.7	2.3
Potash .....	0.94	0.29
Lime .....	0.04	0.58
Phosphoric anhydride .....	0.13	1.03
Silica .....	0.21	0.68

The sum of the percentages of insoluble leaf and moisture subtracted from 100 gives the percentage of extract.

**Determination of Tannin.**—*Löwenthal-Proctor Method.*†—1. *Reagents:*

(a) Potassium permanganate solution containing about 1.33 grams per liter.

(b) Tenth-normal oxalic acid solution (6.3 grams per liter).

(c) Indigo carmine solution, containing 6 grams indigo carmine (free from indigo blue) and 50 cc. concentrated sulphuric acid per liter.

(d) Gelatin solution, prepared by soaking 25 grams gelatin for an hour in a saturated sodium chloride solution, heating till the gelatin is dissolved, and making up to a liter after cooling.

(e) Mixture of 975 cc. saturated sodium chloride solution and 25 cc. concentrated sulphuric acid.

(f) Powdered kaolin.

\* U. S. Dept. of Agric., Bur. of Chem., Bul. 150, 1907, p. 48.

† Jour. Soc. Chem. Ind., 3, 1884, p. 82; U. S. Dept. of Agric., Div. of Chem., Bul. 13, 1892, p. 890.



Obtain the value of the potassium permanganate solution in terms of the tenth-normal oxalic acid solution.

2. *Process*.—Boil 5 grams of the powdered tea for half an hour with 400 cc. of water, cool, and make up to 500 cc. in a graduated flask. To 10 cc. of the infusion (filtered if not clear) add 25 cc. of the indigo carmine solution and about 750 cc. of water. Then add from a burette the potassium permanganate solution, a little at a time while stirring, till the color becomes light green, then cautiously drop by drop till the color changes to bright yellow, or further to a faint pink at the rim, matching in any event the color adopted in standardizing. The volume in cubic centimeters of permanganate furnishes value *a* of the formula.

Mix 100 cc. of the clear infusion of tea with 50 cc. of gelatin solution, 100 cc. of salt acid solution, and 10 grams of kaolin, and shake several minutes in a corked flask. After settling, decant first the clear supernatant liquid through a filter, and finally bring the precipitate upon the filter. Mix 25 cc. of the filtrate (corresponding to 10 cc. of the original infusion) with 25 cc. of the indigo carmine solution, and about 750 cc. of water, and titrate with permanganate as before. The volume in cubic centimeters of permanganate used gives value *b*.

*a*=quantity of permanganate solution required to oxidize all oxidizable substances present.

*b*=quantity of permanganate solution required to oxidize substances other than tannin.

$\therefore a - b = c$ , permanganate required for the tannin. Assuming that 0.04157 gram tannin (gallotannic acid) is equivalent to 0.063 gram oxalic acid, the tannin in the tea is readily calculated.

As recommended by Doolittle and Woodruff\* the determination may be conveniently made on aliquot portions of the solution obtained in the determination of insoluble leaf.

*Method of Fletcher and Allen*.†—This method depends upon the precipitation of the tannin and other astringent matters in tea infusion by lead acetate, the point of complete precipitation being indicated by an ammoniacal solution of potassium ferricyanide.

Five grains of neutral lead acetate are dissolved in water, made up to 1 liter, and after standing the solution is filtered.

As an indicator, 0.05 gram of pure potassium ferricyanide is dissolved in 50 cc. of water, and an equal volume of concentrated ammonia-

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 105, 1907, p. 49.

† Chem. News, 29, pp. 169, 189.



water is added. This indicator produces a red coloration with tannin, gallic acid, or gallotannic acid in solution, being so sensitive that a drop of the indicator will detect 1 part of tannin in 10,000 parts of water.

Three separate quantities of 10 cc. each of the standard lead acetate solution, as above prepared, are measured into as many beakers, and each diluted to 100 cc. with boiling water. Two grams of powdered tea are boiled in 250 cc. of water, and varying quantities of this decoction are measured from a burette or pipette into the beakers containing the lead solution, the first beaker receiving, say, 12 cc., the second 15 cc., and the third 18 cc., in the case of black tea, and, with green tea, 8, 10, and 12 cc., respectively.

About 1 cc. of each of these trial quantities is removed from the various beakers by means of a pipette, passed through small filters, and tested with the ammoniacal ferricyanide indicator, the drops of filtered solution being allowed to fall directly on spots of the indicator, previously placed on a white slab or plate.

It is thus easy to ascertain the *approximate* amount of tea solution which it is necessary to add to produce a pink coloration with the indicator, so that by repeated tests, nearly the right amount may be added at once. If no coloration in a given case is produced when a drop of the filtrate from the solution in the beaker is allowed to fall on the drop of indicator solution, a little more of the tea decoction is added, and this process is repeated until the pink color is apparent.

It should be noted how much of the tea decoction is necessary to add to 100 cc. of pure water, that a drop of the solution may produce the pink coloration with the ferricyanide, and this amount should be subtracted from the amount of decoction found necessary to add to the known lead solution in the beaker. It was found by repeated experiment that 10 cc. of lead solution would precipitate 0.01 gram of pure gallotannic acid; hence, carrying out the process exactly as above described, 125 divided by the number of cubic centimeters of tea decoction required gives the percentage of tannin in the sample.\*

**Theine or Caffeine** ( $C_8H_{10}N_4O_2$ ).—This alkaloid when pure exists in white silky needles. It is odorless and sparingly soluble in cold water, but more so in hot. It is less soluble in alcohol, and almost insoluble in ether. It readily dissolves in chloroform. It is present

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\* This process estimates the total astringent matter, all of which is counted in as tannin.



in tea, coffee, and kola. Graf\* has shown that the amount of caffeine present in tea is in most cases proportional to the commercial value and quality.

*Detection.*—Caffeine may be detected, if present in a suspected residue, by the so-called "murexid test," which is made with the material in a solid state, or with the residue from the evaporation of a liquid. A small quantity of the solid or powdered material is heated in a white porcelain dish and covered with a few drops of strong hydrochloric acid, after which a fragment of potassium chlorate is immediately added. The mixture is then evaporated to complete dryness on the water-bath, whereupon, if caffeine is present, a reddish-yellow or pink color is produced. After cooling, the residue is treated with a very little ammonia water applied on the point of a stirring-rod. In the presence of caffeine, a purple color (that of murexoin) is produced on application of the ammonia.

**Determination of Theine or Caffeine.**—*Dvorkovitsch Method.*†—Digest 10 grams of the powdered tea with 200 cc. of boiling water for 5 minutes and decant the solution; repeat the treatment twice, and boil the residue with 200 cc. of water. Make up the combined solutions to 1000 cc. and extract a portion with petroleum ether to remove fat, etc. To 600 cc. of the fat-free solution (equivalent to 6 grams of tea) add 100 cc. of 4% barium hydroxide solution, mix and filter. To 583 cc. of the filtrate (equivalent to 5 grams of tea) add 100 cc. of a 20% solution of sodium chloride, and extract three times with chloroform. Distil the greater part of the chloroform from the combined extracts, place the residue in a tared dish, evaporate the remainder of the chloroform, dry at 100° C., and weigh. The caffeine is usually of sufficient purity to render a nitrogen determination unnecessary.

*Doolittle and Woodruff*‡ proceed as follows: Extract in a separating funnel with petroleum ether 225 cc. of the filtrate from the determination of insoluble leaf (p. 382) made up to 500 cc. To the fat-free portion add 50 cc. of a 4% barium hydroxide solution, shake well, and filter. To the filtrate add 50 cc. of a 20% sodium chloride solution and proceed as above described.

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\* Forsch, Ber., 4, 1897, pp. 88, 89.

† Ber. d. chem. Ges., 24, 1891, p. 1945; U. S. Dept. of Agric., Bur. of Chem., Bul. 107 (rev.), p. 150.

‡ Loc. cit.



*Stahlschmidt Method\* Modified by Spencer.*†—Boil gently 5 grams of the finely powdered tea in a flask with 420 cc. of water for 30 minutes, cool, add sufficient lead subacetate (solution or powder) to remove precipitable substances, make up to 500 cc., and filter through a dry paper. Delead an aliquot of 400 cc., equivalent to 4 grams, with hydrogen sulphide, boil off the excess of hydrogen sulphide, filter, wash with hot water, and evaporate the filtrate to about 50 cc. Shake out the solution in a separatory funnel with several portions of chloroform until all the theine has been extracted, evaporate off the chloroform from the combined extracts in a tared flask, and dry the theine 2 hours or to constant weight at 75° C.

Bartlett‡ has found that the Stahlschmidt method in essentially the form given by Spencer gives satisfactory results.

**Facing.**—The most common form of tea adulteration, if such it may be called, is the practice of "facing" the dried leaves, or treating them with certain pigments and coloring materials to impart a bright color or gloss to the tea, thus causing an inferior grade to appear of better quality than it really is. This practice is more often applied to green tea. The materials for facing include such substances as Prussian blue, indigo, plumbago, and turmeric, often accompanied by such minerals as soapstone, gypsum, etc. Only a small amount of foreign material is actually added to the tea, but the adulteration consists in the deceptive appearance imparted thereto.

Battershal has examined various samples of the preparations used in Japan for facing tea. He found in one case the following composition: Soapstone, 47.5%; gypsum, 47.5%; Prussian blue, 5%. Another sample consisted of soapstone, 75%; indigo, 25%. A third was composed of soapstone, 60%, and indigo, 40%. In applying the facing to the tea, the latter is first heated in an iron pan over the fire, the facing mixture is then added while still hot, and the whole is stirred briskly till the desired color is imparted. The Chinese and Japanese do not face the tea which they themselves consume, but only that intended for export trade.

**Detection of Facing.**—The most delicate test for facing is to examine under the microscope, or lens, the dust obtained by sifting the leaves or the sediment obtained after shaking them with water. Plumbago appears

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\* Pogg. Ann., 112, p. 441.

† Jour. Anal. Chem., 4, 1890, p. 390.

‡ Jour. Assn. Off. Agric. Chem., 5, 1917, p. 21.



glossy black, soapstone gray, gypsum white, Prussian blue, ultramarine and indigo shades of blue, and turmeric yellow. Prussian blue is decolorized by sodium hydroxide solution. Ultramarine is not affected by alkali but is decolorized by hydrochloric acid. Indigo is not decolorized by either reagent.

Read \* rubs the siftings with a spatula on sheets of white and black paper and removes the loose dust. The colors after this treatment are recognized under the lens as streaks on the paper. West † detects Prussian blue by the blue spots formed by sprinkling the ground tea on filter paper moistened with oxalic acid solution and drying.

Prussian blue if present in considerable amount may be detected in the sediment, as above obtained, by the blue precipitate which forms after dissolving in hot alkali, filtering, acidifying with hydrochloric acid, and then adding a drop of ferric chloride. If the residue on the paper after treatment with hot alkali, on removal to a porcelain dish and treatment with concentrated sulphuric acid, yields hydrogen sulphide (recognized by its odor or by the blackening of lead acetate paper) ultramarine is indicated.

Such minerals as gypsum and soapstone are readily separated as a sediment by shaking the leaves in water, and the sediment is examined by the appropriate qualitative methods for these substances.

**Spent or Exhausted Leaves.**—These consist of leaves of tea that have been previously steeped or infused, and afterwards rerolled and dried. Such leaves are sometimes mixed with tea as an adulterant. Any considerable admixture of spent leaves is evident, both by the extremely low ash, and the abnormally small proportion of water-soluble ash in the sample. It is rare that the total ash of genuine tea is under 5%, while the soluble ash is seldom less than 3%.

The ash of spent tea leaves sometimes runs as low as 2.5%, of which generally not more than 0.3 to 0.8 per cent is soluble. Spent leaves are also naturally low in tannin and in extract.

If the extract is much below 32%, spent leaves may be suspected. Allen determines the per cent of spent leaves by subtracting the per cent of extract from 32, multiplying by 100 and dividing by 30.

The use of spent or exhausted leaves as an adulterant is very rare at present, though formerly of common occurrence.

**Foreign Leaves as a Substitute for Tea.**—This sophistication is not common, but the detection of leaves other than tea is readily accom-

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\* U. S. Treasury Decision, No. 32322.

† Jour. Ind. Eng. Chem., 4, 1912, p. 528.



plished by a careful examination of the shape and character of the leaves. For this purpose the dried leaves are opened out by soaking a short time in hot water, after which they are spread upon a glass plate, and examined by the aid of a magnifying-glass.

The genuine tea leaf (Fig. 73) is very characteristic, and is readily distinguished from other leaves. It is oval or lanceolate, 5 to 8 cm. long and 2 to 3 cm. wide. It is short-stemmed, somewhat thick and fleshy, attenuated at the bottom and usually pointed at the top. At a certain height from the base, from a third to a quarter up, the smooth or wavy border becomes peculiarly, though not deeply, serrated in a regular manner, the serrations, which are hook-shaped, continuing to the tip of the leaf. Mature leaves always show these serrations, but they are somewhat obscure in young leaf buds. The latter, however, are rarely found in this country. The veins extend outward from the central rib nearly parallel to each other, but before reaching the border, each bends upward to form a loop with the one above.

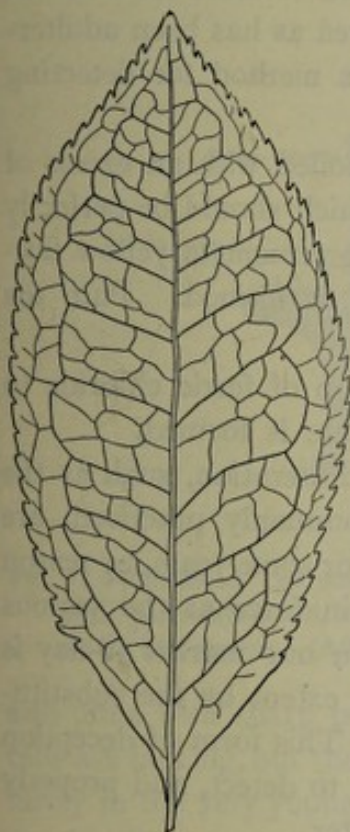


FIG. 73.—The Leaf of Genuine Tea.

Foreign leaves, said to be used as adulterants, are those of the willow, poplar, elder, birch, elm, and rose, but the writer has never found any of these in tea. All of them differ materially from the genuine tea leaf, and if foreign leaves are apparent in a sample under

examination, they should be compared with various leaves collected by the analyst for the purpose.

**Stems and Fragments.**—These, as well as “tea dust,” are apparent by an examination of the leaves, opened out in hot water as explained above. The ash of tea stems and dust is abnormally high.

Besson\* and Deuss† oppose fixing a maximum limit for stems on the ground that the more expensive sorts often contain more stems than the cheaper. This is due partly to methods of gathering and partly to the presence of siftings with low stem content in the cheaper grades.

The term “lie tea” is applied to an imitation of tea, consisting of

\* Chem. Ztg., 39, 1915, p. 82.

† Chem. Weekbl., 13, 1916, p. 66.



fragments, stems, and tea dust, mixed with foreign leaves, mineral matter, gum, etc. The ash of such "tea" has been found as high as 50%. Such imitations are now almost unknown. Make-weight substances, such as brick-dust, iron salts, metallic iron, sand, etc., have been found in tea. If present, they are to be found in the sediment, obtained on shaking out the tea in water.

**Added Astringents.**—Catechu is sometimes said to be added to tea to give it increased astringency, especially to such tea as has been adulterated by the addition of exhausted tea. Hagar's method for detecting catechu is as follows:

A hot-water extract of the tea (1 to 100) is boiled with an excess of litharge and filtered. To a part of the filtrate, which should be perfectly clear, nitrate of silver is added. If catechu be present, a yellow flocculent precipitate, rapidly becoming dark-colored, is formed. Pure tea treated in like manner gives a gray precipitate.

Spencer\* adds, instead of silver nitrate, a drop of ferric chloride to the clear filtrate. With catechu a green precipitate is formed.

As a matter of fact the worst forms of tea adulteration, such as the actual substitution of foreign leaves, once so commonly practiced, are now extremely rare in this country and have been for some years, by reason of the careful system of government inspection in force at the various ports of entry. The greater portion of the tea on our market to-day is genuine, but fraud is practiced to a considerable extent by the substitution of inferior grades for those of good quality. This form of deception is in many cases beyond the power of the analyst to detect, and properly comes within the realm of the professional tea-taster.

**Tea Tablets.**—Finely ground tea of varying quality is sometimes pressed into tablets, to be used by travelers and campers for preparing a beverage, by simply dissolving in hot water.

The composition of one of these preparations sold under the name of Samovar Tea Tablets, analyzed by the Mass. State Board of Health, is as follows:

Water .....	8.7
Theine .....	2.25
Extract. ....	54.4
Ash .....	5.4
Soluble ash. ....	2.8
Insoluble ash .....	2.6

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\* U. S. Dept. of Agric., Div. of Chem., Bul. 13, p. 885.



**Microscopical Structure of Tea.**—The powdered tea may be examined directly in water-mount. Schimper recommends treating the powdered tea with chloral hydrate or potash lye, to render it more transparent.

By far the most characteristic element is the peculiarly shaped sclerenchyma, or stone cell, *st*, Fig. 74, entirely unlike anything to be found in other leaves. These cells are very irregular in form, being sometimes star-shaped, sometimes branched, almost always with deeply wrinkled sides,

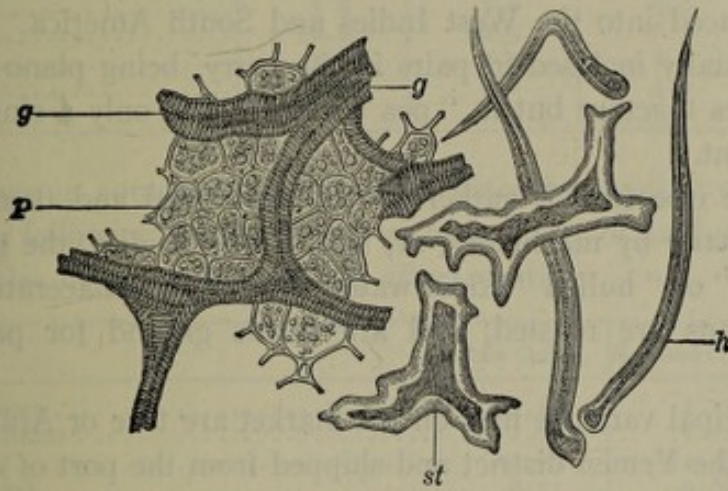


FIG. 74.—Powdered Tea under the Microscope.  $\times 160$ . *g*, end of leaf nerve; *p*, chlorophyll parenchyma; *st*, stone cells; *h*, hairs. The tissues were warmed in potash to render transparent. (After Moeller.)

and often with sharp points. In most foreign leaves such sclerenchyma cells are lacking, but they are abundant in all genuine tea leaves, excepting rarely in the very young leaves, where they are sometimes not fully developed. They are especially numerous in the main vein and in the stem. They may be seen to best advantage in a section of the stem, or midrib, made parallel to the surface of the leaf. To make such a section, soak the leaf first in water, and afterwards dry in alcohol. The interior of the leaf is composed chiefly of ground tissue, having rounded cells full of chlorophyll grains and the fibro-vascular bundles of the veins.

Other important characteristics are the peculiar hair growth on the under epidermis, *B*, which is apparent in nearly all teas, also crystal rosettes of calcium oxalate, which are nearly always present, even in fragments of tea leaves, but not in all foreign leaves. The peculiar structure of the lower epidermis, *B*, with its numerous stomata is also to be noted. See Figs. 189 and 190, Pl. XVIII.



## COFFEE.

**Nature of Coffee.**—Coffee is the seed of the *Coffea arabica*, a tree which, when under cultivation, is not allowed to exceed twelve feet in height, but when wild sometimes reaches a height of twenty feet. It is indigenous in Southern Abyssinia, and was cultivated in Arabia in the sixteenth century, and in the East Indies in the seventeenth, afterward being introduced into the West Indies and South America. The coffee-beans are usually inclosed in pairs in the berry, being plano-convex with their flat sides together but in "pea berry" coffee only a single, rounded bean is present.

When the ripe fruit is gathered, it is first dried and then freed from the hulls, usually by machinery, or, in the West Indies, the green berries are "pulped" or "hulled" under water by a peculiar macerating machine. The raw beans are roasted, and afterwards ground for preparing the infusion.

The principal varieties now on the market are true or Arabian Mocha produced in the Yemen district and shipped from the port of Aden, Abyssinian or long berry Mocha, Java produced on the island of Java under government supervision, Rio and Santos, the leading Brazilian varieties shipped from the ports of Rio Janeiro and San Paulo respectively, Maracaibo a Venezuelan coffee, and Bogota produced in Colombia. Porto Rican and other West Indian varieties, also the product produced in the islands of the Pacific, often shipped under the name of Java, and various African coffees, are of considerable importance.

Brazil furnishes more than half the world's supply of coffee, and nearly 75% of that consumed in the United States.

**Constituents of Coffee.**—Most of the coffee in the retail market is roasted, being sold either in the whole bean or ground.

The chief constituents of raw coffee, besides water, are oil, cellulose, sugar, pentosans, dextrins, "caffetannic acid" (chlorogenic and coffalic acids), protein, caffeine, coffearine (an alkaloid), and ash.

During roasting the sugar is largely caramelized, the caffetannic acid reduced, the bean rendered more brittle, and certain flavors are developed. Various substances have been named as products of roasting. Of these caffeol, a volatile oily substance, has long been considered the chief aromatic constituent, but its identity is now disputed. Several authors have detected pyridine. There is a slight loss of caffeine during roasting.



The following summary of analyses of coffee of various kinds made by König show in general its composition:

	Raw Coffee.		Roasted Coffee.	
	Minimum.	Maximum.	Minimum.	Maximum.
Water.....	8.0	12.0	0.4	4.0
Caffeine.....	0.8	1.8	0.8	1.8
Fat.....	11.4	14.2	10.5	16.5
Reducing sugar.....	5.8	7.8	0.0	1.1
Cellulose.....	16.6	42.3	26.3	51.0
Total nitrogen.....	1.1	2.2	1.3	2.7
Ash.....	3.5	4.0	4.0	5.0

The change in composition that takes place in roasting coffee is well shown by the following figures, which give the mean of analyses by König of four samples of coffee before and after roasting:

	Raw Coffee.	Roasted Coffee.
Water.....	11.23	1.15
Caffeine.....	1.21	1.24
Fat.....	12.27	14.48
Sugar.....	8.55	0.66
Cellulose.....	18.17	10.89
Nitrogenous substances.....	12.07	13.98
Other non-nitrogenous matter.....	32.58	45.09
Ash.....	3.92	4.75

#### COMPOSITION OF THE ASH OF COFFEE.\*

Constituents.	Mocha.	Maracaibo.	Java.	Rio.
Sand.....	1.44	0.72	0.74	1.34
Silica (SiO <sub>2</sub> ).....	0.88	0.88	0.91	0.69
Ferric oxide (Fe <sub>2</sub> O <sub>3</sub> ).....	0.80	0.89	1.16	1.77
Lime (CaO).....	7.18	5.06	4.84	4.94
Magnesia (MgO).....	10.68	11.30	11.35	10.60
Potash (K <sub>2</sub> O).....	59.84	61.82	62.08	63.60
Soda (Na <sub>2</sub> O).....	0.48	0.44	.....	0.17
Phosphoric acid (P <sub>2</sub> O <sub>5</sub> ).....	12.93	13.20	14.09	11.53
Sulphuric acid (SO <sub>3</sub> ).....	4.43	5.10	4.10	4.88
Chlorine (Cl).....	1.25	0.59	0.73	0.48
	100.00	100.00	100.00	100.00

The following are analyses of common varieties of roasted coffee, also of coffee substitutes and adulterated coffee made by Lythgoe:†

\* U. S. Dept. of Agric., Div. of Chem., Bul. 13, p. 904.

† An. Rep. Mass. State Board of Health, 1904, p. 320. U. S. Dept. of Agric., Bur. of Chem., Bul. 90, pp. 43-45.



## COMPOSITION OF ROASTED COFFEE.

Variety.		Moisture.	Ash.	Water Soluble Ash.	Sand.	Chlorine.	Alkalinity (cc. N/10 Acid) of		Soluble P <sub>2</sub> O <sub>5</sub> .	Insoluble P <sub>2</sub> O <sub>5</sub> .	Petroleum Ether Extract.	Index of Refraction of Extract at 30°.	Total Nitrogen.
							Ash of 1 Gram of Substance.	1 Gram of Ash.					
Santos	A	1.40	4.16	3.46	0.00	0.023	2.97	71.4	0.319	0.346	14.58	1.4754	2.26
	B	1.87	4.31	3.62	.00	.023	3.36	75.7	.286	.295	13.84	1.4754	2.26
	C	1.31	3.80	3.00	.00	.019	3.35	85.6	.273	.295	13.86	1.4750	2.39
Porto Rico	A	1.29	4.05	3.30	.00	.016	3.53	87.2	.305	.337	13.00	1.4752	2.28
	B	1.26	4.06	3.27	.00	.020	3.72	92.6	.226	.351	13.34	1.4750	2.26
	C	1.48	4.12	3.32	.00	.016	3.66	88.8	.333	.328	14.12	1.4760	2.33
Rio	A	1.76	4.06	3.40	.00	.020	4.16	102.3	.213	.166	13.38	1.4758	2.14
	B	2.34	3.91	3.24	.00	.021	3.17	81.2	.356	.227	13.71	1.4753	2.18
	C	2.10	3.74	3.06	.00	.023	3.22	86.6	.363	.236	13.53	1.4756	2.26
Mocha	A	2.05	4.05	3.25	.00	.016	3.94	97.4	.282	.351	14.84	*1.4737	2.28
	B	2.95	3.85	3.07	.00	.021	3.26	84.7	.333	.364	14.47	*1.4743	2.00
	C	2.40	3.80	3.00	.00	.012	3.54	93.3	.337	.545	15.18	*1.4740	2.02
Java	A	3.34	4.09	3.27	.00	.016	3.88	95.0	.258	.421	12.61	1.4752	2.48
	B	3.35	4.38	3.56	.00	.019	3.54	80.8	.194	.388	12.28	1.4758	2.35
	C	3.44	3.96	3.10	.00	.011	2.95	74.5	.235	.383	13.54	1.4752	2.56
Highest.....		3.44	4.38	3.62	.00	.023	4.16	102.3	.424	.545	15.18	1.4760	2.56
Lowest.....		1.26	3.74	3.00	.00	.011	2.95	71.4	.194	.166	12.28	1.4750	2.00
Average....		2.16	4.03	3.26	.00	.018	3.55	87.1	.285	.329	13.75	1.4754	2.27

Variety.		Cold Water Extract.	Alcohol Extract.	Reducing Sugars.	Starch by Diastase.	Crude Fiber.	Caffeine.	Ten Per Cent Extract.				
								Specific Gravity at 15°.	Immersion Re- fractometer Reading at 20°.	Index of Re- fraction at 20°.	Solids.	Ash.
Santos	A	20.80	16.83	0.52	2.28	13.41	1.25	1.0107	26.7	1.33770	2.64	0.40
	B	22.72	17.11	.68	1.00	11.02	1.10	1.0108	26.9	1.33777	2.66	.39
	C	21.70	17.80	.75	2.32	14.71	1.20	1.0101	26.0	1.33743	2.46	.30
Porto Rico	A	22.48	15.70	.50	2.17	13.11	1.38	1.0107	26.6	1.33766	2.60	.37
	B	21.76	16.36	.63	1.58	12.93	1.21	1.0104	26.3	1.33754	2.50	.36
	C	24.44	16.91	.54	2.62	12.50	1.32	1.0113	27.6	1.33804	2.77	.30
Rio	A	22.66	17.00	.68	2.82	14.08	1.11	1.0103	25.5	1.33724	2.48	.40
	B	22.61	17.34	.78	1.47	13.10	1.10	1.0101	25.8	1.33735	2.46	.36
	C	22.75	17.37	.61	2.62	11.91	1.17	1.0101	26.0	1.33743	2.46	.30
Mocha	A	24.00	18.01	1.78	2.30	11.22	1.16	1.0106	26.4	1.33758	2.65	.40
	B	20.27	17.06	.94	1.85	12.34	1.10	1.0101	26.3	1.33754	2.47	.36
	C	24.18	19.55	1.42	2.90	13.20	1.18	1.0111	27.3	1.33793	2.72	.40
Java	A	23.85	15.95	.32	2.95	13.43	1.34	1.0110	29.6	1.33777	2.63	.39
	B	22.19	15.45	.42	2.32	13.77	1.30	1.0107	26.5	1.33762	2.58	.38
	C	23.20	16.21	.66	3.34	14.75	1.27	1.0108	26.6	1.33766	2.62	.38
Highest....		24.44	19.55	1.78	3.34	14.75	1.34	1.0113	27.6	1.33804	2.77	.40
Lowest.....		20.27	16.45	.32	1.00	11.02	1.10	1.0101	26.0	1.33743	2.46	.30
Average....		22.63	17.03	.75	2.30	13.03	1.20	1.0105	26.6	1.33766	2.72	.37

\* Omitted from average.



## COMPOSITION OF COFFEE SUBSTITUTES AND OF ADULTERATED COFFEE.

Variety.	Moisture.	Ash.	Water Soluble Ash.	Sand.	Chlorine.	Alkalinity (cc. N/10 Acid) of		Soluble $P_2O_5$ .	Insoluble $P_2O_5$ .	Petroleum Ether Extract.	Index of Refraction of Extract at 30°.	Total Nitrogen.
						Ash of 1 Gram of Substance.	1 Gram of Ash.					
Roasted wheat.	5.60	5.71	2.82	0.00	.....	0.34	6.0	0.649	1.460	2.40	.....	1.84
Roasted chicory	5.55	4.37	2.27	.81	0.080	.95	21.8	.277	.314	.88	.....	1.10
Coffee and chicory. ....	5.08	3.96	3.14	.06	.026	3.05	77.0	.286	.323	8.32	.....	1.89
Coffee, chicory and pea hulls	3.64	4.97	4.05	.24	*.284	2.60	65.6	.472	.740	9.56	1.4745	2.17

Variety.	Cold Water Extract.	Alcohol Extract.	Reducing Sugars.	Starch by Diastase.	Crude Fiber.	Caffeine	Ten Per Cent Extract.				
							Specific Gravity at 15°.	Immersion Re- fractometer Reading at 20°.	Index of Re- fraction at 20°.	Solids.	Ash.
Roasted wheat	25.88	10.72	4.10	28.58	6.23	0.00	.....	.....	.....	.....	.....
Roasted chicory	72.92	34.39	19.34	2.10	5.91	.00	1.0307	45.0	1.34463	7.44	0.26
Coffee and chicory. ....	31.79	21.66	5.06	2.21	14.31	.95	1.0142	30.5	1.33915	3.62	.29
Coffee, chicory and pea hulls	25.00	14.25	3.00	3.78	17.87	1.00					

\* Admixture of salt.

## METHODS OF ANALYSIS.

**Preparation of the Sample.**—Grind so as to pass a sieve with holes 0.5 mm. in diameter.

**Determination of Moisture, Ether Extract, Fiber, Protein, and Ash** (including total, water-soluble, water-insoluble, acid-insoluble and alkalinity) is carried out as in the case of tea pp. 381 and 382). **Starch, Reducing Matters by Acid Conversion, Sucrose, and Reducing Sugars** may be estimated as in cereals, **Acidity** as described on page 1035.

**Determination of Ten per Cent Extract.** (See page 403).

**Determination of Caffeic Acid.**—*Krug Method*. \*—Although so-called caffeic acid has been shown to be a mixture of chlorogenic

\* U. S. Dept. of Agric., Div. of Chem., Bul. 13, p. 908.



and coffalic acids, the method of determining this mixed substance is still retained.

Two grams of the coffee are digested for 36 hours with 10 cc. of water, after which 25 cc. of 90% alcohol are added, and the digestion continued for 24 hours more. The liquid is then filtered, and the residue washed with 90% alcohol on the filter.

The filtrate, which contains tannin, caffeine, fat, etc., is heated to boiling and a boiling concentrated solution of acetate of lead is added, which precipitates out a caffetannate of lead,  $Pb_3(C_{15}H_{15}O_8)_2$ , containing 49% of lead. When this has become flocculent, it is separated by filtration, and washed on the filter with 90% alcohol, till the washings show

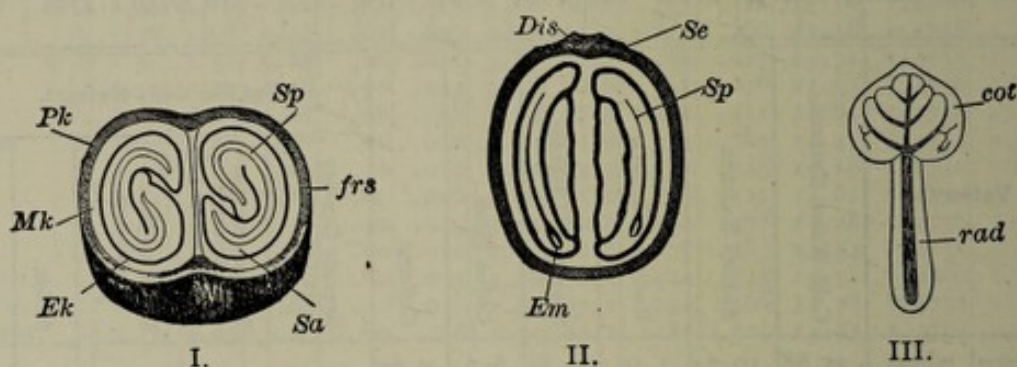


FIG. 75.—Coffee. I. cross-section of berry, natural size. *Pk* outer pericarp; *Mk* endocarp; *Ek* spermoderm; *Sa* hard endosperm; *Sp* soft endosperm. II. Longitudinal section of berry, natural size; *Dis* bordered disc; *Se* remains of sepals; *Em* embryo. III. Embryo enlarged; *cot* cotyledon; *rad* radicle. (TSCHIRCH AND OESTERLE.)

no lead with ammonium sulphide, and afterwards with ether, till free from fat. It is dried at 100° and weighed.

The weight of caffetannic acid is obtained by multiplying the weight of the precipitate by 652, and dividing by 1263.63.

*Woodman and Taylor's Modification.\**—To 2 grams of finely ground coffee (passing 0.5 mm. sieve), add 10 cc. of water, and shake for an hour in a mechanical shaking device. Add 25 cc. of 90% alcohol and shake again for half an hour. Filter and wash with 90% alcohol. Bring the united filtrate and washings, about 50 cc., to boiling, and add 6 cc. of saturated lead acetate solution. Separate the precipitated lead caffetannate by means of a centrifuge, decanting the supernatant liquid through a tared filter. Repeat the centrifugal treatment twice with 90% alcohol, decanting each time through the filter. Transfer the precipitate to the filter, and wash free from lead. Wash with ether, dry at 100°, and

\* U. S. Dept. of Agric., Bur. of Chem., Bul. 122, p. 82.



weigh. The weight of the precipitate multiplied by 0.516 gives the weight of caffetannic acid.

**Caffeine.**—*Gorter Method.\**—Moisten 11 grams of the finely powdered coffee with 3 cc. of water, allow to stand for half an hour, and extract for 3 hours in a Soxhlet or Johnson extractor with chloroform. Evaporate the extract, treat the residue of fat and caffeine with hot water, filter through a cotton plug, and wash with hot water. Make up the filtrate and washings to 55 cc., pipette off 50 cc., and extract four times in a separatory funnel with chloroform. Evaporate this chloroform extract in a tared flask, dry the caffeine at 100° C., and weigh.

Calculate the caffeine also from the nitrogen content.

*Lendrich and Nottbohm Method.†*—This method is recommended by Murray ‡ for the examination of decaffeinated coffee and other preparations containing small amounts of caffeine, as it yields lower and more accurate results than the Gorter method.

Moisten 20 grams of the sample, ground to pass a 1-mm. mesh, with 10 cc. of water, stir occasionally for 1–2 hours, and extract 3 hours with carbon tetra-chloride in an extraction thimble. To the extract add 1 gram of paraffin, distil off the solvent, and exhaust the residue with 1 portion of 50 cc. and 3 of 25 cc. of boiling water. Cool the combined aqueous extract, filter through a moistened paper, wash with hot water, add 10–30 cc. of 1% potassium permanganate solution, and allow to stand 15 minutes. Precipitate the excess of manganese as peroxide by means of a little 3% hydrogen peroxide solution containing 1% of glacial acetic acid added drop by drop, heat 15 minutes on a boiling water-bath, filter, wash with boiling water, evaporate the filtrate to dryness, and dry further in a boiling water-oven. Exhaust the dry residue with warm chloroform, decanting into a tared dish, remove the chloroform by evaporation, dry the residue 30 minutes in a boiling water-oven, and weigh the caffeine. Murray recommends that nitrogen be determined in the weighed residue as in the Gorter method.

#### ADULTERATION OF COFFEE.

According to the U. S. Standard roasted coffee is coffee which, by the action of heat, has become brown and developed its characteristic aroma, and contains not less than 10% of fat, and not less than 3% of ash.

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\* Annalen, 1908, 358, p. 327.

† Zeits. Unters. Nahr. Genussm., 17, 1909, p. 241.

‡ Jour. Ind. Eng. Chem., 5, 1913, p. 668.



**Imitation Coffee.**—Formerly, artificial coffee-beans containing no coffee whatever, but cleverly molded to imitate the original, were occasionally to be found, mixed with genuine, whole coffee.

“Coffee pellets” are occasionally sold in bulk to dealers as an adulterant of whole coffee. These do not closely resemble the real berries in appearance, but are approximately of the same size, and are not apparent to the purchaser when the whole coffee is ground at the time of purchase. A sample of these “pellets” examined recently was found to consist of roasted wheat mash, colored with red ocher.

**Coloring Coffee Beans.**—The practice of treating raw coffee beans in a manner somewhat analogous to the facing of tea leaves has been sometimes practiced, with a view to giving to cheaper or inferior grades the appearance of high-priced coffee. For this purpose various pigments have been employed, such as yellow ocher, chrome yellow, burnt umber, Venetian red, Scheele’s green, iron oxide, turmeric, indigo, Prussian blue, etc., the coffee beans being first moistened with water containing a little gum, and shaken with the pigment. As a rule such pigments, especially when inorganic, are best sought for either in the ash, or in the sediment obtained by shaking the coffee beans in cold water, using the ordinary qualitative chemical methods. Organic coloring matters can be best extracted with alcohol. Prussian blue and indigo are tested for as in the case of tea leaves (p. 387).

**Glazing.**—This is a more recent form of treatment of the whole bean, which consists in coating the beans by dipping in egg or sugar, or a mixture of the two, sometimes using various gums. Such glazing is alleged to improve the keeping qualities of the coffee, as well as to aid in clarifying the infusion, and if this is the sole purpose, the practice cannot be condemned as a form of adulteration. If, however, it is done to give inferior varieties of coffee a better appearance, in order to deceive the consumer, it clearly constitutes adulteration within the meaning of the law.

**Adulterants of Ground Coffee.**—Of the adulterants used in ground coffee the following have been found in Massachusetts: Roasted peas, beans, wheat, rye, oats, chicory, brown bread, pilot bread, charcoal, red slate, bark, and dried pellets, the latter consisting of ground peas, pea hulls, and cereals, held together with molasses.

**Methods of Detecting Adulterants.**—These methods are, as a rule, physical rather than chemical. A rough test of the genuineness of ground coffee consists in shaking some of the sample in cold water. Pure coffee,



under these conditions, usually floats on the surface, while the ordinary adulterants, such as cereals, chicory, mineral ingredients, etc., sink, the grains of chicory coloring the water a brownish-red as they subside.

Macfarlane recommends the use of a saturated solution of common salt, in which a portion of the suspected sample, divided in small grains, is shaken in a test-tube. If the liquid is colored pale amber, while all or nearly all the material floats, the coffee is pure. Any considerable sediment at the bottom of the tube, accompanied by a dark-yellow to brown color imparted to the liquid, indicates adulteration by roasted cereals, or chicory, or both.

A careful examination of the coarsely crushed grains of a ground sample with the naked eye will often serve to detect, and in some cases identify, certain adulterants, such as chicory and ground peas or beans. A magnifying-glass will aid in such an examination, and the observer can often separate the various ingredients of a coffee mixture, first spreading a small portion of the sample on a sheet of white paper. The chicory grains are apparent from their dark and somewhat gummy appearance, and can usually be recognized by crushing them between the teeth. Their soft consistency and sweetish bitter taste are very distinctive. The dull outer surface of the crushed coffee grains is in marked contrast to the polished appearance of the surface of the broken peas or beans, often to be found as adulterants, while fragments of broken cereal grains are readily distinguished from coffee with a low-power magnifier, though perhaps not easily identified by the eye alone.

**Determination of Added Starch.**—Starch is determined in the finely powdered sample as directed on page 292.

**Microscopical Examination of Coffee.**—By far the best means of detecting adulteration is furnished by the microscope. The individual grains of coarsely ground coffee and adulterants, separated by the cold water test or by picking over the mixture, are identified by microscopic examination either after sectioning with a razor or crushing to a powder. In addition, examination is made of a small portion of the sample pulverized in a mortar to a degree fine enough to allow the cover-glass to lie flat on the wetted powder, yet not so fine that it ceases to feel granular when rubbed between the fingers. The writer finds it sufficient to examine this powder in water without further treatment, although Schimper recommends maceration for twenty-four hours with ammonia, in order to render the tissues more transparent, using this reagent also as a mountant.



In general the interior of the coffee tissue or endosperm consists of polygonal cells with highly characteristic, knotty, thickened walls, which are best seen in razor sections, Fig. 76, 2. These cells contain brilliant, colorless, spherical oil drops, and also proteins.

The seed coat is also very characteristic, showing in the powder as occasional delicate silver-like patches, with peculiar, spindle-shaped, thick-sided cells, some of which are loosened from the tissue.

Plates XIV and XV illustrate photomicrographs of pure and adulterated coffee. Fig. 174 shows genuine coffee, with its loose mesh of irregularly polygonal cells, thick-walled, and inclosing oil drops with amorphous material. It is not to be expected that every pulverized sample of genuine coffee, mounted as above, will show in every microscopic field the even, continuous structure that Fig. 174 illustrates, but careful examination will show in nearly every field fragments, and more or less disjointed portions of the polygonal cells, grouped in the form so characteristic of coffee. See Fig. 176.

**Chicory under the Microscope.**—Fig. 77, after Moeller, shows structural features of chicory. The most striking elements are the fine, thick-walled, long-celled, parenchyma of the bark *rp* and *bp* with its delicate tracery, and the vessels or ducts *g* of the wood fibers. These ducts are tubular, resembling jointed cylinders, often with overlapping joints. Less distinct, but very characteristic of certain roots of the composite family, are the narrower branching milk ducts *sch* which do not exist in beets and turnips, which are sometimes substituted for chicory.

Fig. 178, Pl. XV, is a photomicrograph of an adulterated sample of coffee, showing in this particular field chicory alone. It is a mass of confused cellular tissue, traversed by two broad bands of the vessels, with their striking, transverse, dotted markings.

Fig. 177, Pl. XV, shows a sample of coffee adulterated with roasted peas and pea hulls. No genuine coffee appears in this field. The chief masses in the center are characteristic aggregations of the round starch granules of the roasted pea. The rectangular billets, like bunches of matches, are from the outer or palisade layer of the pea.

Fig. 164, Pl. XI, and Fig. 154, Pl. IX, show the close resemblance between the starches of the pea and bean, both of which are commonly used in coffee.

The palisade structures of the hulls of these legumes also bear a close resemblance, but the cells of the next layer in the pea are hour-glass





FIG. 76.—Powdered Coffee under the Microscope.  $\times 125$ . (After Moeller.) 1, seed coat (surface). 2, endosperm parenchyma.

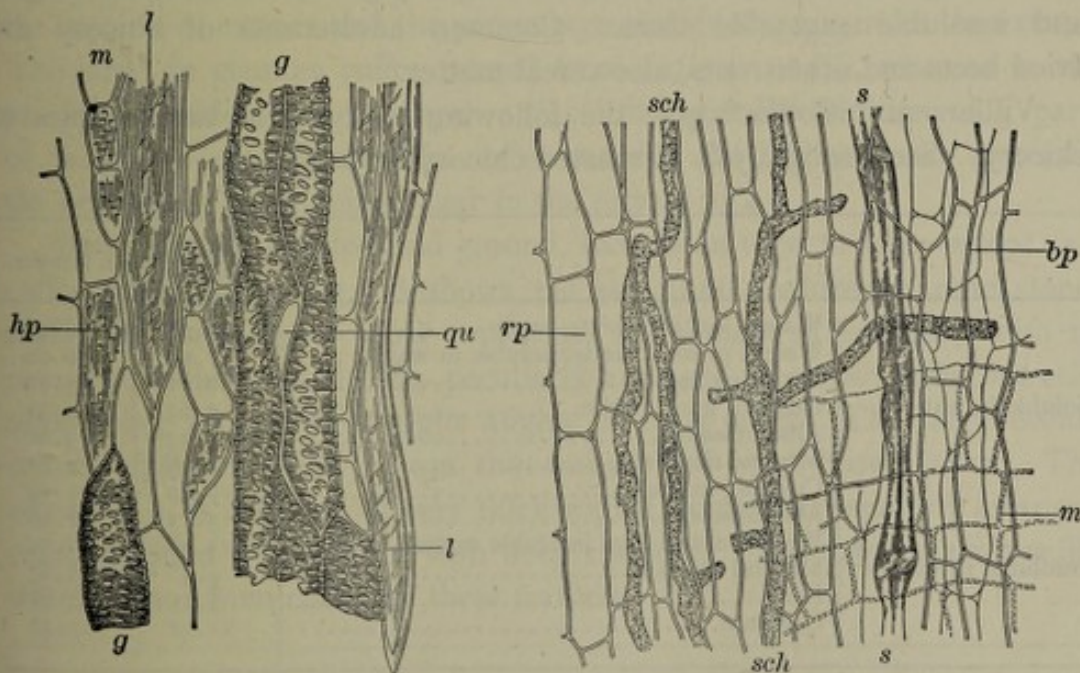


FIG. 77.—Chicory Root in Tangential and Radial Sections.  $\times 160$ . g, reticulated ducts with perforations qu; hp, wood parenchyma; l, wood fibers; rp, bark parenchyma; sch, milk ducts; bp, bast parenchyma; m, medullary rays. (After Moeller.)



shaped, while in the bean they are not remarkable for their shape, but for the single crystal of calcium oxalate contained in each.

The effect of roasting on starches used as adulterants of coffee is to twist and distort the granules, in some cases destroying largely the even structure of the raw starch. Starch granules of wheat, barley, and rye, for example, are almost perfect circular disks in the case of the raw starch, while in roasted products, such as pilot biscuit and stale bread, the granules are twisted and distorted, sometimes almost forming the letter "S."

**Use of Chicory in Coffee.**—Chicory is a perennial herb (*Cichorium intybus*) of the same family (*Compositæ*) as the dandelion. The roasted and pulverized chicory root is so much used in ground coffee to impart a peculiar flavor thereto, that by many it is considered as not strictly an adulterant. The taste imparted to coffee by a small admixture of pure chicory is to some desirable, but if its unrestricted use is sanctioned in this manner, the door would soon be opened to a more unlimited form of adulteration, wherein the chicory might predominate. It is, therefore, best to regard chicory as an adulterant, and to require the package containing a mixture of coffee and chicory, if sold legally, to have plainly printed thereon the percentage of chicory in the mixture.

Chicory, when roasted, consists of gum, partly caramelized sugar, and insoluble vegetable tissue. Common adulterants of chicory are dried beets and other roots, also cereal matter.

Villiers and Collin\* give the following analyses of two samples of chicory. See also analysis of roasted chicory on page 395.

		In Large Granules.	In Powder.
Soluble in water:	Water (loss at 100° to 103°).....	16.28	16.96
	Weight of total matter soluble in water. ....	57.96	56.90
	Reducing sugar.....	26.12	23.79
	Dextrin, gum, inulin.....	9.63	9.31
	Albuminoids.....	3.23	3.66
	Mineral matter.....	2.58	2.55
	Coloring matter.....	16.40	17.59
Insoluble in water:	Albuminoids.....	3.15	2.98
	Weight of the total insoluble matter.....	25.76	26.14
	Mineral matter.....	4.58	5.87
	Fat.....	5.71	3.92
	Cellulose.....	12.32	13.37

\* Falsifications et Alterations des Substances Alimentaires, p. 234.



**Detection and Estimation of Chicory.**—Various chemical tests for detection of chicory in coffee infusions have been suggested, depending on color reactions,\* but these are, as a rule, unreliable. By far the best means for detecting chicory in coffee is furnished by the microscope.

In mixtures containing coffee and chicory only, the approximate amount of the latter can be obtained by McGill's method,† as follows: Weigh a quantity of the pulverized sample, corresponding to 10 grams of the dry substance, into a counterbalanced flask, and add water till the weight of the contents is 110 grams. Bring to boiling in ten to fifteen minutes and continue the boiling for an hour under a reflux condenser. Cool for fifteen minutes, pass through a dry filter, and determine the specific gravity at 15°. McGill found the average specific gravity of a 10% decoction as above carried out to be, in the case of pure coffee 1.00986, and in the case of chicory 1.02821, the difference being 0.01835. The specific gravity of the 10% decoction of the suspected sample at 15° being  $d$ , the per cent of chicory,  $c$ , can be calculated roughly by the formula:

$$c = 100 - \frac{(1.02821 - d)100}{0.01835}.$$

As a means of detecting chicory in the beverage La Wall ‡ determines the amount of extract and the percentage of reducing sugars in the extract. The latter in genuine coffee ranged from 1.92 to 2.64%, whereas in two samples of chicory it was over 25%; consequently addition of 5 parts of chicory to 100 parts of the coffee showing the highest ratio, increased the percentage of reducing sugar in the extract to 4.6.

**Date Stones**, roasted and ground, have been used to some extent as a coffee adulterant. Fig. 78 shows the structural features of date stones under the microscope. *End* represents a fragment of endocarp with its elongated, thick-walled cells, peculiarly arranged as shown, adjacent cells often lying with axes at right angles to each other. The more evenly formed episperm cells, *e*, are thin-walled and of a brown color. The albumen, *a*, is made up of very thick-walled, somewhat regularly arranged cells, indented from within with deep channels. Date stones are readily distinguished from coffee by these features.

\* See Allen's Commercial Org. Analysis, 4 Ed., Vol. VI, pp. 671, 672.

† Trans. Royal Soc. of Canada, 1887.

‡ Am. J. Pharm., 1913, p. 535.



**Hygienic Coffee.**—Various processes have been devised for removing the caffeine from coffee. One of these, patented in Germany, has recently come into extensive use, as the flavor of the beverage is not greatly injured by the treatment. In following out this process the whole beans are first exhausted with water in a vacuum, and the infusion extracted with a suitable solvent for caffeine. The exhausted beans are then impregnated with the decaffeinated infusion and dried in a vacuum. This treatment, as shown by the investigations of Lendrich and Murdfield,\* does not

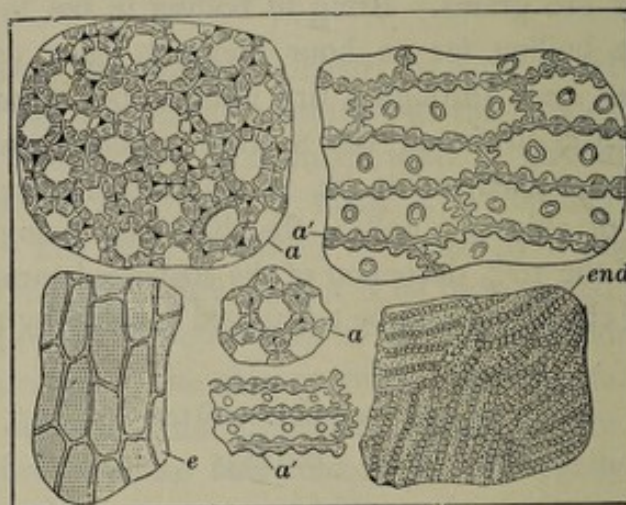


FIG. 78.—Powdered Date Stones under the Microscope. *end*, endocarp; *e*, episperm; *a*, albumen in cross-section; *a'*, albumen in longitudinal section. (After Villiers and Collins.)

completely remove all the caffeine, the quantity remaining being from 0.14 to 0.26%, or about one-sixth of that in the untreated coffee. Further effects of the treatment are a decrease in the water extract and an increase in the fat. The following are the average of analyses, made by these authors, of caffeine-free and untreated coffee:

	Number of Analyses.	Moisture.	Analysis of the Dry Substance.						
			Ash.	Water Soluble Ash.	Alkalinity of Ash (cc. N/1 HCl per 100 grams of Coffee).	Water Extract.	Fat (Petroleum Ether Extract).	Caffeine (Juck-nack and Hilger Method).	Protein (Protein Nitrogen $\times 6.25$ ).
"Caffeine-free Coffee"...	14	% 2.13	% 4.23	% 3.22	47.72	21.30	% 17.13	% 0.22	% 11.83
Untreated coffee. ....	9	% 1.46	% 4.71	% 3.77	56.43	26.17	% 15.73	% 1.19	% 11.75

\* Zeits. Unters. Nahr. Genussm., 15, 1908, p. 705.



Several brands of coffee advertised to be free from tannin and in some cases also from caffeine, have been placed on the market in the United States. Some of these consist merely of ground coffee from which the chaff (which is represented to contain not only the tannin but also most of the caffeine) has been removed by mechanical means. The absurdity of the claims of the manufacturers is shown by the following analyses made in New Hampshire by C. D. Howard.\*

	Water.	Ash.	Fat.	Fiber.	Caffeine.	Caffe-tannic Acid.
Tanninless coffee No. 1. ....	2.70	4.10	13.18	18.46	1.17	10.76
Tanninless coffee No. 2. ....	2.70	4.05	14.12	15.70	1.33	11.04
Tanninless coffee No. 3. ....	2.26	3.61	12.55	22.70	0.87	7.61
Java and Mocha. ....	3.13	4.13	14.10	15.50	1.29	11.17
Coffee chaff. ....	2.60	5.65	9.30	26.50	0.40	5.98

The following analyses made at the Connecticut Station by E. J. Shanley,† corroborate those of Howard:

	Caffeine in the Coffee.	Caffetannic Acid in the Coffee.	Caffetannic Acid in the Chaff.	Per Cent of Chaff in the Coffee.
Tanninless coffee A. ....	1.14	9.89		
Tanninless coffee B. ....	1.11	9.45		
Tanninless coffee C. ....	1.12	9.96		
Java coffee. ....	1.13	9.51	5.46	1.80
Mocha coffee. ....	1.26	9.96	7.55	2.38
Rio coffee. ....	1.13	9.47	6.79	1.77

The Asa process consists in treating the raw coffee with water vapor under a pressure of 4.5 atmospheres and distilling *in vacuo*, thus removing, it is claimed, volatile toxic substances.

Vacuum packed coffee has been extensively advertised as being free from the objectionable qualities of ordinary coffee. Gould's experiments ‡ indicate that the composition of the vacuum-packed coffee is not quite the same as that of ordinary coffee, nor is the gas given off the same, but whether these changes render the coffee more wholesome appears uncertain.

\* U. S. Dept. of Agric., Bur. of Chem., Bul. 105, p. 41.

† Ann. Rep. Conn. Exp. Sta., 1907, p. 141.

‡ Eighth Int. Cong. App. Chem., 26, 1912, p. 389.



**Coffee Substitutes.**—A large number of preparations sold as “coffee substitutes” or “cereal coffee” are now on the market in the United States, most of which are composed, as alleged on the labels, of cereals, ground peas, etc. Some contain roasted wheat, malt or some other cereal alone, others are mixtures of cereals or cereal products and peas, and a few contain chicory. Some of these preparations have labels calling attention to the evil effects of coffee, and one of the latter class, extensively advertised, and purporting to contain nothing but the entire wheat kernel roasted and ground, was found to contain peas, and about 30% of that “most harmful ingredient” coffee itself. Various substitutes are also made from dried fruits such as figs, prunes and bananas.

In addition to the materials named the following have been used in Europe: beans, lupine seeds, cassia seeds, astragalus seeds, *Parkia* seeds, chick peas, soy beans, dried pears, carob bean pods, date stones, ivory nuts, acorns, grape seeds, fruit of the wax palm, cola nuts, false flaxseed, dandelion roots, beets, turnips and carrots.\*

As in the case of coffee the analyst must depend chiefly on the microscope in identifying the constituents of coffee substitutes. Coffee itself should properly be considered in the light of an adulterant.

#### COCOA AND COCOA PRODUCTS.

**Nature of the Cocoa Beans.**—The various chocolate and cocoa preparations are made from the bean of the tree *Theobroma cacao*, of the family of *Byttneriaceæ*. This tree averages 13 feet in height, and its main trunk is from 5 to 8 inches in diameter. It is a native of the American tropics, where it is still most successfully grown for supplying the world's market.

The cocoa beans of commerce are derived chiefly from Ariba, Bahia, Caracas, Cayenne, Ceylon, Guatemala, Haiti, Java, Machala, Maracaibo, St. Domingo, Surinam, and Trinidad. Besides these, the Seychelles and Martinique furnish a small amount.

The plant seeds, or beans, grow in pods, varying in length from 23 to 30 cm., and are from 10 to 15 cm. in diameter. The beans, which are about the size of almonds, are closely packed together in the pod. Their color when fresh is white, but they turn brown on drying.

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\* Winton's Microscopy of Foods, p. 435.



The gathered pods are first cut open, and the seeds removed to undergo the process of "sweating" or fermenting, which is carried out either in boxes or in holes made in the ground. This process requires great care and attention, as upon it depends largely the flavor of the seed. The sweating operation usually takes two days, after which the seeds are dried in the sun till they assume their characteristic warm red color, and in this form are shipped into our markets.

**Manufacture of Chocolate and Cocoa.**—For the production of chocolate and cocoa the beans are cleaned and carefully roasted, during which process the flavor is more carefully developed, and the thin, paper-like shell which surrounds the seed is loosened, and is very readily removed. The roasted seeds are crushed, and the shells, which are separated by winnowing, form a low-priced product, from which an infusion may be made, having a taste and flavor much resembling chocolate.

The crushed fragments of the kernel or seed proper are called *cocoa nibs*, and for the preparation of chocolate they are finely ground into a paste and run into molds, either directly, or after being mixed with sugar and vanilla extract or spices, according to whether plain or sweet chocolate is the end product.

For making cocoa, however, a portion of the oil or fat known as the cocoa butter is first removed, by subjecting the ground seed fragments to hydraulic pressure, usually between heated plates, after which the pressed mass is reduced to a very fine powder, either directly, or by treatment with ammonia or alkalies, to render the product more "soluble." It is held that the large amount of fat contained in the cocoa seeds (varying from 40 to 54%) is difficult of digestion to many, such as invalids and children, and hence the desirability of removing part of the fat.

**Composition of Cocoa Products.**—The chief constituents of the raw cocoa nib are fat, starch, pentosans, proteins, theobromine, caffeine, tannin, and mineral matter. Minor constituents are oxalic acid (combined), acetic and tartaric acids. During roasting there is reason to believe a volatile substance is developed much in the nature of an essential oil, which gives to the product its peculiar flavor, and is somewhat analogous to the caffeine of coffee.

Tannin, the astringent principle of cocoa, exists as such in the raw bean, but rapidly becomes oxidized to form *cocoa red*, to which the color of cocoa is due.

Weigmann gives the following results of analyses of cocoa nibs and shells:



## COMPOSITION OF COCOA NIBS.

Commercial Varieties.	Moisture.	Nitrogenous Substances, including Theobromine.	Theobromine.	Fat.	Starch.	Other Carbohydrates.	Cellulose.	Ash.	Sand.
Caracas. ....	7.77	14.13	1.48	45.54	19.40	.....	6.19	4.91	2.06
Trinidad. ....	7.87	14.06	1.31	44.62	25.30	.....	4.55	3.48	0.10
Surinam. ....	7.53	13.69	1.66	44.74	26.45	.....	4.30	3.16	0.13
Port au Prince. ....	7.77	14.56	.....	46.35	5.97	15.53	5.19	4.15	1.48
Machata. ....	8.17	14.06	.....	45.93	5.69	17.50	4.36	4.09	0.22
Puerto Cabello. ....	8.08	13.50	1.51	46.61	22.9	.....	4.43	4.28	0.18
Ariba. ....	8.27	15.37	.....	45.15	5.83	16.96	4.48	3.88	0.14

## COMPOSITION OF COCOA SHELLS.

Commercial Varieties.	Moisture.	Nitrogenous Substances.	Theobromine.	Fat.	Nitrogen-free Extract.	Cellulose.	Ash.	Sand.	Total Nitrogen.
Caracas. ....	12.49	13.18	0.58	2.38	40.30	16.33	9.06	6.26	2.11
Trinidad. ....	14.64	14.62	0.74	3.45	44.89	15.79	6.19	0.42	2.34
Surinam. ....	13.93	16.25	0.78	2.54	42.47	17.04	6.63	0.85	2.60
Puerto Cabello. ....	14.89	16.18	0.75	2.01	43.32	15.25	8.08	0.27	2.59

On page 409 are the summarized results of the analyses of seventeen varieties of cocoa seeds and shells, made by Winton, Silverman, and Bailey.\*

According to Bell † the ash of cocoa nibs has the following composition:

	Per Cent.
Sodium chloride. ....	0.57
Soda. ....	0.57
Potash. ....	27.64
Magnesia. ....	19.81
Lime. ....	4.53
Alumina. ....	0.08
Ferric oxide. ....	0.15
Carbonic acid. ....	2.92
Sulphuric acid. ....	4.53
Phosphoric acid. ....	39.20
	100.00

\* Ann. Rep. Conn. Agric. Exp. Sta., 1902, p. 270.

† Analysis and Adulteration of Foods.



	Roasted Cocoa Nibs.					
	Air-dry Material.			Water- and Fat-free Material.		
	Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.
Water .....	3.18	2.29	2.72			
Total ash .....	4.15	2.61	3.32	8.81	5.76	7.04
Water-soluble ash .....	1.86	0.73	1.16	3.96	1.60	2.46
Ash insoluble in acid. ....	0.07	0.00	0.02	0.14	0.00	0.05
Alkalinity of ash.....	3.35	1.50	2.51	7.12	3.29	5.32
Theobromine. ....	1.32	0.82	1.04	2.92	1.66	2.21
Caffeine. ....	0.73	0.14	0.40	1.55	0.31	0.86
Other nitrogenous substances .....	13.06	11.00	12.12	28.05	23.37	25.69
Crude fiber. ....	3.20	2.21	2.64	6.56	4.70	5.61
Crude starch (acid conversion). ....	12.37	9.30	11.16	25.68	19.80	23.66
Pure starch (diastase conversion). ....	8.99	6.49	8.07	18.61	13.82	17.10
Other nitrogen-free substances .....	21.07	17.69	19.57	44.08	38.78	41.49
Fat.....	52.25	48.11	50.12			
Total nitrogen .....	2.54	2.20	2.38	5.41	4.74	5.05
Constants of fat (ether extract):						
Melting-point, degrees C. ....	35.0	32.3	33.3			
Zeiss refractometer reading at 40° C....	48.00	46.00	47.23			
Refractive index at 40° C.....	1.4579	1.4565	1.4573			
Iodine number. ....	37.89	33.74	34.97			
Per cent of nibs in whole bean. ....	92.90	86.12	88.46			
" " "shells" " " " .....	13.88	8.83	11.54			

	Roasted Cocoa Shells.					
	Air-dry Material.			Water- and Fat-free Material.		
	Maximum.	Minimum.	Mean.	Maximum.	Minimum.	Mean.
Water .....	6.57	3.71	4.87			
Total ash .....	20.72	7.14	10.48	21.97	5.63	11.33
Water-soluble ash .....	5.67	2.02	3.67	6.11	2.16	3.97
Ash insoluble in acid. ....	11.18	0.05	2.51	11.86	0.05	2.70
Alkalinity of ash.....	5.92	5.02	5.52	6.47	5.32	5.97
Theobromine. ....	0.90	0.20	0.49	0.97	0.22	0.52
Caffeine. ....	0.28	0.04	0.16	0.31	0.04	0.17
Other nitrogenous substances .....	18.06	10.69	14.54	19.40	11.34	15.70
Crude fiber. ....	19.21	12.93	16.63	20.72	13.71	18.01
Crude starch (acid conversion). ....	13.89	9.87	11.62	15.42	10.47	12.59
Pure starch (diastase conversion). ....	5.16	3.36	4.14	5.59	3.65	4.47
Other nitrogen-free substances. ....	51.86	43.71	46.40	55.84	47.04	50.08
Fat.....	5.23	1.66	2.77			
Total nitrogen .....	3.17	1.74	2.34	3.41	1.87	2.54



**Theobromine** ( $C_7H_8N_4O_2$ ), the chief alkaloid of cocoa, when pure, forms a white, crystalline powder, having a bitter taste. It is slightly soluble in water and alcohol, very slightly soluble in ether, insoluble in petroleum ether, but readily soluble in chloroform. It sublimes at  $290^\circ$  to  $295^\circ$  C. It is a weak base, and much resembles caffeine. A small amount of caffeine has also been found in cocoa, but in most analyses is reckoned in with the theobromine.

**The Nitrogenous Substances of Cocoa**, aside from the alkaloids, have been little studied. Stutzer has, however, separated them roughly as in the following analyses of four samples, of which A was manufactured without chemicals, B with potash, and C and D with ammonia:

	A.	B.	C.	D.
Total nitrogen .....	3.68	3.30	3.95	3.57
Theobromine .....	1.92	1.73	1.98	1.80
Ammonia .....	0.06	0.03	0.46	0.33
Amido compounds .....	1.43	1.25	0.31	1.31
Digestible albumin .....	10.25	7.68	10.50	7.81
Indigestible nitrogenous substances .....	7.18	9.19	7.68	8.00
Containing nitrogen .....	1.15	1.47	1.23	1.28
Proportion of total nitrogen indigestible .....	31.2	44.5	31.2	35.8

**Pentosans.**—Several authors have called attention to the value of these substances as a means of detecting added shells in cocoa products.

Lührig and Segin\* found in cocoa nibs from 2.51 to 4.58% of pentosans calculated to the dry, fat-free substance, and in the shells from 7.59 to 11.23% calculated to the dry substance.

**Milk Chocolate**, a product of comparatively recent introduction, consists of a mixture of chocolate, sugar, milk powder, and cocoa butter. It is especially prized by travelers and others who desire a concentrated, and at the same time palatable food.

The following analyses by Dubois† show the composition of three of the leading brands on the market, and also illustrate the accuracy of Dubois' method of determining sucrose and lactose given on page 415.

**Various Compounds** of chocolate or cocoa with other materials have been placed on the market. Zipperer‡ gives formulas or analyses of seventy-four such preparations, containing one or more of the following ingredients: oatmeal, barley meal, malt, malt extract, wheat flour, potato

\* Zeits. Unters. Nahr. Genussm., 12, 1906, p. 161.

† Jour. Am. Chem. Soc., 29, 1907, p. 556.

‡ The Manufacture of Chocolate and Cacao Preparations, 2d ed., 1902.



	Polarization.				Su- crose, Per Cent.	Lac- tose, Per Cent.	Reich- ert- Meissl Num- ber of Fat.	Approx. Per Cent Butter Fat in Total Fat.
	Direct.	After Inver- sion.	Temp. °C.	At 86°.				
Commercial milk chocolate:								
A.....	+ 21.00	- 2.00	24	+ 1.36	40.90	8.24	5.3	22.1
B.....	+ 23.22	- 2.22	23	+ 1.50	45.73	9.12	5.5	22.9
C.....	+ 23.88	- 2.20	23	+ 1.36	46.78	8.24	5.8	24.2
Milk chocolate made in the laboratory:								
D { Found. ....	+ 19.00	- 1.50	20	+ 1.40	35.99	8.52	4.83	20.1
Calculated. ....					35.82	8.82		
E { Found. ....	+ 19.70	- 2.20	21	+ 0.99	39.84	6.03	3.48	14.5
Calculated. ....					39.80	5.88		

flour, rice, peas, peanuts, acorns, cola nuts, sago, arrowroot, Iceland moss, gum Arabic, salep, dried meat, meat extract, peptones, milk powder, plasmon (a preparation of casein), eggs, saccharin, vanilla, spices, and inorganic salts. Certain medicinal preparations also contain cocoa products.

**Cocoa Butter.**—See Chapter XIII.

#### METHODS OF ANALYSIS.

**Preparation of the Sample.**—Cocoa is usually in a fine powder, and needs merely to be put through a sieve, to break up lumps, and mixed. Chocolate should be grated or shaved so as to permit mixing. It cannot be ground, as the heat of grinding reduces it to a paste.

**Determination of Moisture.**—Dry 2 grams of the material to constant weight at 100° C. in a current of dry hydrogen. Somewhat lower results are obtained by drying in a dish in air.

**Determination of Ash.**—*Total, Water-soluble, and Acid-soluble.*—Proceed as described under tea (page 382).

**Alkalinity (Ewell Method \*).**—To the ash of 2 grams of the sample add 100 cc. of water, an excess of N/10 sulphuric acid, and boil until carbon dioxide is removed. Titrate the excess of acid with N/10 alkali, using phenolphthalein as indicator. Calculate the number of cubic centimeters of N/10 acid required to neutralize the ash from 1 gram of the sample.

This method was used by Winton, Silverman, and Bailey, in the

\* U. S. Dept. of Agric., Div. of Chem., Bul. 13, 1892, p. 956.



analyses as summarized on page 409. It is essentially the same as the French official method\* and differentiates cocoa and chocolate from cocoa shells more sharply than the A. O. A. C. method employing methyl orange as indicator.

**Determination of Protein.**—Determine total nitrogen by the Kjeldahl or Gunning method. From the percentage of total nitrogen subtract the nitrogen of the theobromine and caffeine, obtained by multiplying the percentages found by 0.311 and 0.289 respectively, and multiply the remainder by 6.25.

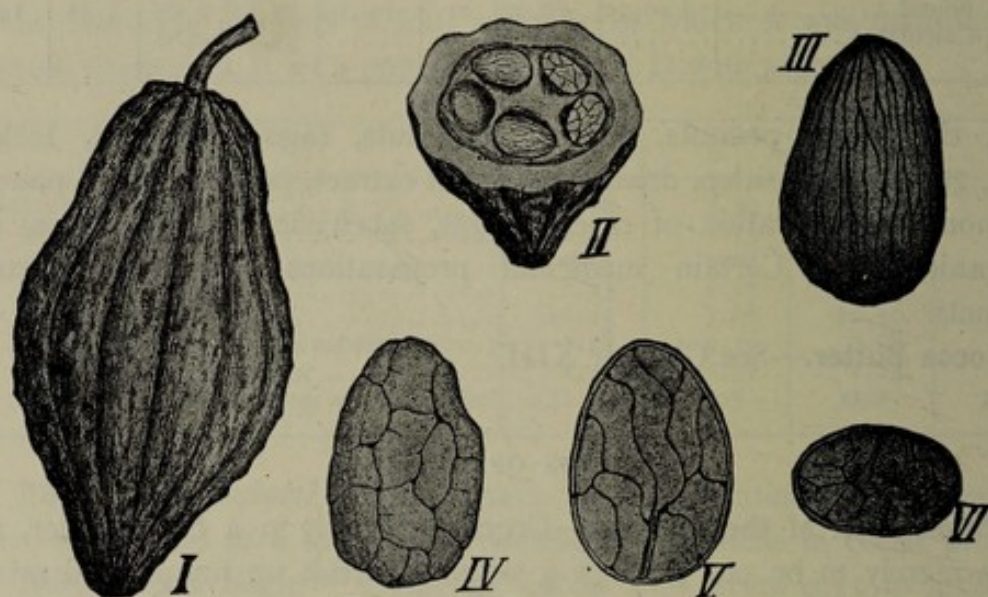


FIG. 79.—Cocoa. *I* entire fruit,  $\times \frac{1}{4}$ ; *II* fruit in cross-section; *III* seed (cocoa bean) natural size; *IV* seed deprived of seed coat; *V* seed in longitudinal section, showing radicle (germ); *VI* seed in cross-section. (WINTON.)

**Determination of Casein.**—*Hammarsten Method Modified by Baier and Neumann.*†—Extract 20 grams of the sample with ether, dry at room temperature, and weigh. Rub up 10 grams of the dry fat-free material with a small quantity of 1% sodium oxalate solution, wash into a 250-cc. graduated flask with about 200 cc. of the oxalate solution, heat to boiling, and make up nearly to the mark with boiling oxalate solution. Allow to stand 18 hours with occasional shaking, fill to the mark with cold oxalate solution, and filter through a dry paper. Pipette off 100 cc. of the filtrate, add 5 cc. of 5% uranium acetate solution and 30% acetic acid, drop by drop with constant stirring until a precipitate of casein begins to form,

\* Ann. fals., 4, p. 417.

† Zeits. Unters. Nahr. Genussm., 18, 1909, p. 13.



then add 5 additional drops of the acid. Centrifuge, filter, wash free from oxalate with a solution containing in 100 cc. 5 grams of uranium acetate and 3 cc. of 30% acetic acid, and determine nitrogen in the filter and precipitate by the Kjeldahl or Gunning method. Calculate casein using the factor 6.37.

Bolton and Revis \* and Lythgoe † have found this method satisfactory.

**Determination of Theobromine and Caffeine** (*Decker-Kunze Method*).‡

—This combination of the Decker and the Kunze methods was first employed by Winton, Silverman, and Bailey, and afterwards adopted by the Assn. of Official Agricultural Chemists. Boil 10 grams of the powdered material and 5 grams of calcined magnesia for 30 minutes with 300 cc. of water. Filter by the aid of suction on a Büchner funnel, using a round disk of filter paper. Transfer the material and paper to the same flask used for the first boiling, add 150 cc. of water, and boil 15 minutes. Filter as before, and repeat the operation of boiling with 150 cc. of water and filtering. Wash once or twice with hot water. Evaporate the united filtrates (with quartz sand if sugar be present) to complete dryness in a thin glass dish of about 300 cc. capacity.§

Grind to a coarse powder in a mortar provided with a suitable cover to prevent loss by flying. Transfer to the inner tube of a continuous fat extractor, and dry thoroughly in a water oven. Extract with chloroform for 8 hours, or until the theobromine and caffeine are completely removed, into a weighed flask. It is important that the material be thoroughly dry, that an extractor be used that permits of a hot extraction, and that a considerable volume of chloroform passes through the material. Distil off the chloroform, and dry at 100° C. to constant weight.

If the material be pure chocolate or cocoa, the extract thus obtained is practically pure theobromine and caffeine, but if the material is cocoa shells or a cocoa product mixed with a large amount of shells, the extract may be brown in color, due to the presence of considerable amounts of impurities.

In either case, separate the caffeine by treating the extract in the flask at the room temperature for some hours with 50 cc. of pure benzol.

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\* Fatty Foods, Phila., 1913, p. 317.

† Jour. Assn. Off. Agric. Chem., 1, 1915, p. 200.

‡ Schweiz. Wchschr. Phar., 40, 1902, pp. 527, 541, 553; Conn. Agric. Exp. Sta. Rep., 1902, p. 274.

§ A "Hoffmeister Schälchen" may be used, or dishes may be made from broken flasks by making a scratch with a diamond and leading a crack from this scratch about the flask by means of a glowing springcoal.



Filter through a small paper into a tared dish, evaporate to dryness, and dry to constant weight at  $100^{\circ}$  C., thus obtaining the amount of caffeine.

Determine theobromine by Kunze's \* method, as follows:

Add to the residue and paper 150 cc. of water, enough ammonia water to make the liquid slightly alkaline, and an excess of decinormal silver nitrate solution. Boil to half the original volume, add 75 cc. of water, and repeat the boiling. The solution should be perfectly neutral. If it contains the slightest amount of free ammonia, add water and boil until it is completely removed.

Filter from the insoluble silver theobromine compound, and wash with hot water. In the filtrate determine the excess of silver nitrate by Volhard's † method as follows:

Add 5 cc. of cold saturated solution of ferric ammonium sulphate (ferric-ammonium alum), and enough boiled nitric acid to bleach the liquid. Titrate with decinormal ammonium sulphocyanide solution until a permanent red color appears.

One cc. of decinormal  $\text{AgNO}_3$  solution is equivalent to 0.01802 gram of theobromine. If the mixed alkaloids were colorless, the theobromine obtained by subtracting the weight of caffeine from the weight of the mixed alkaloids will usually agree closely with that obtained by silver titration.

**Determination of Crude Fiber.**—Proceed as in the analysis of cereal products, using the residue from the ether extraction.

**Determination of Reducing Matters by Acid Conversion (Crude Starch).**—*Winton, Silverman, and Bailey* ‡ proceed as follows: Weigh 4 grams of the material into a small Wedgwood mortar, add 25 cc. of ether, and grind with a pestle. After the coarser material has settled out, decant off the ether with the fine suspended matter on an 11-cm. paper. Repeat this treatment until no more coarse material remains. After the ether has evaporated, transfer the fat-free residue from the filter to the mortar by means of a jet of cold water, and rub to an even paste. Filter the liquid on the paper previously employed. Repeat the process of transferring from the filter to the mortar, grinding, and filtering, until all sugar is removed. In the case of sweetened cocoa products, at least 500 cc. of water should be used.

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\* *Zeits. anal. Chem.*, 33, 1894, p. 1.

† *Ibid.*, 13, 1874, p. 171.

‡ *Conn. Agric. Exp. Sta., Rep.*, 1902, p. 275.



Transfer the residue to a 500-cc. flask by means of 200 cc. of water, and convert the starch into dextrose by Sachsse's method (page 292).

Cool the acid solution, nearly neutralize with sodium hydroxide solution, add 5 cc. of lead sub-acetate solution (page 610), make up to 250 cc. and filter through a dry filter. To 100 cc. of the filtrate, add 1 cc. of 60% sulphuric acid, shake thoroughly, allow to settle, and filter through a dry filter.

Determine reducing matters by Allihn's method (page 632).

*Dubois*,\* instead of treating with ether as above described, shakes 4 grams of the unsweetened product or 8 grams of the sweetened with 100 cc. of gasoline, and whirls in a centrifuge to separate from the insoluble matter. After decanting off the gasoline layer, sweetened products are treated in like manner with two portions of 100 cc. of water to remove the bulk of the sugar, and finally washed on the paper.

**Determination of Pure Starch.**—*Diastase Method.*—Remove the fat and sugar from 4 grams of the material by treatment with ether and water, as described in the preceding section, and determine starch in the residue by the diastase method (page 292).

*Revis and Burnett*† employ a method with the following features: (1) taká-diastase is substituted for malt extract, (2) the solution containing dextrose and maltose into which the starch has been converted is cleared with acid mercuric nitrate, the excess being removed by sodium phosphate solution, (3) the copper-reducing power and polarization of the solution are determined without acid conversion, and (4) the dextrose and maltose and from these the starch are calculated by appropriate formulæ. The authors obtain by this method lower results on cocoa shells than by the diastase method, which is consistent with the absence of starch as shown by microscopic examination.

**Determination of Pentosans.**—See page 294.

**Determination of Sucrose and Lactose.**—*Dubois Method.*‡—Place 26 grams of the material in an 8-ounce nursing bottle, add about 100 cc. petroleum ether and shake for 5 minutes. Whirl in a centrifuge until the solvent is clear, draw off the same by suction and repeat the treatment with petroleum ether. Keep the bottle containing the defatted residue in a warm place until the petroleum ether is practically expelled. Add 100 cc. water and shake until all the chocolate is loosened from the sides

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 122, p. 214.

† Analyst, 40, 1915, p. 429.

‡ U. S. Dept. of Agric., Bur. of Chem., Circ. 66, p. 15.



and bottom of the bottle and continue the shaking for 3 minutes longer. Add 10 cc. of lead subacetate solution (page 610), mix thoroughly and filter through a folded filter. Make the direct polariscopic reading (*a*) in a 200-mm. tube, then precipitate the excess of lead by dry potassium oxalate. Invert by one of the methods given on page 611, polarize, and multiply the invert reading by 2 to correct for dilution (*b*). Calculate the approximate percentages of sucrose (*S*) and lactose (*L*) by the following formulas:

$$S = \frac{(a-b) \times 110}{142.66 - \frac{t}{2}} \quad L = \frac{(a \times 1.10) - S}{0.79}.$$

From the sum of *S* and *L* calculate the approximate number of grams of total sugar *G* present in the 26 grams of sample taken and determine the factor *X* thus:

$$X = 110 + (G \times 0.62),$$

in which 0.62 is the volume in cc. displaced by 1 gram of sugar in water solution. Applying this correction,

$$\text{True per cent sucrose} = \frac{SX}{110} \quad \text{True per cent lactose} = \frac{LX}{110}.$$

The following method of solution may be substituted for that given above:

Transfer 26 grams to a flask, add 100 cc. water, cork, and heat in steam-bath for twenty minutes, releasing the pressure occasionally during the first five minutes. Twice during the twenty minutes shake thoroughly so as to emulsify completely. Finally cool to room temperature, add 10 cc. lead subacetate solution, mix, and filter.

**Determination of Cocoa-Red.**—*Blyth Method.*\*—Make 2-3 grams of the fat-free sample into a paste with hydrochloric acid, add sufficient silver oxide to fix the hydrochloric acid, and extract in a Soxhlet extractor with 100 cc. of absolute alcohol. Cool, filter the alcoholic liquid, precipitate with an alcoholic solution of lead acetate, collect the purple-black precipitate on a filter, and wash well with boiling water. Transfer the precipitate to a small flask, add 70% alcohol, and decompose the lead

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\* Blyth, *Foods: Their Composition and Analysis*, London, 1909, p. 368.



salt with hydrogen sulphide. Drive off the excess of hydrogen sulphide by heating, filter, evaporate the filtrate, dry, and weigh. Purify by converting again into the lead salt and decomposing with hydrogen sulphide.

*Zipperer's Method* is more elaborate and requires correction for resin and phlobophene formed by the decomposition of the cocoa-red.

*Ulrich Method.\**—Boil for 3 hours 1 gram of the dry, fat-free, finely powdered sample with 120 cc. acetic acid (50–51%) in a 300-cc. Erlenmeyer flask having a reflux condenser. Cool, make up to 150 cc. with water, shake, and allow to stand 12 hours. Filter through dry paper and boil 135 cc. of the filtrate with 5 cc. of concentrated hydrochloric acid and 20 cc. of a 20% ferrous chloride solution under a reflux condenser for 10 minutes. Cool quickly, pour into a beaker, allow to stand 6 hours, and filter through a weighed paper, washing with hot water until free from iron, dry 6 hours at 105° C., and weigh.

#### ADULTERATION OF COCOA PRODUCTS AND STANDARDS OF PURITY.

The following are the U. S. standards:† *Standard chocolate* should contain not more than 3% of ash insoluble in water, 3.5% of crude fiber, and 9% of starch, nor less than 45% of cocoa fat.

*Standard sweet chocolate and standard chocolate coating* are plain chocolate mixed with sugar (sucrose), with or without the addition of cocoa butter, spices, or other flavoring material, containing in the sugar- and fat-free residue no higher percentage of either ash, fiber, or starch than is found in the sugar- and fat-free residue of plain chocolate.

*Standard cocoa* should contain percentages of ash, crude fiber, and starch corresponding to those of plain chocolate, after correcting for fat removed.

*Standard sweet cocoa* is cocoa mixed with sugar (sucrose) containing not more than 60% of sugar, and in the sugar- and fat-free residue no higher percentage of either ash, crude fiber, or starch than is found in the sugar- and fat-free residue of plain chocolate.

The removal of fat, or the addition of sugar beyond the above prescribed limits, or the addition of foreign fats, foreign starches, or other foreign substances, constitutes adulteration, unless plainly stated on the label.

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\* Inaug. Dis. Detmold, 1911; Arch. Pharm., 249, p. 524; Jour. Assn. Off. Agric. Chem., 1, 1916, p. 550.

† U. S. Dept. of Agric., Off. of Sec., Circ. 19.



The most common adulterants of cocoa are sugar and various starches, especially those of wheat, corn, and arrowroot. Starch is sometimes added for the alleged purpose of diluting the cocoa fat, instead of removing the latter by pressure, thus, it is claimed, rendering the cocoa more digestible and more nutritious. Unless its presence is announced on the label of the package, starch should be considered as an adulterant. Cocoa shells are also commonly employed as a substitute for, or an adulterant of, cocoa. Other foreign substances found in cocoa are sand and ground wood fiber of various kinds. Iron oxide is occasionally used as a coloring matter, especially in cheap varieties.

Such adulterants as the starches and cocoa shells are best detected by the microscope. The presence of any considerable admixture of sugar is made apparent by the taste. Mineral adulterants are sought for in the ash.

**Addition of Alkali.**—The amount of water soluble matter in cocoa is very small (about 20 to 25%), and in preparing the beverage, the desideratum aimed at is to produce as perfect an emulsion as possible. The legitimate means of accomplishing this is by pulverizing the cocoa very fine, so that particles remain in even suspension and form a smooth paste. Another means sometimes resorted to for producing a so-called "soluble cocoa" is to add alkali in its manufacture, the effect being to act upon a part of the fat, and produce a more perfect emulsion with less separation of oil particles. Such treatment with alkali is regarded with disfavor, even if not considered as a form of adulteration. Cocoa thus treated is generally darker in color than the pure article.

The use of alkali is usually rendered apparent by the abnormally high ash, and by the increased alkalinity of the ash, the latter constant being expressed in terms of the number of cubic centimeters of decinormal acid necessary to neutralize the ash of 1 gram of the sample. In pure, untreated cocoa, the ash rarely exceeds 5.5%, and the alkalinity of the ash is generally not more than 3.75. In cocoa treated with alkali, the ash sometimes reaches 8.5%, with the alkalinity running as high as 6 or even 8.

**Microscopical Structure of Cocoa.**—Fig. 80 shows elements of the powdered cocoa bean, both of the shell and of the kernel. The powder of the latter should constitute pure cocoa, with occasional fragments only of the shell. The irregular lobes constituting the kernel are each inclosed in a membrane made up of angular cells, filled with granular matter. (4), (5), and (6) show elements of the powdered cotyledons,



or seed kernels. The polygonal tissue of the cotyledon is shown in cross-section at (4). In the powder one finds also dark granular matter, bits of debris, and fragments, with masses of yellow, reddish-brown, and sometimes violet coloring matter, together with numerous starch granules and aleurone grains.

The starch granules are nearly circular, with rather indistinct central nuclei, and range in size from 0.0024 to 0.0127 mm., averaging about 0.007 mm. They are more often found in single detached grains, but sometimes in groups of two or three. Occasional spiral ducts, *sp*, are seen, but these are not abundant in the pure cocoa.

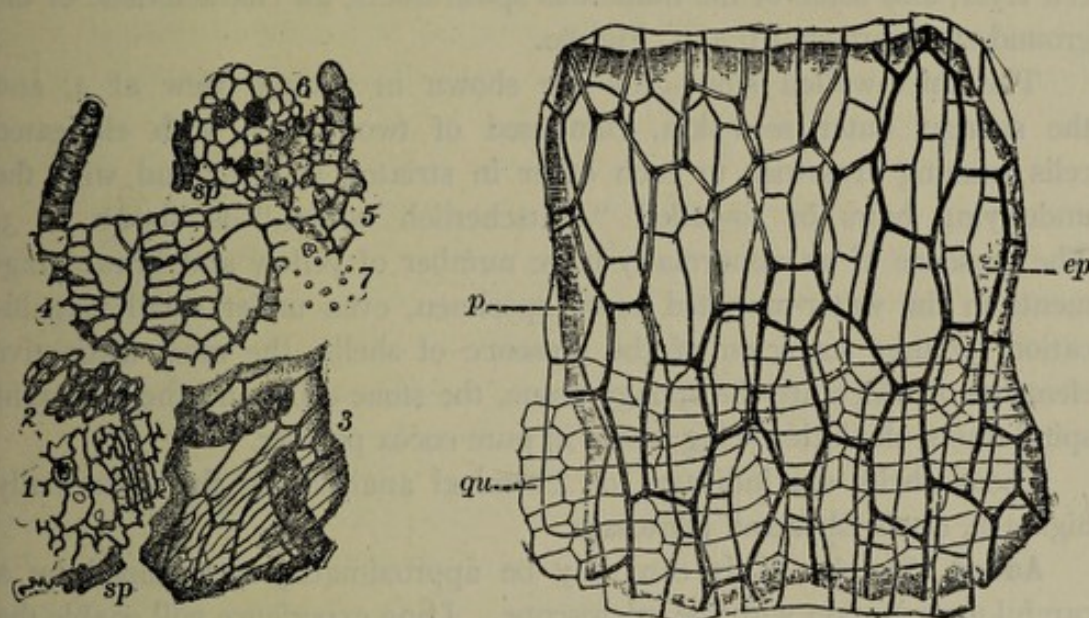


FIG. 80.—Cocoa under the Microscope.

A. Powdered Cocoa under the Microscope.  $\times 125$ . (After Moeller.) 1, cross-section through shell parenchyma; 2, thick-walled cells; 3, epidermis of shell (surface section); 4, cross-section of cotyledon tissue; 5, 6, cotyledon parenchyma; 7, starch.

B. Cocoa Shell in Surface Section.  $\times 160$ . *ep*, epicarp; *p*, parenchyma of the fruit; *qu*, layer of transverse cells. (After Moeller.)

The masses of color pigment are shown up with striking clearness, according to Schimper, by applying a drop of sulphuric acid to the edge of the cover-glass and allowing it to penetrate the tissue. The bits of coloring matter are for a short time colored a brilliant red, which, however, soon fades. Ferric chloride colors them indigo blue.

Schimper recommends mounting the powder in a drop of chloral hydrate, which soon renders most of the tissues transparent. It is sometimes necessary to allow the chloral to act on the powder in a closed



vessel for twenty-four hours, before all the elements of pure cocoa are rendered transparent. If after that time opaque masses are still found, these are due to foreign material.

Ammonia may be used instead of chloral with even better results, but this reagent requires longer treatment, soaking for several days or a week being sometimes necessary.

Fig. 185, Pl. XVII, shows the microscopical appearance of genuine powdered cocoa with its variously sized starch grains and the débris of the ground cotyledons. Fig. 186 shows cocoa adulterated with arrowroot.

**Cocoa Shells.**—A cross-section of the shell parenchyma and the stone-cell layer, also some of the numerous spiral ducts, all characteristic of the ground shell, are shown at 1, Fig. 80.

The thick-walled stone-cells are shown in surface view at 2, and the spongy, outer seed-skin, composed of two layers, with elongated cells running crosswise to each other in striated fashion, and with the underlying hairs or so-called "Mitscherlich bodies," is shown at 3. The presence of an abnormally large number of yellow and brown fragments in the water-mounted cocoa specimen, even under small magnification, arouses suspicion of the presence of shells, the most distinctive elements of which are the spongy tissue, the stone-cells, and the abundant spiral ducts, the latter being scarce in pure cocoa powder.

Cocoa shells are indicated on chemical analysis by the abnormally high ash, crude fiber and pentosans.

**Added Starch.**—This can only be approximately determined by a careful examination with the microscope. Long experience will enable the analyst to familiarize himself with the appearance and abundance of starch grains of various kinds in a series of fields, so that he can roughly estimate the amount of each starch present in the mixture, by careful comparison with mixtures of known percentage composition.

If the amount of starchy adulterant is considerable, evidence may be secured by determinations of starch by the diastase method and reducing matters by acid conversion.

**Added Sugar.**—Any appreciable amount of added cane sugar is shown by the sweet taste. The amount of cane sugar may be determined by means of the polariscope, as described on page 415.

An abnormally low ash is indicative of the addition of starch or sugar or both.

**Foreign Fat.**—Certain manufacturers have found it profitable to remove a portion of the cocoa butter from chocolate and substitute for



it a cheaper fat, such as cocoanut oil, tallow or even paraffine. Such adulteration is detected by determination of the physical and chemical constants of the fat obtained by extraction with ether.

**Dyes and Pigments**, such as Bismarck brown and Venetian red, have been employed to hide the presence of diluents. They are detected by dyeing tests, and by examination of the ash.



## CHAPTER XII.

### SPICES.

THESE aromatic vegetable substances are classed as condiments, and depend for their use on the pungency which they possess in giving flavor or relish to food. As such seasoning or zest-giving substances, they are of considerable importance dietetically, but from the fact that they are used in comparatively insignificant amount, the determination of their chemical composition or actual value as nutrients *per se* is of little importance to the food economist.

**Adulteration.**—Formerly ground spices were subject to the grossest forms of adulteration, all kinds of cheap material being reduced to powders for the purpose, the aim being to match the genuine spice in color and general appearance. The foreign materials were for the most part detected by microscopic examination although chemical analysis furnished valuable corroboratory evidence. At present such frauds have for the most part disappeared in the United States, and the analyst is called on chiefly to examine spices for an excess of shells or fibrous material, dirt, and similar impurities, the presence of which indicates a low grade rather than intentional adulteration, or for exhausted spices.

In a few instances the substitution of the products of inferior species or varieties belonging to the same family as those which yield the standard spice is still practiced. Examples are Bombay and Macassar mace substituted for true or Banda mace, the seed of charlock substituted for the seeds of the more valuable species of the mustard family, and Spanish red pepper or pimiento substituted for the Hungarian product known as paprika.

Microscopic and chemical examination is still necessary partly to detect the occasional fraud of the old type, which still may be practiced, and partly to distinguish varieties and grades and detect an excess of natural impurities.

**General Methods of Proximate Analysis.**—The following methods common to all the spices are specially designed to determine quality or grade, or to detect adulteration.

Methods of analysis peculiar to individual spices will be treated



under the discussion of the spice in question. For these determinations the spices should be powdered fine enough to pass through a 60-mesh sieve.

**Determination of Moisture.**—*Richardson's Method*.<sup>\*</sup>—Two grams of the sample are weighed in a tared platinum dish and dried in an air-oven at 110° to a constant weight, which generally requires about twelve hours. The loss in weight includes the moisture and the volatile oil. The latter is determined from the ether extract, as described on page 424, and deducted from the total loss to obtain the moisture.

McGill † determines the moisture by exposure of a weighed portion of the sample *in vacuo* over perfectly colorless sulphuric acid. The spice gives up its moisture before the volatile oil comes off, and any appreciable amount of the volatile oil, when absorbed by the acid, causes the latter to be discolored, so that by carefully observing the beginning of the discoloration, and removing the sample, the loss due to moisture may be obtained by weighing at the proper stage. The abstraction of the moisture in this manner requires about twenty-four hours.

**Determination of Ash.**—Two grams of the spice are burned in a platinum dish heated to faint redness on a piece of asbestos paper by means of a Bunsen burner. The burning is best finished in a muffle furnace. If the ash contains an appreciable amount of carbon, it is exhausted on a filter with hot water, and the filter with the residue is burnt in the dish previously used. After adding the aqueous extract and a few drops of ammonium carbonate solution, the whole is evaporated to dryness and ignited at a faint red heat.

*The Water-soluble Ash* ‡ is found by boiling the total ash as above obtained with 50 cc. of water, and filtering on a tared Gooch crucible, the insoluble residue being washed with hot water, dried, ignited, and weighed. The insoluble ash, subtracted from the total, leaves the water-soluble ash.

*Sand.*—This is assumed to be the percentage of ash insoluble in hydrochloric acid. The ash from 2 grams of the substance, obtained as above described, is boiled with 25 cc. of 10% hydrochloric acid (specific gravity 1.050) for five minutes, the insoluble residue is collected on a tared Gooch crucible, thoroughly washed with hot water, and finally dried and weighed.

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<sup>\*</sup> U. S. Dept. of Agric., Div. of Chem., Bul. 13, pt. 2, p. 165.

<sup>†</sup> Canada Dept. of Inland Rev., Bul. 73, p. 9.

<sup>‡</sup> Conn. Agric. Exp. Sta., Rep. 1898, p. 186.



*Lime* is determined from the ash as directed on page 312, having first separated the iron and phosphates.

The *sulphuric acid* due to calcium sulphate (added as an adulterant) is determined by precipitation with barium chloride of a very weak hydrochloric acid solution of the ash, the separated barium sulphate being washed, dried, ignited and weighed.

**Ether Extract.**—*Total, Volatile, and Non-volatile.\**—Two grams of the air-dry, powdered substance are placed in some form of continuous extraction apparatus, such as Soxhlet's or Johnson's (Chapter IV), and are subjected to extraction for sixteen hours with anhydrous, alcohol-free ether.† The ether solution is then transferred to a tared evaporating-dish, and allowed to evaporate spontaneously at the temperature of the room. After the disappearance of the ether, the evaporating-dish is placed in a desiccator over concentrated sulphuric acid and left over night, or for at least twelve hours, after which it is weighed, the residue in the dish being regarded as the total ether extract.

The dish and its contents are then subjected to a heat of about 100° C. for several hours, taking a long time to bring the temperature up to that point so as to avoid oxidation of the oil. Finally heat at 110° C. till the weight is constant. The final residue is the non-volatile, and the loss in weight the volatile ether extract.

**Alcohol Extract.**—*Method of Winton, Ogden, and Mitchell.‡*—Two grams of the powdered sample are placed in a 100-cc. graduated flask, which is filled to the mark with 95% alcohol. The flask is stoppered and shaken at half-hour intervals during eight hours, after which it is allowed to stand for sixteen additional hours without shaking, and the contents poured upon a dry filter. Of the filtrate, 50 cc. are evaporated to dryness in a tared platinum dish on the water-bath, and heated at 110° C. in an air-oven to constant weight. This method, while only approximate, is so much simpler than the tedious operation of continuous extraction, considering the long time required, that it is regarded as preferable for ordinary work, and, unless great care is taken, is nearly as accurate.

**Determination of Nitrogen.**—This, in spices other than pepper, is best done by means of the Gunning or Kjeldahl method.

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\* Richardson, U. S. Dept. of Agric., Div. of Chem., Bul. 13, p. 165.

† Petroleum ether may be used, yielding results which differ but slightly from those obtained with ethyl ether. As the latter has been used in the analyses of a large number of samples of spices, if these analyses are to be taken for standards of comparison it is evident that the same solvent should be used.

‡ Conn. Agric. Exp. Sta., Rep. 1898, p. 187.



**Determination of Starch.**—In spices like white pepper, ginger, and nutmeg that normally contain a high content of starch and very little other copper-reducing matter, the direct acid conversion process of starch determination is satisfactory.

In spices normally free from starch, such as cloves, mustard, and cayenne, where a starch determination indicates the amount of a foreign starch present as an adulterant, it is safer to use the diastase process.

Four grams of the powdered sample are extracted on a filter-paper (fine enough to retain all starch particles) first with five successive portions of 10 cc. of ether, then with 150 cc. of 10% alcohol. Owing to difficulty of filtering in the case of cassia and cinnamon, Winton recommends that all washing in the determination of starch in these substances be omitted. The residue is washed from the filter-paper by means of a stream of water into a 500-cc. flask, if the direct acid conversion method is used, using 200 cc. of water; 20 cc. of hydrochloric acid (specific gravity 1.125) are added, and the method from this point on followed, as detailed on page 292.

If the starch is to be determined by the diastase method, wash the residue from the filter-paper into a beaker with 100 cc. of water, and proceed as on page 292.

Determine the dextrose in either case by the Defren or Allihn method, or volumetrically, and convert dextrose to starch by the factor 0.9.

**Determination of Crude Fiber.**—Two grams of the substance are extracted with ordinary ether (or the residue left from the determination of the ether extract may be taken) and subjected to the regular method for determining crude fiber, by boiling successively with acid and alkali (page 286).

McGill recommends the use of the centrifuge in separating the crude fiber, after boiling with the alkaline solution.

**Determination of Volatile Oil.**—*Method of Girard and Dupré.\**—The spice is mixed with water and subjected to distillation, receiving the distillate in a graduated cylinder. The volume occupied by the essential oil (which is immiscible with water) can be thus read off and its content roughly determined. If the volatile oil is slightly soluble in water, separate out the water layer, having first read the volume of the oil layer, and extract the aqueous solution with petroleum ether. Evaporate the petroleum ether extract to dryness at room temperature

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\* *Analyse des Matières Alimentaires*, 2nd ed., p. 787.



in a tared dish, and add the volume due to the weight of the residue to the volume read off in the graduate.

**Microscopical Examination of Powdered Spices.**—As a rule few microscopical reagents are necessary in the routine examination of powdered spices for adulteration, unless a more careful study of the structure than is necessary to prove the presence of adulterants is desirable. The simple water-mounted specimen is usually sufficient to show the purity or otherwise of the sample. If in doubt as to the presence of starch in small quantities, iodine in potassium iodide should be applied to the specimen, well rubbed out under the cover-glass.

The tissues may be cleared by adding to the water mount a small drop of 5% sodium hydroxide, or by soaking a portion of the spice for a day in chloral hydrate solution. A valuable means of clearing dense tissues is to boil about 2 grams of the material successively with dilute acid and alkali as in the crude fiber process (p. 286), decanting (not filtering) the solution after each boiling.

The presence of occasional traces of a foreign substance, when viewed under the microscope, is hardly sufficient to condemn the sample as adulterated, since such traces are apt to be accidental.

**Composition of Miscellaneous Spice Adulterants.**—The chemical analyses of various spice adulterants commonly met with are given on page 427.

### CLOVES.

**Nature and Composition.**—Cloves are the dried, undeveloped flowers of the clove tree (*Caryophyllus aromaticus* or *Eugenia caryophyllata*), which belongs to the myrtle family (*Myrtaceæ*). The tree is an evergreen, from twenty to forty feet in height, cultivated extensively in Brazil, Ceylon, India, Mauritius, the West Indies, and Zanzibar. Its leaves are from 7.5 to 13 mm. long, and its flowers, of a purplish color, grow in clusters. The green buds in the process of growth change to a reddish color, at which stage they are removed from the tree, spread out in the sun, and allowed to dry, the color changing to a deep brown. Each whole clove consists of a hard, cylindrical calyx tube, having at the top four branching sepals, surrounding a ball-shaped casing, which consists of the tightly overlapping petals, and within which are the stamens and pistil of the flower. In taste the clove possesses a strong and peculiar pungency. One of its most valuable ingredients is the volatile clove oil. This is composed largely of *eugenol* ( $C_{10}H_{12}O_2$ ), which forms 70 to



## COMPOSITION OF SPICE ADULTERANTS.

	Moisture.	Ash.			Ether Extract.		Alcohol Extract.
		Total.	Soluble in Water.	Insoluble in HCl.	Volatile.	Non-volatile.	
English-walnut shells *	7.69	1.40	0.77	0.00	0.12	0.55	1.84
Brazil-nut shells *	9.08	1.59	1.06	0.17	0.07	0.57	1.01
Almond shells *	7.80	2.86	2.39	0.05	0.16	0.64	5.16
Cocconut shells *	7.36	0.54	0.50	0.00	0.00	0.25	1.12
Date stones *	8.24	1.24	0.76	0.04	0.36	8.38	16.72
Spruce sawdust *	8.77	0.23	0.16	0.00	0.07	0.77	1.50
Oak sawdust *	5.73	1.22	0.32	0.02	0.07	0.84	6.25
Linseed meal *	8.71	5.72	1.74	0.55	0.04	6.58	9.46
Cocoa shells *	10.44	8.40	4.66	0.83	1.00	2.99	4.77
Red sandalwood *	4.42	0.70	0.28	0.07	1.21	11.47	19.37
Ground olive stones †	9.50	0.88	0.24	0.44	0.06	0.24	
Buckwheat hulls. . . . .	7.63	1.84	1.24	0.00	0.07	0.38	2.17

	Reducing Matters by Acid Con- version.	Starch by Diastase Method.	Crude Fiber.	Nitrogen X 6.25.	Oxygen Ab- sorbed by Aqueous Extract.	Quercitan- ic Acid Equivalent.	Total Nitro- gen.
English-walnut shells *	19.30	1.01	56.58	1.69	0.53	2.08	0.27
Brazil-nut shells *	12.96	0.73	50.98	4.19	0.33	1.30	0.67
Almond shells *	22.72	0.84	49.89	1.75	0.40	1.56	0.28
Cocconut shells *	20.88	0.73	56.19	1.13	0.47	1.82	0.18
Date stones *	20.88	2.19	5.72	5.31	0.61	2.34	0.85
Spruce sawdust *	15.48	1.13	64.03	0.56	0.30	1.17	0.09
Oak sawdust *	17.10	1.68	47.79	1.63	3.13	12.22	0.26
Linseed meal *	21.15	14.06	8.30	31.81	1.00	3.90	5.09
Cocoa shells *	8.68	3.15	14.12	16.19	1.26	4.94	2.59
Red sandalwood *	6.79	1.12	52.30	3.06	0.59	2.29	0.49
Ground olive stones †	.....	1.73	57.46	1.06	.....	.....	0.17
Buckwheat hulls. . . . .	20.51	1.46	43.76	3.06	.....	.....	0.49

85 per cent of the oil, and a sesquiterpene known as caryophyllene. There are also in cloves a notable amount of fixed oil and resin, and also a peculiar form of tannin.

Very few complete analyses of cloves are on record. Richardson † seems to have been the earliest worker in the field to give anything at all satisfactory in the way of a number of determinations of value.

The following are maximum and minimum figures from the tabulated results of Richardson's analyses:

\* Winton, Ogden, and Mitchell, Conn. Exp. Sta. An. Rep., 1898, p. 210.

† Doolittle, Mich. Dairy and Food Dept. Bul. 94, 1903, p. 12.

‡ U. S. Dept. of Agric., Div. of Chem., Bul. 13.



	Water.	Ash.	Volatile Oil.	Fixed Oil and Resin.	Crude Fiber.	Albuminoids.	Nitrogen.	Oxygen Equivalent.	As Quercitannic Acid.
Whole cloves (7 samples):									
Maximum.....	10.67	13.05	18.89	10.24	9.75	7.	1.12	5.43	22.13
Minimum.....	2.90	5.50	10.23	7.12	6.18	4.73	.76	3.00	11.70
Stems (1 sample).....	10.18	6.96	4.40	4.03	13.58	5.78	.92	5.96	23.24
Ground cloves (9 samples):									
Maximum.....	9.58	10.73	13.93	7.44	13.80	6.48	1.04	6.20	24.18
Minimum.....	5.93	5.79	3.94	4.02	9.38	4.20	.70	2.89	11.28

McGill \* gives tables of analyses of pure and adulterated samples of cloves. Analyses of upwards of twenty samples of genuine cloves, both whole and ground, from these tables show the following maximum and minimum figures:

	Maximum.	Minimum.
Moisture.....	11.80	5.05
Volatile oil.....	19.63	9.24
Total volatile matter.....	30.68	16.25
Fixed oil.....	10.23	0.94
Total extraction.....	31.40	22.23
Ash.....	7.00	5.03

McGill also made analyses of whole cloves of several varieties, the following table being a summary of his results:

	No. of Analyses.	Moisture.	Total Volatile Matter.	Volatile Oil.	Total Extractive Matter.	Fixed Oil.
Penang cloves: Maximum.....	8	7.4	24.3	17.2	28.2	12.0
Minimum.....	....	5.0	20.7	14.8	24.4	9.5
Mean.....	....	6.2	22.4	16.2	27.0	10.8
Amboyna cloves: Maximum.....	8	6.7	25.9	19.2	29.2	10.0
Minimum.....	....	5.5	23.5	18.0	26.5	8.2
Mean.....	....	6.1	24.6	18.5	27.5	9.0
Zanzibar cloves: Maximum.....	13	6.7	23.6	18.3	28.1	10.7
Minimum.....	....	4.1	18.6	12.1	21.3	8.0
Mean.....	....	5.7	21.7	16.0	25.5	9.6

Maximum and minimum figures of thirteen samples of unadulterated cloves, as purchased from retail dealers in Connecticut and analyzed by Winton and Mitchell,† are as follows:

\* Canada Inland Rev. Dept. Bul. 73.

† Conn. Exp. Sta. Rep., 1898, pp. 176-177



	Maximum.	Minimum.
Ash, total .....	7.92	5.99
Ether extract, volatile. ....	18.25	11.03
“ “ non-volatile.....	7.19	4.87

Winton, Ogden, and Mitchell\* give more complete analyses of eight samples of whole cloves of known purity, representing Penang, Amboyna, and Zanzibar varieties, and two samples of clove stems, as follows:

	Moisture.	Ash.			Ether Extract.		Alcohol Extract.
		Total.	Soluble in Water.	Insoluble in HCl.	Volatile.	Non-volatile.	
Maximum.....	8.26	6.22	3.75	0.13	20.53	6.67	15.58
Minimum.....	7.03	5.28	3.25	0.00	17.82	6.24	13.99
Mean.....	7.81	5.92	3.58	0.06	19.18	6.49	14.87
Clove stems, mean. ....	8.74	7.99	4.26	0.60	5.00	3.83	6.79

	Reducing Matters by Acid Conversion, as Starch.	Starch by Diastase Method.	Crude Fiber.	Nitrogen, $\times 6.25$ .	Oxygen Absorbed by Aqueous Extract.	Quercitannic Acid.	Total Nitrogen.
Maximum.....	9.63	3.15	9.02	7.06	2.63	20.54	1.13
Minimum.....	8.19	2.08	7.06	5.88	2.08	16.25	0.94
Mean.....	8.99	2.74	8.10	6.18	2.33	18.19	0.99
Clove-stems, mean.....	14.13	2.17	18.71	5.88	2.40	18.79	0.94

**The Tannin Equivalent in Cloves.**—The amount of tannin in cloves was shown by Ellis to be so constant as to be of valuable assistance as a guide to their purity. The actual determination of tannin is, however, a long and difficult proceeding, and Richardson† has pointed out that it is not necessary, but that simply using the first part of the Löwenthal tannin process, and noting the “oxygen absorbed” as expressed by the oxidizing power of permanganate of potash on the material after extraction with ether, is quite as useful as determining the tannin, and is in effect proportional to the tannin present. The result is sometimes expressed as in Richardson’s figures above, as the *oxygen equivalent*, or as *quercitannic acid*.

**Determination of Tannin Equivalent.†—Reagents: Indigo Solution.**—Six grams of the indigo salt § are dissolved in 500 cc. of water by heat-

\* Conn. Exp. Sta. Rep., 1898, pp. 206, 207.

† U. S. Dept. of Agric., Div. of Chem., Bul. 13, p. 167.

‡ U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 60; Bul. 107 rev., p. 164.

§ The quality of the indigo used is of great importance since with inferior brands it is



ing. After cooling, 50 cc. of concentrated sulphuric acid are added, the solution made up to a liter and filtered.

*Standard Permanganate Solution.*—Dissolve 1.333 grams of pure potassium permanganate in a liter of water. This should be standardized by titrating against 10 cc. of tenth-normal oxalic acid (6.3 grams pure crystallized oxalic acid in 1,000 cc.), diluted to 500 cc. with water, heated to 60° C., and mixed with 20 cc. of dilute sulphuric acid (1:3 by volume). The permanganate solution is added slowly, stirring constantly, till a pink color appears.

Two grams of the material are extracted for twenty hours with pure anhydrous ether. The residue is boiled for two hours with 300 cc. of water, cooled, made up to 500 cc., and filtered.

Twenty-five cc. of the filtrate are pipetted into a 1200-cc. flask, 750 cc. of distilled water are added and 20 cc. of indigo solution.

The standard permanganate solution is then run in from a burette a drop at a time with constant shaking, until a bright golden yellow color appears, which indicates the end-point. Note the number of cubic centimeters required, represented by (a).

In a similar manner determine the number of cubic centimeters of standard permanganate solution consumed by 20 cc. of the indigo solution alone, represented by (b), and subtract this from (a).

The oxygen equivalent, or, as it is sometimes called, the "oxygen absorbed," is calculated from the equivalent in tenth-normal oxalic acid of the number of cubic centimeters of standard permanganate represented by  $a-b$ . 10 cc. of tenth-normal oxalic acid are equivalent to 0.008 gram of oxygen absorbed, or 0.0623 gram of quercitannic acid.

**Microscopical Examination of Cloves.**—Unless the finely powdered, water-mounted sample is well rubbed out under the cover-glass, many of the masses of cellular tissue will be too dense to recognize. With a little care, however, it is possible to make a very satisfactory water mount, though by soaking for twenty-four hours in chloral hydrate solution the more opaque masses are rendered very translucent.

Fig. 81, from Moeller, shows some of the characteristics of powdered cloves. The outer skin of the calyx tube is shown at (1) with its polygonal cells and large oil spaces showing through them; (2) shows the epidermis of the outer part of the lobes or wings of the calyx, with stomata

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impossible to get a sharp end-point. The indigo solution should be made from the very best variety of sulphindigotate, which may be obtained from Grueber & Co., of Leipzig, or Gehe & Co., of Dresden, under the name of *carminium caruleum*.



surrounded by irregularly shaped cells; (3) represents the epidermis of the petals, with crystals of calcium oxalate; a cross-section of the epidermis of the calyx is shown at (4); (5) shows the parenchyma, with calcium oxalate crystals and with one of the slender spiral ducts; (6) and (7) represent in cross-section and longitudinal section respectively the parenchyma of the middle layers of the ovary, one of the rounded, triangular pollen grains being shown at (12).

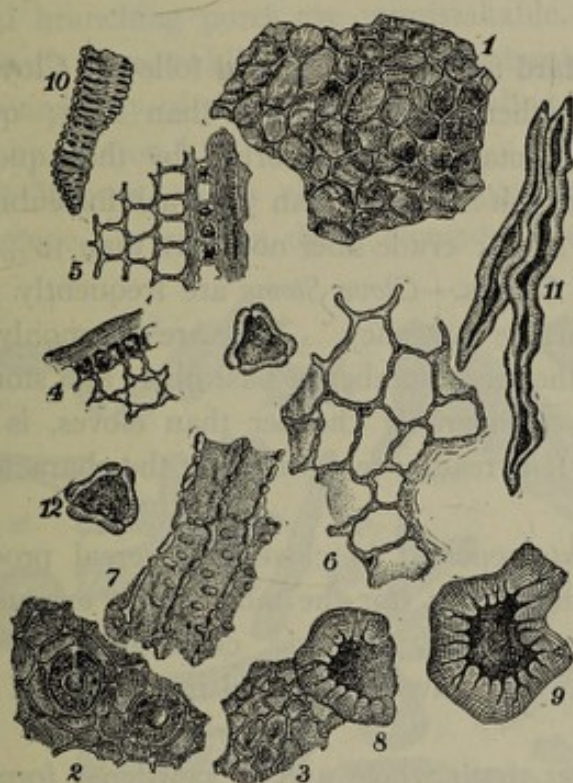


FIG. 81.—Powdered Cloves under the Microscope.  $\times 125$ . (After Moeller.)

Characteristics of clove stems, which are frequently used as adulterants of cloves, are found in (8), (9), (10), and (11). Stone cells of the outer skin and the inner portion of the clove stem are shown at (8) and (9) respectively; (10) shows one of the vascular ducts, and (11) two of the bast fibers. Both the vascular ducts and the stone cells are very characteristic of clove stems. Pure cloves have no stone cells and comparatively few bast fibers. Stems under the microscope show a large number of bast fibers and frequent stone cells, the latter being of a distinctly yellow color.

A plain water-mounted slide rarely shows all the structural details depicted in Fig. 81, but is nearly always sufficiently characteristic to



prove the purity of the sample. Fig. 220, Pl. XXV, shows the actual appearance of powdered cloves, mounted in water and examined under a magnification of 130. The general appearance of the cellular tissue is that of a loose, spongy mass filled with brown, granular material. Throughout the masses of tissue are to be seen small oil globules.

Cloves have no starch whatever. Aside from the stems, cloves are sometimes adulterated with clove fruit or "mother cloves," which have a small amount of a sago-like starch, and also contain some stone cells.

**The U. S. Standard** for pure cloves is as follows: Clove stems not more than 5%; volatile ether extract not less than 15%; quercitannic acid, calculated from the total oxygen absorbed by the aqueous extract, not less than 12%; total ash not more than 7%; ash insoluble in hydrochloric acid not more than 0.5%; crude fiber not more than 10%.

**Adulteration of Cloves.**—*Clove Stems* are frequently present in cloves and possess considerable pungency. They are commonly identified under the microscope by the large number of bast fibers and stone cells.

*Allspice*, being considerably cheaper than cloves, is sometimes used as an adulterant. It is readily recognized by the characteristics described on page 436.

*Other Adulterants* reported in cloves are cereal products (especially corn and wheat) and ginger (for the most part "exhausted"). Besides the above, pea starch, rice, turmeric, charcoal, sand, pepper, ground fruit stones, and sawdust have been found in samples of cloves examined in Massachusetts.

*Exhausted Cloves*, both whole and in powdered form, are not infrequently found on the market. These have been deprived of a portion of the volatile oil, and are much less pungent than the pure article, so that the difference in taste between the two varieties is quite marked. It is, however, rare that powdered cloves are sold consisting entirely of the exhausted variety, the more common practice being to mix from 10 to 25% of exhausted cloves with the pure powder, so that the sophistication is less apparent.

A determination of the volatile oil is the only reliable means of showing whether or not the material has been wholly or in part exhausted, though Villiers and Collin claim that under the microscope an exhausted sample of cloves shows the oil glands to be nearly empty, or to inclose much smaller droplets of oil than the pure variety.



With the exception of exhausted cloves, the presence of nearly every foreign ingredient is best and most quickly shown by the use of the microscope, though much information as to the purity of the sample can be gained by the ether extract, the percentage of ash, and of crude fiber.\*

**Cocoanut Shells.**—Figs. 226 and 227, Pl. XXVII, show samples of cloves adulterated with ground cocoanut shells. The long, spindle-shaped, yellow-brown and deeply furrowed stone cells of the adulterant with their thick walls and central branching pores are unmistakable. The dark-brown contents of the cells turn reddish brown when treated with potassium hydroxide. The anatomy of the cocoanut, including the shell, has been carefully studied by Winton.†

Fig. 82, after Winton, shows elements of powdered cocoanut shell under the microscope. *st* are the dark, elongated, yellow, porous stone



FIG. 82.—Cocoanut-shell Powder. *st*, dark-yellow stone cells with brown contents; *t*, reticulated trachea; *sp*, spiral trachea; *g*, pitted trachea; *w*, colorless, and *br*, brown, parenchyma of mesocarp; *f*, bast fibres, with stegmata (*ste*).  $\times 160$ . (After Winton.)

cells with their brown contents, these stone cells being the most distinctive characteristic of the ground shells. *t*, *sp*, and *g* are the various forms of trachea; *w* and *br* are respectively colorless and brown parenchyma of the mesocarp or outer coat, portions of which always adhere to the nutshell and are ground with it.

\* Note especially the sharp distinction between these values in the case of pure cloves and of clove stems in Richardson's table.

† The Anatomy of the Fruit of the Cocoanut. Conn. Exp. Sta. Rep., 1901, p. 208.



Fig. 264, Pl. XXXVI, shows a photomicrograph of powdered cocoanut shells, mounted in gelatin. The long, spindle-shaped stone cells are especially apparent,

Ground cocoanut shells have been used in various spices besides cloves, especially allspice and pepper. In the following tabulated results of analyses by Winton, Ogden, and Mitchell \* are shown the wide deviation between the chemical constants of cocoanut shells and several of the spices in which they appear as adulterants.

	Black Pepper.	Cloves.	Allspice.	Nutmeg.	Cocoanut Shells.
Water. ....	11.96	7.81	9.78	3.63	7.36
Total ash. ....	4.76	5.92	4.47	2.28	0.54
Ash soluble in water. ....	2.54	3.58	2.47	0.86	0.50
Ash insoluble in hydrochloric acid. ....	0.47	0.06	0.03	0.00	0.00
Volatile ether extract. ....	1.14	19.18	4.05	3.02	0.00
Non-volatile ether extract. ....	8.42	6.49	5.84	36.70	0.25
Alcohol extract. ....	9.62	14.87	11.79	10.77	1.12
Reducing matters, as starch, acid conversion	38.63	8.99	18.03	25.56	20.88
Starch by diastase method. ....	34.15	2.74	3.04	23.72	0.73
Crude fiber. ....	13.06	8.10	22.39	2.51	56.19
Total nitrogen. ....	2.26	0.99	0.92	1.08	0.18
Oxygen absorbed by aqueous extract. ....	.....	2.33	1.24	.....	0.23
Quercitannic acid equivalent. ....	.....	18.19	9.71	.....	1.83

#### ALLSPICE, OR PIMENTO.

**Nature and Composition.**—Allspice is the dried fruit of the *Eugenia pimenta*, an evergreen tree belonging to the same family (*Myrtaceæ*) as the clove. It is indigenous to the West Indies, and is especially cultivated in Jamaica.

The allspice berry is grayish or reddish brown in color, and is hard and globular, measuring from 4 to 8 mm. in diameter, being surmounted by a short style. This is imbedded in a depression, and around it are the four lobes of the calyx, or the scars left by them after they have fallen off. The berry has a wrinkled, ligneous pericarp, with many small excrescences filled with essential oil. The pericarp is easily broken between the fingers, showing the berry to be formed of two cells with a single, brown, kidney-shaped seed in each, covered with a thin, outer coating, inclosing an embryo rolled up in a spiral.

The berries are gathered when they have attained their largest size, but before becoming fully ripe. If allowed to mature beyond this stage, some of the aroma is lost.

\* Conn. Ag. Exp. Sta. Rep., 1901, p. 225.



Though considerably less pungent than other spices, allspice possesses an aroma not unlike cloves and cassia. In chemical composition it most resembles cloves, containing both volatile oil and tannin; but, unlike cloves, it contains much starch, the starch being contained in the seeds. The volatile oil of allspice is very similar to clove oil. It is slightly laevorotary, and is composed of eugenol and a sesquiterpene not determined. It is present in allspice to the extent of 3 to 4.5 per cent. The boiling-point of the oil is  $255^{\circ}\text{C}$ .

Authoritative full analyses of allspice are even more meager than of cloves. Analyses of one sample of whole allspice and five samples of the ground spice, made by Richardson,\* are thus summarized:

	Water.	Ash.	Volatile Oil.	Fixed Oil.	Undetermined.	Crude Fiber.	Albuminoids.	Nitrogen.	Tannin Equivalent.	Oxygen Required.
Whole .....	6.19	4.01	5.15	6.15	59.28	14.83	4.38	.70	10.97	2.81
Ground:										
Maximum .....	8.82	5.53	3.32	6.92	58.24	18.98	5.42	.87	12.74	3.36
Minimum .....	5.51	3.45	2.07	3.77	56.86	13.45	4.03	.64	8.27	2.12

Seventeen samples of unadulterated allspice, as sold on the Connecticut market, were analyzed by Winton and Mitchell,† with maximum and minimum results as follows:

Ash.	Maximum.	Minimum.
Total .....	7.51	4.34
Insoluble in hydrochloric acid (sand) ..	.95	.40
Ether extract, volatile .....	3.50	1.34
Ether extract, non-volatile .....	6.22	3.78

Three samples of pure whole allspice were more fully analyzed by Winton, Mitchell, and Ogden with the results given on page 436.‡

**The Tannin Equivalent in Allspice.**—Tannin is present in allspice, though to a less extent than in cloves. The exact amount present is rarely determined, but rather the "oxygen equivalent," or quercitannic acid, as explained on page 429, the determination being carried out as there detailed.

\* U. S. Dept. of Agric., Div. of Chem., Bul. 13, p. 229.

† An. Rep. Conn. Exp. Sta., 1898, pp. 178, 179.

‡ Ibid., pp. 208, 209.



	Moisture.	Ash.			Ether Extract.		Alcohol Extract.
		Total.	Soluble in Water.	Insoluble in HCl.	Volatile.	Non-volatile.	
Maximum.....	10.14	4.76	2.69	0.06	5.21	7.72	14.27
Minimum.....	9.45	4.15	2.29	0.00	3.38	4.35	7.39
Average.....	9.78	4.47	2.47	0.03	4.05	5.84	11.79

	Reducing Matters by Acid Conversion, as Starch.	Starch by Diastase.	Crude Fiber.	Nitrogen, $\times 6.25$ .	Oxygen Absorbed by Aqueous Extract.	Quercitannic Acid.	Total Nitrogen.
Maximum.....	20.65	3.76	23.98	6.37	1.59	12.48	1.02
Minimum.....	16.56	1.82	20.46	5.19	1.03	8.06	0.83
Average.....	18.03	3.04	22.39	5.75	1.24	9.71	0.92

**Microscopical Examination of Powdered Allspice.**—By soaking the powder twenty-four hours or more in chloral hydrate, many of the harder portions are rendered much more transparent than would otherwise be possible. Fig. 83, after Moeller, shows the microscopical structure of various elements that go to make up allspice powder.

The epidermis, or outer layer of the berry with its small cells, is shown in cross-section at (1a) and in surface view at (2). Just beneath the outer coat are the large oil spaces (1b) and still further below the stone-cells (1c). The fruit parenchyma (3) has vascular tissues running through it. (4) and (5) are the inner epidermis and stone cells of the dividing partitions between the seeds. Small hairs connected with the outer epidermis are shown at (6). (7) and (8) show in cross-section a portion of the seed-shell and inclosed seed or embryo, with the starch (8a) and the colored lumps of gum or resin (8b) of a port-wine color. These colored cells exist in the seed coating, and, although only one is here shown, constitute a very important and striking characteristic of allspice. (9) represents the spongy parenchyma of the seed shell, and (10) shows its epidermis. In the parenchyma of the fruit and of the partitions between the cells are seen, but not always plainly, minute crystals of calcium oxalate (see (4) and (5)).

These details so closely drawn by Moeller are idealized, but serve well to indicate what should be looked for. In practice the water-mounted specimen shows all the characteristics necessary to identify pure allspice, and most if not all its adulterants. In fact pimento is one of the easiest spices to identify under the microscope, by reason of its striking characteristics.



Three distinctive features are especially typical, viz.: First, the starch grains, which are very uniform in size, measuring about 0.008 mm. in diameter, being nearly circular as a rule, and often arranged in groups not unlike masses of buckwheat starch. Ordinarily these masses contain fewer granules than do those of buckwheat. The granules are

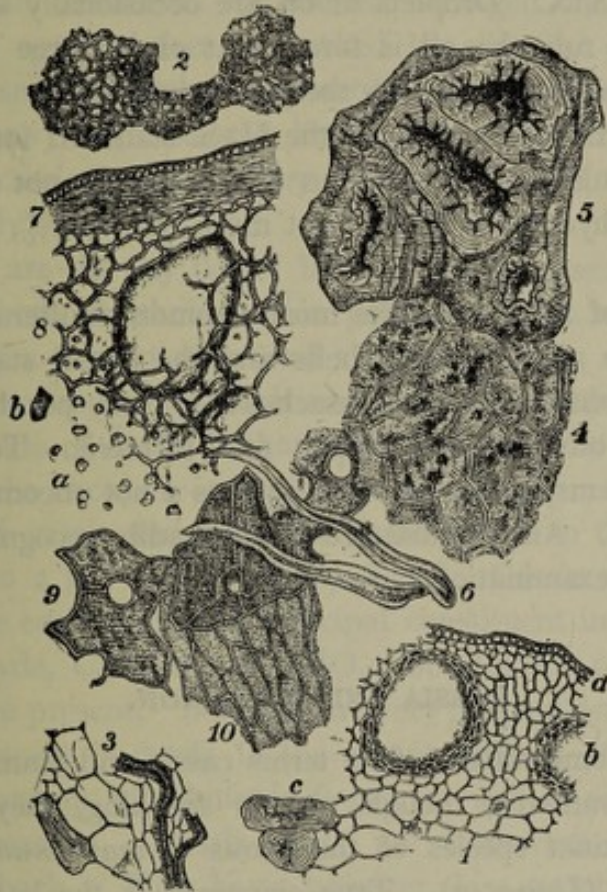


FIG. 83.—Powdered Allspice under the Microscope.  $\times 125$ . (After Moeller.)

smaller and more inclined to the circular than to the polygonal form, while in many cases they have distinct central hila. The starch grains are very numerous and are found in nearly every field. See Fig. 195, Pl. XIX.

A second distinctive feature of allspice is the stone cells, of which there are many. These are more often colorless, and in most cases very large and plainly marked. They are sometimes seen singly and at other times grouped together. Frequently they are attached to pieces of brown parenchyma.



The third and most characteristic feature of allspice powder under the microscope is the striking appearance of the lumps of gum or resin, which are of a more or less deep port-wine or amber color and are contained in the middle layers of the seed coat. These cells are very striking, occurring sometimes in isolated bits, and in other cases in aggregations of from 2 to 4 or even 6 to 8 cells. These resinous lumps appear plainly in Fig. 194, Pl. XIX. Droplets of oil are occasionally seen, but not in profusion. As a rule the oil is forced out of its large containing cells and into the surrounding tissue by the process of drying.

**U. S. Standards.**—According to the U. S. standard for allspice, quercitannic acid should not be less than 8%, total ash not more than 6%, ash insoluble in hydrochloric acid not more than 0.4%, crude fiber not more than 25%.

**Adulteration of Allspice.**—The most common adulterants reported in powdered allspice are cocoanut shells and the cereal starches. Besides these the writer has found in Massachusetts, peas, pea hulls, exhausted ginger, cayenne, olive stones, pepper, and turmeric. To this list may be added clove stems, which are on record as a not uncommon adulterant in some localities. All of these are to be readily recognized by a careful microscopical examination.

#### CASSIA AND CINNAMON.

**Nature and Composition.**—The terms cassia and cinnamon are interchangeable in commerce, though, strictly speaking, they represent two separate and distinct species of the genus *Cinnamomum*, belonging to the laurel family (*Lauraceæ*). True cinnamon is the bark of *Cinnamomum zeylanicum*, a tree from 20 to 30 feet high, having horizontal or drooping branches, and native to the island of Ceylon, but cultivated also in some parts of tropical Asia, in Sumatra, and in Java. The entire yield of pure Ceylon cinnamon is extremely small, and but little of it is found in this country. It is the very thin, inner bark of the tree, and is of a pale, yellowish-brown color, being found on the market in long, cylindrical, quill-like rolls or pieces, the smaller rolls being inclosed in the larger. The outer surface is marked by round dark spots, corresponding to points of insertion of the leaves, and it is also furrowed lengthwise by somewhat wavy, light-colored lines. The inner surface of the bark is darker colored, and has no lines. In thickness the bark varies from 1.5 to 3 mm. Both the inner and outer coatings of the bark of Ceylon cinnamon are usually removed in the process of preparation, so



that it is of a much cleaner and more even texture than the cassia bark, which is thicker and heavier by reason of the outer cork layer usually left on it.

The cheaper and more common cassia is the bark of the *Cinnamomum cassia*, which comes from China, Indo-China, and India. It is of a darker color than that of cinnamon, of coarser texture, and as a rule about four times as thick. Most varieties of cassia bark are less tightly rolled than cinnamon, and are not arranged one within the other in layers. The outer surface is marked by elliptical spots left by the leaves, and by small, dark-brown, wart-like protuberances. Cassia does not have the wavy, light-colored lines found in the cinnamon. Both cinnamon and cassia barks are very aromatic in taste, somewhat astringent, and slightly sweet.

*Cassia buds* are the dry flower buds of China cassia, and are found in the market both in whole and in powdered form. Powdered cassia often consists of a mixture of several varieties of bark, while the cheaper grades sometimes contain an admixture of the ground buds.

The best grade of cassia is that from Saigon, a much cheaper, from Batavia, while the cheapest is the China cassia.

The odor of cassia and cinnamon bark is due to the volatile oil, of which from 1 to 2 per cent is usually found. Cassia and cinnamon oil greatly resemble each other, the principal constituent in either case being cinnamic aldehyde,  $C_6H_5CH:CH.CHO$ . Besides this, one or more esters of acetic acid are present. Both oils are very pungent and intensely sweet.

Starch is present in cassia to the extent of from 16 to 30 per cent. A very small amount of tannin is found, as well as cinnamic acid and mucilaginous matters. Cassia buds are somewhat similar in composition to the bark. They have, however, less starch and crude fiber, and higher contents of volatile oil and nitrogen than the bark.

Richardson\* has made analyses of a few samples of pure whole cinnamon and cassia, from which the following are taken:

	Water.	Ash.	Volatile Oil.	Fixed Oil, etc.	Crude Fiber.	Albuminoids.	Undetermined.	Nitrogen.
Ceylon cinnamon, 1.....	5.40	4.55	1.05	1.66	33.08	2.98	51.28	.48
" " 2.....	7.43	3.40	.82	1.58	25.63	3.80	56.84	.62
Cassia buds.....	4.79	5.58	3.59	5.21	8.60	7.00	65.23	1.12
Cassia bark (4 samples):								
Maximum.....	17.45	8.23	3.51	2.38	26.29	4.55	65.33	.73
Minimum.....	9.32	2.48	.55	.74	14.33	2.63	48.65	.42

\* U. S. Dept. of Agric., Div. of Chem., Bul. 13, p. 221.



Winton, Ogden, and Mitchell's\* results of analyses of whole samples of cinnamon, cassia, and cassia buds are thus summarized:

	Moisture.	Ash.			Ether Extract.	
		Total.	Soluble in Water.	Insoluble in HCl.	Volatile.	Non- volatile.
Ceylon cinnamon (6 samples):						
Maximum.....	10.48	5.99	2.71	0.58	1.62	1.68
Minimum.....	7.79	4.16	1.40	0.02	0.72	1.35
Average.....	8.63	4.82	1.87	0.13	1.39	1.44
Cassia bark (20 samples):						
Maximum.....	11.91	6.20	2.52	2.42	5.15	4.13
Minimum.....	6.53	3.01	0.71	0.02	0.93	1.32
Average.....	9.24	4.73	1.68	0.56	2.61	2.12
Cassia buds (2 samples):						
Average.....	7.93	4.64	2.88	0.27	3.88	5.96

	Alcohol Extract.	Reducing Matters by Acid Conversion, as Starch.	Crude Fiber.	Nitrogen, × 6.25.	Total Nitrogen.
Ceylon cinnamon (6 samples):					
Maximum.....	13.60	22.00	38.48	4.06	0.65
Minimum.....	9.97	16.65	34.38	3.25	0.52
Average.....	12.21	19.30	36.20	3.70	0.59
Cassia bark (20 samples):					
Maximum.....	16.74	32.04	28.80	5.44	0.87
Minimum.....	4.57	16.65	17.03	3.31	0.53
Average.....	8.29	23.32	22.96	4.34	0.69
Cassia buds (2 samples):					
Average.....	10.88	10.71	13.35	7.53	1.20

**Structure of Powdered Cassia under the Microscope.**—Fig. 84, from Moeller, shows various elements of cassia bark as viewed microscopically. (1) shows in cross-section a portion of the cork and outer layer of the bark rind, with flat cells nearest the surface, having somewhat thick walls and reddish-brown contents, and, farther in, the cells *s*, with mucilaginous material.

The stone cells of the intermediate layer of bark are shown at (2). Here the tendency of the stone cells is to be thicker on one side than on the other, as is plainly shown. (3) represents the structure of the inner layer of the bark, showing bast fibers *b* cut across, and more of the so-called mucilaginous cells *s* of large size, which normally contain the ethereal or volatile oil. The starch granules (4) are contained in great abundance in the polygonal cells of the parenchyma of the intermediate

\* Twenty-second Annual Report Conn. Exp. Sta., 1898, pp. 204, 205.



and inner bark layers. (6) represents a fragment of a bast fiber, which is often shown in cassia powder with connecting parenchyma. The stone-cells of the cork are shown in plan view at (7). Very small, needle-like crystals of oxalate of calcium are occasionally to be seen if looked for carefully. They occur in the parenchyma cells of the inner and intermediate layers of the bark.

The microscopical structure of Ceylon cinnamon much resembles that of cassia. Cassia starch grains measure from 0.0132 to 0.0222 mm.,

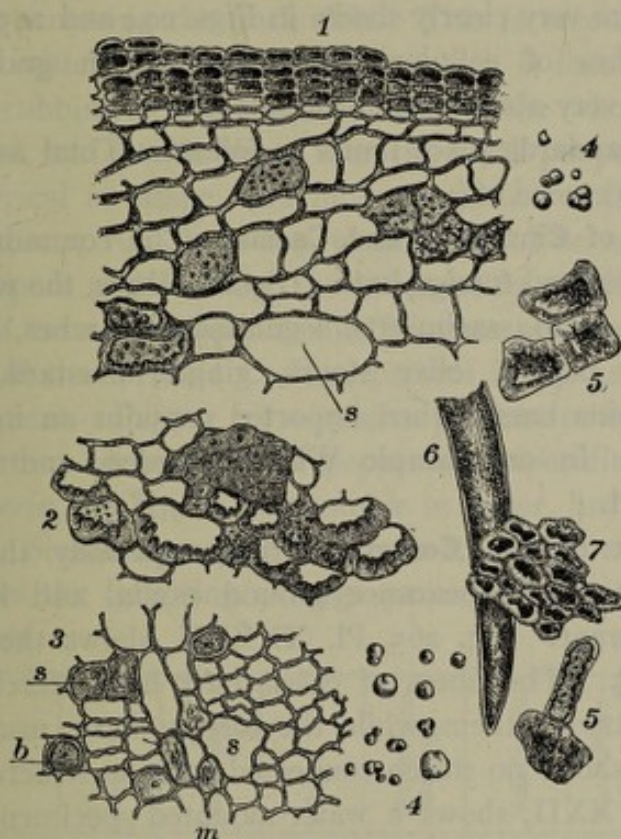


FIG. 84.—Powdered Cassia under the Microscope.  $\times 125$ . (After Moeller.)

being considerably larger and more abundant than those of true cinnamon. As a rule the bast fibers of cassia are larger, but shorter, than those of cinnamon, and provided with thicker walls.

Figs. 203 and 204, Pl. XXI, show various phases of pure cassia bark as photographed from water-mounted specimens of the powder. Cassia starch somewhat resembles that of allspice, but it is not as a rule found in masses containing as many granules as does the allspice starch. Very commonly two or three of the starch granules are arranged together in



such a manner that at first sight they appear to form a single large granule, but on more careful examination are seen to be two- and three-lobed, consisting of several smaller grains. Stone cells, which are very abundant in the powdered cassia, do not happen to be included to any extent in the photographed fields. Cassia stone cells are generally more oblong than those of allspice, and are more often brown in color, while the allspice stone cells are generally colorless.

A distinctive feature of powdered cassia consists in the long-amber-colored wood fibers, some distributed in bundles, and others arranged singly. These are very clearly shown in Figs. 204 and 205.

Yellow patches of cellular tissue with starch grains interspersed among them are very abundant in the powder.

**The U. S. Standards** place limits as follows: Total ash not to exceed 5%; sand not to exceed 2%.

**Adulteration of Cinnamon and Cassia.**—The commonest adulterants are cereal products and foreign bark. Besides these, the writer has found, in samples sold in Massachusetts, leguminous starches, pea hulls, nutshells, turmeric, pepper, olive stones, ginger, mustard, and sawdust. Much of the China cassia when imported contains an inexcusably large amount of dirt. In one sample Winton, Ogden, and Mitchell found over 15% of sand.

**Ground Bark of the Common Trees**, especially that of the elm, resembles in physical appearance ground cassia, and is to be looked for as an adulterant. Fig. 265, Pl. XXXVII, shows the appearance of ground elm bark. The fibers of cassia bark have starch granules as a rule interposed among them, while the foreign bark, usually of a much coarser texture, shows no starch connected with its structure.

Fig. 206, Pl. XXII, shows a water-mounted specimen of adulterated cassia powder, chosen from samples purchased in the Massachusetts market. Nothing but the adulterant (a foreign bark) shows in the field. The tissue is loose and considerably coarser than that of cassia bark.

#### PEPPER.

**Nature and Composition.**—Pepper is the dried berry of the pepper plant (*Piper nigrum*), a climbing shrub belonging to the family *Piperaceæ*, native to the East Indies, but cultivated in many tropical countries. The height of the pepper plant is from twelve to twenty feet. When the fruit begins to turn red, it is gathered and then dried, by which process it turns black and shrivels up, forming the black peppercorns of commerce. They are spherical single-seeded berries, about 5 mm. in diam-



merce. They are spherical single-seeded berries, about 5 mm. in diameter, covered with a brownish-gray epicarp, and having on the under side the remains of a short stem. At the top of the berry is an indistinct trace of a style, and of a lobed stigma.

Varieties of black pepper are named from the localities in which they are grown or from which they are shipped, as Singapore, Lampong, Sumatra, Tellicherry, Malabar, Acheen, Penang, Alleppi, Trang, Mangalore, etc.

White pepper is obtained by decorticating the fully ripened black peppercorns, or removing the dark skin. This is accomplished by macerating them in water to loosen the skin, which is then removed readily by drying and rubbing between the hands. White whole pepper grains are grayish white, and a trifle larger than the black pepper berries. They are nearly spherical in shape, and have a number of light-colored lines that, like meridians, run from top to bottom. The common varieties are Siam, Singapore and Penang, the latter being coated with lime.

The pungent taste of pepper is due in great part to its essential oil, a hydrocarbon of the formula  $C_{10}H_{16}$ , present in amounts varying from 0.5 to 1.7 per cent. Pepper oil contains phellandrene and a terpene.

Other important constituents of pepper are piperidine, and the crystalline base *piperin*,  $C_{17}H_{19}NO_3$ , insoluble in water, but soluble in ether, and in alcohol. Starch is present in pepper to a large extent.

Burcker gives the following average percentage composition of black and white pepper:

	Ash.	Cellulose.	Water.	Nitrogenous Matter.	Volatile Oil.	Fat, Piperin, and Resin.	Starch and Dextrin.	Other Non-nitrogenous Matters.
Black pepper .....	4.57	12.45	12.50	11.98	1.36	6.85	42.90	7.39
White pepper. ....	1.80	6.08	13.56	11.12	0.94	7.11	56.04	3.35

Richardson's\* analyses of three samples of whole black and two samples of whole white pepper, all pure, are as follows:

	Water.	Ash.	Volatile Oil.	Piperin and Resin.	Alcohol Extract.	Starch (Acid Conversion)
Black pepper: West coast. ....	8.91	4.04	.70	7.29	....	36.52
Acheen. ....	8.29	4.70	1.69	7.72	6.06	37.50
Singapore. ....	9.83	3.70	1.60	7.15	5.74	37.30
White pepper: West coast. ....	9.85	1.41	.57	7.24	....	40.61
Singapore. ....	10.60	1.34	1.26	7.76	2.57	43.10

\* U. S. Dept. of Agric., Bur. of Chem., Bul. 13, part 2, p. 206.



	Undeter- mined.	Crude Fiber.	Albumin- oids.	Total N $\times 6.25$ .	Total N.
Black pepper: West coast. ....	24.62	10.23	7.69	9.81	1.57
Acheen. ....	13.64	10.02	10.38	12.60	2.02
Singapore . ....	17.66	10.02	10.00	12.08	1.93
White pepper: West coast. ....	23.28	7.73	9.31	11.48	1.83
Singapore . ....	19.55	4.20	9.62	11.90	1.90

Richardson gives the following variations in the constituents of pure pepper:

	Black.	White.
Water .....	8.0 to 11.0	8.0 to 11.0
Ash.....	2.75 to 5.0	1.0 to 2.0
Volatile oil.....	.50 to 1.75	.50 to 1.75
Piperin and resin. ....	7.0 to 8.0	7.0 to 8.0
Starch .....	32.0 to 38.0	40.0 to 44.0
Crude fiber. ....	8.0 to 11.0	4.11 to 8.0
Albuminoids.....	7.0 to 12.0	8.0 to 10.0

McGill's \* analyses of six samples of whole black, and five samples of whole white pepper, all genuine, are thus summarized:

	Moisture, etc., Lost at 100° C.	Ash.					Alcohol Extract.
		Soluble in Hot Water.	Insoluble in Water.	Total.	Insoluble in Hydro- chloric Acid.	Sand Expressed as Per Cent of Total Ash.	
Black: Maximum .....	14.10	2.64	3.06	5.16	1.08	21	9.06
Minimum .....	10.62	2.07	1.46	3.98	.06	2	8.28
Mean .....	12.03	2.41	2.05	4.47	0.36	8	8.71
White: Maximum .....	13.00	0.72	3.04	3.65	0.88	42	8.92
Minimum .....	11.30	0.14	1.50	1.64	0.26	9	7.00
Mean .....	12.34	0.54	2.46	3.00	0.55	21	7.73

Winton, Ogden, and Mitchell's, and Winton and Bailey's † analyses of whole black pepper and whole white pepper, representing the leading varieties imported into the United States, also of pepper shells and long pepper, are summarized in the following table:

\* Canada Int. Rev. Dept. Bul. 20, 1890.

† An. Rep. Conn. Exp. Sta., 1898, pp. 198-199; 1903, pp. 158-164.



	Number of Samples.	Moisture.	Ash.			Ether Extract.		Alcohol Extract.	Reducing Matters by Acid Conversion, as Starch.	Starch by Diastase.	Crude Fiber.	Total Nitrogen, less N X 6.25.	Nitrogen.	
			Total.	Soluble in Water.	Insoluble in HCl.	Volatile.	Non-volatile.						Total.	In Non-volatile Ether Extract.
Whole Black Pepper:														
Singapore. ....	5	12.00	3.49	2.10	0.12	1.06	7.73	8.89	42.80	38.51	11.04	12.71	2.36	0.31
Tellicherry. ....	2	11.56	4.21	2.75	0.01	0.83	6.94	8.80	41.67	37.01	12.20	11.79	2.15	0.27
Lampong. ....	4	11.46	6.05	2.37	1.06	1.22	8.57	9.41	39.69	35.08	12.37	10.90	2.08	0.33
Acheen A*. ....	1	12.09	5.04	2.78	0.48	1.09	9.17	10.04	38.17	33.30	13.07	10.88	2.11	0.37
Acheen B. ....	3	12.41	5.65	2.91	1.08	1.49	8.59	9.36	37.11	32.17	14.80	11.79	2.24	0.35
Acheen C. ....	5	11.78	5.99	3.06	1.05	1.64	9.29	10.15	31.97	25.76	17.19	12.32	2.35	0.38
All varieties. ....	20	12.95	6.85	3.20	1.63	2.20	10.37	11.86	43.47	39.66	18.25	13.81	2.53	0.39
Minimum		10.63	3.09	1.75	0.00	0.65	6.86	8.31	28.15	22.05	10.75	10.50	2.03	0.27
Average		11.86	5.10	2.60	0.70	1.28	8.41	9.44	38.28	33.28	13.62	11.93	2.25	0.33
Whole White Pepper:														
Decorticated. ....	2	12.89	1.06	0.47	0.01	0.56	7.24	7.83	64.85	63.16	0.60	11.03	2.08	0.32
Singapore. ....	2	13.47	1.33	0.33	0.09	0.92	7.89	8.45	56.71	53.89	4.10	11.06	2.11	0.34
Siam. ....	3	13.63	1.47	0.38	0.10	0.69	6.64	7.36	59.01	56.18	3.52	10.69	2.00	0.29
Penang. ....	3	13.68	2.84	0.65	0.15	0.76	6.32	7.32	57.17	54.01	3.80	10.88	2.01	0.27
All Varieties. ....	10	14.47	2.96	0.80	0.20	0.95	7.94	8.55	64.92	63.60	4.25	11.19	2.13	0.34
Maximum		12.72	1.03	0.28	0.00	0.49	6.26	7.19	56.43	53.11	0.54	10.44	1.95	0.26
Minimum		13.47	1.77	0.47	0.10	0.73	6.91	7.66	59.17	56.47	3.14	10.89	2.04	0.30
Average		10.66	11.91	3.20	4.70	1.06	4.97	6.30	21.69	15.30	32.15	14.19	2.36	0.15
Pepper Shells. ....	3	10.52	10.25	2.28	2.63	0.68	3.04	4.00	11.43	2.30	23.27	12.31	2.12	0.09
Minimum		9.47	5.93	4.20	0.22	1.55	6.61	8.67	42.88	39.55	5.76	12.25	2.18	0.22
Whole Long Pepper. ....	1													

\* Acheen pepper contains hollow kernels and shells varying in amount with the grade.



The following table summarizes the results of full analyses of pepper and pepper shells recently made by Doolittle:\*

	No. of Samples.	No. of Varieties.	Moisture.	Ash.			Starch by Diastase Method.
				Total.	Insoluble in HCl.	Soluble in Water.	
Black pepper:	45	12					
Maximum.....	....	....	11.06	8.04†	2.59†	3.32	41.75
Minimum.....	....	....	8.09	3.43	0.05	1.65	25.09
Average.....	....	....	9.54	4.99	0.58	2.49	36.69
White pepper:	25	9					
Maximum.....	....	....	13.34	4.28	0.86	1.16	63.55
Minimum.....	....	....	8.04	0.86	0.05	0.12	48.88
Average.....	....	....	9.87	1.69	0.19	0.34	54.37
Long pepper:	3						
Maximum.....	....	....	10.13	14.39	5.92	4.39	45.87
Minimum.....	....	....	8.43	6.12	0.45	1.72	28.43
Pepper shells:	4						
Maximum.....	....	....	11.01	28.81	22.90	4.66	11.70
Minimum.....	....	....	7.00	7.82	0.79	1.53	9.28

	Ether Extract.		Crude Fiber.	Nitrogen.		Total N less N in non-volatile Ether Extract X 6.25.
	Volatile.	Non-volatile.		Total.	In Non-volatile Ether Extract.	
Black pepper:						
Maximum.....	2.10	10.44	18.89	2.38	0.45	13.12
Minimum.....	0.85	6.60	10.05	1.86	0.25	9.25
Average.....	1.30	7.67	11.12	2.11	0.31	11.20
White pepper:						
Maximum.....	1.66	7.26	7.65	2.14	0.34	11.56
Minimum.....	0.78	5.65	0.10	1.85	0.24	9.69
Average.....	1.17	6.46	4.17	1.97	0.30	10.44
Long pepper:						
Maximum.....	1.01	7.53	10.01	2.04	0.22	12.06
Minimum.....	0.79	5.71	7.19	2.13	0.18	11.37
Pepper shells:						
Maximum.....	1.11	4.67	28.22	1.82	0.12	11.25
Minimum.....	0.89	1.51	21.06	1.72	0.02	10.00

† Two samples of Acheen C pepper had a total ash of 8.00% and 8.04%, with "ash insoluble in HCl" of 2.50% and 2.40% respectively. Eliminating these two samples, which were evidently abnormally high in sand and dirt, the highest total ash of the remaining 43 samples was 7.00%, while the highest ash insoluble in HCl was 1.80%.

**Determination of Nitrogen in Black and White Pepper.**—Winton, Ogden, and Mitchell have shown that the Kjeldahl and Gunning methods are inapplicable in the case of pepper, owing to the presence of piperin, but that the Gunning-Arnold † method gives accurate results. In accordance with this method, 1 gram of the sample is mixed with a gram each of copper sulphate and red oxide of mercury, about 16 grams of potassium

\* Mich. Dairy and Food Comm. Bul. 94.

† Zeits. anal. Chem., 31, 1892, p. 525.



sulphate, and 25 cc. of sulphuric acid in a Kjeldahl flask, for both digestion and distillation, of about 600-cc. capacity. The heating is conducted in the usual manner, beginning with a gentle heat till the frothing ceases, and gradually increasing the temperature till the mixture boils. The boiling is continued for three or four hours, after which the flask is cooled, and to it are added 300 c.c. of water, 50 cc. of potassium sulphide solution,\* and enough of a saturated solution of sodium hydroxide to render the reaction alkaline.

The flask is then connected to the condenser, and the distillation conducted as in the usual Gunning method, using zinc dust to prevent bumping, receiving the distillate into standard acid, and titrating against standard alkali.

**Nitrogen Determination in the Ether Extract.**†—Ten grams of the sample are extracted with absolute ether for twenty hours in a continuous-extraction apparatus, the extract being collected in a tared Kjeldahl extraction- and distillation-flask, the same as used in the preceding section. The ether is then evaporated off, the residue dried to constant weight at 110° C. and its weight ascertained. The nitrogen is then determined in the ether extract by the Gunning-Arnold method.

**Determination of Piperin.**‡—Fifty grams of the sample are thoroughly exhausted with hot alcohol, and the alcohol extract evaporated to dryness. The dry residue is then treated with a solution of potassium hydroxide, and washed upon a filter. The residue is washed several times with the caustic alkali, which dissolves the resinous matters, and afterwards with water. It is then dissolved in alcohol, from which crystals of crude piperin separate on evaporation. These are redissolved in alcohol, and precipitated by the addition of water. The crystalline precipitate is collected on a tared filter, washed with water, dried, and weighed.

Piperin may be roughly estimated by multiplying the nitrogen in the ether extract by the factor 20.36.

The amount of piperin varies considerably, ranging in black pepper from 4 to 9 per cent.

**Microscopical Characteristics of Ground Pepper.**—Moeller's representation of powdered black pepper shows what should be looked for under the microscope with the best conditions (Fig. 85). The shell of the peppercorn, a cross-section of which is shown at (1), consists of the

\* Forty grams  $K_2S$  in 1 liter or water.

† Method of Winton, Ogden and Mitchell.

‡ Villiers et Collin, *Substances Alimentaires*, p. 371.



epidermis, *a*, under which is a thin layer of brown parenchyma, *c*, while below this layer is shown the most characteristic portion of the pepper shell, viz.: the thickened, colored, stone cells, *b*. These are as a rule inclined to be rectangular rather than rounded. At *d* is shown a bit of the colorless parenchyma of the fruit itself.

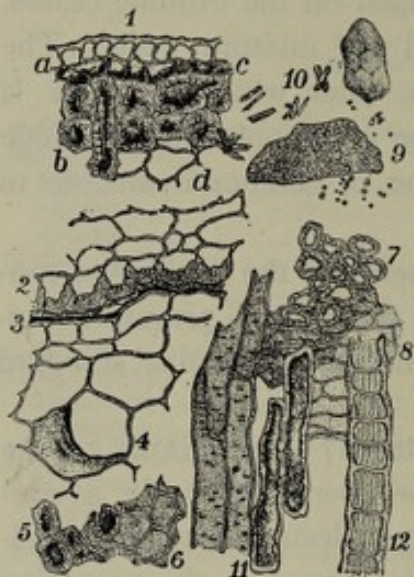


FIG. 85.—Powdered Black Pepper under the Microscope.  $\times 125$ . (After Moeller.)

(2), (3), and (4) show a cross-section of the outer part of the berry, (2) representing the inner stone-cell layer, a single row of horseshoe-like cells, (3) the thin seed coat, and (4) the white perisperm, with its large cells. Here and there through the perisperm certain yellow contents are visible, consisting largely of resinous matter. A dark resin cell is shown at (4). The ethereal oil, starch, and piperin are found in this part of the berry.

(5) shows in surface view the mostly rectangular stone cells of the pepper shell, resting upon the epidermis (6). Groups of stone cells are frequently thus found with portions of the epidermis.

The inner rounded, or cup-shaped cells are shown in plan view at (7) and the seed skin at (8), masses of starch and separate starch granules are shown at (9), and crystals of piperin at (10).

The bast-parenchyma of the pepper stem is shown at (11), pieces of which are commonly found in powdered pepper, and (12) shows a fragment of one of the many-celled hairs which grow on the stem.

The rounded cup cells (7) are readily distinguished from the more rectangular stone cells (5). The walls of the cup cells are nearly always colorless, and the cells themselves empty.\*

A water-mounted specimen of finely ground, black pepper, when viewed microscopically, shows most of the elements above described, at least in fragmentary form, though, in the case of the coarser particles,

\* The harder portions of the pepper, especially of the shell, are best examined by soaking for at least twenty-four hours in chloral hydrate, and mounting in this reagent on the slide.



by no means as clearly as by the use of chloral hydrate. Large polygonal masses of starch appear grouped as photographed in Fig. 256, Pl. XXXIV, if not rubbed out too fine under the cover-glass. Starch, indeed, is the most conspicuous element of pepper, being distributed more or less evenly throughout the mass. The powder may, however, be so finely reduced by abrasion under the cover-glass as to break up these starch masses wholly or in part, so that the granules may appear in much smaller groups or even singly. Fig. 255 shows such a field under a higher magnification. The individual granules of pepper starch average 0.003 mm. in diameter.

Besides the starch, and next to it the most numerous, one finds in the water-mounted black-pepper specimen many of the dark-yellow, thick-walled stone cells, patches of the colored parenchyma, and epidermis of the shell. Other elements of the perisperm, besides the starch, are seen in fragments, such as bits of resin, small droplets of oil, pieces of stems, and occasionally the needle-shaped crystals of piperin. Some of the rounded, cup-shaped cells are also usually found.

White pepper contains, of course, the same elements, but without the deeply colored stone cells and other characteristics of the shell, which has been removed from it.

**U. S. Standards for Pepper.**—The following limits of constituents have been adopted: For white pepper, non-volatile ether extract should not be less than 7%; starch should not be less than 52%; total ash should not be more than 3.5%; ash insoluble in hydrochloric acid should not exceed 0.3%; crude fiber should not exceed 5%. For black pepper, non-volatile ether extract should not be less than 6.75%; starch should not be less than 30%; total ash should not exceed 7%; ash insoluble in hydrochloric acid should not exceed 1.5%.

**Adulterants of Pepper.**—*Pepper shells* obtained in preparing white pepper, are not infrequently ground and added to the cheaper grades of black pepper. When a sample of black pepper is shown by the microscope to contain more shells in proportion to the other elements than could be possible in a ground whole berry, added shells are indicated. The analyst should, for comparison, grind in a mortar single berries of various grades, and familiarize himself with the appearance of the ground powder under the microscope, when the maximum amount of shells possible under natural conditions are present, noting especially the apparent number of stone cells of the outer coating. The familiar title of P. D. (pepper dust) originally given to ground pepper shells, stems, and "sweep-



ings" is now applied in the trade not only to almost any cheap and appropriate material for admixture with pepper, but also, in a broader sense, to ground powder suitable as an adulterant for any spice.

The presence of pepper shells is indicated by an excess of ash, sand, and crude fiber, and a deficiency of starch.

Hilger and Bauer, also Hanus and Bien, advocate the determination of pentosans as a means of detecting pepper shells.

*Ground Olive-stones* constitute one of the most commonly found foreign materials used as an adulterant of pepper. The powder, sometimes called "poivrette," is very like white pepper in appearance, is wholly inert in taste, and thus forms an admirable adulterant. While best detected by their characteristic appearance under the microscope, their presence may be shown by various color tests, although these do not differentiate olive stones from nutshells and similar woody materials.

Pabst has adopted for this purpose a test first suggested by Wurster for the detection of wood pulp in paper. The reagent is prepared as follows: In a porcelain capsule 10 grams of commercial dimethyl anilin are mixed with 20 grams of pure concentrated hydrochloric acid, and at least 100 grams of cracked ice are added. Then, while stirring, a solution of 8 grams of nitrite of soda in 100 cc. of water are added little by little, and the mixture allowed to remain for half an hour, after which 30 or 40 cc. of hydrochloric acid are added, and 20 grams of tin-foil. The reduction is allowed to go on for half an hour, heating on the water-bath, if necessary. The tin is then precipitated by granulated zinc, the liquid is filtered, and the filtrate neutralized with carbonate of potassium or sodium to the point of forming a precipitate, the precipitate being dissolved by a few drops of acetic acid. Finally the volume is made up with water to 2 liters, adding, before doing so, 3 or 4 cc. of a concentrated solution of sodium bisulphite, to prevent oxidation. The reagent thus prepared will keep for several years in a brown, tightly stoppered bottle.

If a pinch of pepper, which contains ground olive stones, be heated gently with a little of the above reagent in a test-tube, the stone cells of the adulterant will be colored a bright red brown, and the colored particles will be seen to settle to the bottom of the tube, after shaking, more quickly than the rest of the powder. Or, if the whole is poured from the test-tube into a porcelain dish, the color is more marked. Pure pepper is not colored under this treatment with the reagent.

Jumeau uses for a color reagent 5 grams of iodine in 100 cc. of a mixture of equal parts of ether and alcohol. Enough of the finely ground pepper to be examined is placed in a porcelain capsule to cover the



bottom of the dish, and sufficient iodine reagent is added to wet the entire mass, carefully avoiding excess. The thick paste is first mixed till homogeneous, and then allowed to dry in the air, after which it is broken up by a pestle, and the powder examined, either under the microscope, or by the naked eye. With pure pepper, a more or less deep-brown color is produced uniformly through the powder, but if olive stones are present, particles of these are colored yellow. With the naked eye as small an admixture as 2% of olive stones can thus be detected.

A solution of anilin acetate colors olive stones yellowish brown, while pure pepper appears grayish, or white.

Under the microscope olive stones are readily apparent, since the stone cells differ in size, form, and mode of grouping from those of pepper. Fig. 263, Pl. XXXVI, is a photograph of a water-mounted specimen of olive stones. They are for the most part entirely devoid of color, being long and narrow. In shape and manner of grouping they much resemble cocoanut shells (p. 433), but are distinguished from the latter from their lack of color.

Fig. 261 shows under low magnification a sample of pepper, bought on the market in Massachusetts, highly adulterated with olive stones. A large mass of the stone cells of the adulterant appears in the center of the field. Many of the stone cells are shown arranged end to end, so that what at first sight appear to be single, very long cells are in reality made up of several shorter ones. In ground olive stones one frequently finds, besides the stone cells, bits of the outer tegument of the seed, showing large cells with sinuous, rather thick walls; also bits of parenchyma, crossed frequently by fibro-vascular duct bundles.

*Buckwheat Products.*—Both the hulls and the middlings have been added to black pepper, and the middlings to white pepper. The starch of buckwheat possesses the added advantage, from the point of view of the spice-grinder, that it somewhat resembles pepper starch in microscopical appearance, not only in the shape of the starch granules, but also in the manner of grouping into masses. Compare Figs. 128 and 129, Plates II and III, showing buckwheat starch, with Figs. 255 and 256, Pl. XXXIV, respectively, showing pepper starch made under similar conditions of magnification, etc. The starch granules and masses are coarser in the case of buckwheat than of pepper.

Fig. 260, Pl. XXXV, shows a photograph of a pepper sample adulterated with buckwheat, masses of both starches appearing in the same field.

*Other Adulterants* found in Massachusetts samples of pepper have been wheat and corn products, nutshells, cayenne, charcoal, turmeric, rice,



sand, and sawdust. Charred cocoanut shells were at one time extensively used (see pp. 433 and 434).

**Long Pepper**, according to English analysts, has been used to a considerable extent as an adulterant. This is the fruit of the *Piper longum* and *P. officinarum*, both oriental species. The fruit, as its name implies, is long and cylindrical, while of about the same diameter as the spherical true peppercorns. Long pepper contains, as a rule, less than half the amount of piperin that true pepper does, and rather more starch than black pepper. Its taste is much less pungent than that of true pepper.

From its method of growth, long pepper is found with considerable dirt and sand adhering to the outer surface of the dried grains. This is due to the fact that the fruit often trails on the ground, and in gathering it the natives are not particular about removing the adhering soil. The surface of the fruit grains being very rough and irregular, much of the dirt remains dried thereon. The presence of long pepper thus materially increases the ash.

Long pepper possesses a very disagreeable, but peculiar odor, developed more especially when slightly warmed. For this reason, if for no other, it is not an ideal adulterant, since pepper containing it would not be palatable with warm food. At the present time it costs more than black pepper, and is used chiefly in mixed whole spices for pickles.

Brown gives the following analyses of samples of long pepper:

	Total Ash.	Sand and Ash Insoluble in Hydrochloric Acid.	Starch and Matters Convertible into Sugar.	Albuminous Matter Soluble in Alkali.	Cellulose.	Alcoholic Extract.	Ether Extract.	Total Nitrogen.
1	8.91	1.2	44.04	15.47	15.7	7.7	5.5	2.1
2	8.98	1.1	49.34	17.42	10.5	7.6	4.9	2.0
3	9.61	1.5	44.61	15.51	10.37	10.5	8.6	2.3

According to Brown and Heisch, the granules of long pepper starch under the microscope are larger than those of true pepper, and more angular. Stokes,\* however, finds no such marked difference in the size of starch granules and his experience is shared by the writer. When the two specimens (long and true pepper) are viewed side by side in water mounts under the microscope, the average size of the long pepper-starch grains is a trifle larger than those of true pepper, though, unless compared directly, the difference is not readily apparent. Stokes suggests a method of distinguishing the two by polarized light. With crossed

\* Analyst 13, p. 109.



Nicols, so that a dark field is given, and with the specimen mounted in glycerin, true pepper starch shows an evenly dark appearance, using a low power, while with long pepper a "ghostly white" image is shown. Long pepper, when present in true pepper powder, may generally be rendered apparent by the development of the characteristic odor on heating. Bits of fluffy fiber from the catkin of the long pepper will always be found in the ground powder, and will be apparent under the magnifying-glass.

Microscopic examination of the crude fiber discloses the highly characteristic, large, beaded cells of the endocarp, also elements of the spindle.

### RED PEPPER.

**Nature and Varieties.**—According to the U. S. Standards red pepper is the red, dried, ripe fruit of any species of *Capsicum*, a genus of the nightshade family (*Solanaceæ*), indigenous to the American tropics, but now cultivated in nearly all warm and temperate countries, and is of two distinct kinds: cayenne pepper or cayenne, the dried ripe fruit of *C. frutescens*, *C. baccatum*, or some other small fruited species of *Capsicum*, and paprika, the dried ripe fruit of *C. annuum*, or some other large-fruited species of the genus, excluding seeds and stems.

Boyles \* states that in the trade, the larger podded varieties are usually called capsicums and the smaller, chillies, the term cayenne being applied only to the ground product made from either or both capsicum and chillies.

*Chillies* are characterized by their extreme pungency and the small size of the pods, which seldom exceed 2 cm. in length. The leading commercial varieties come from Africa and Japan, the latter being the more brilliant in color. Zanzibar chillies, formerly the leading African variety, have given place in the market to Mombassa.

*Capsicums* formerly denoted low grade peppers of a brown color with pods 2 to 3 cm. in length, or even longer, produced in Africa, especially in the vicinity of the river Niger, also in Japan, Korea, and India, but now is used in a broader sense as noted above.

*Paprika* is a variety of *C. annuum* grown in Hungary. The powder is of a deep red color and has a sweetish, mildly pungent flavor.

Tolman and Mitchell † state that of the five grades of ground Hungarian paprika, named by Csonka and Varadi,‡ only three enter the

\* Jour. Ind. End. Chem., 9, 1917, p. 301.

† Ibid., 5, 1913, p. 747.

‡ Der Szegeder Paprika und der Szegeder Paprikahandel, 1907.



United States: *Rosenpaprika*, prepared from selected pods with removal of placentæ, stalks, and stems, *Königspaprika*, ground whole with the stems, and *Merkantilpaprika*, produced from spotted pods separated from better grades without removing stalks, stems, and other waste.

*Pimiento* is a large-fruited pepper, a variety of *C. annuum*, grown in Spain. This has come to be known as Spanish paprika, which leads to its sale under the simple name paprika, thus eliminating the distinction from true paprika. The succulent pericarp is much used for stuffing olives, while the dried pod is ground as a spice, often being substituted for the more valuable Hungarian varieties. Its coloring properties are more pronounced than its flavor.

The kitchen garden peppers, of which over thirty varieties are cultivated in the United States, also belong to the species *C. annuum*.

The species of *Capsicum* have solitary flowers, with a five-cleft corolla, and the fruit is of an elongated, conical form. The surface of the fresh fruit is smooth and bright red or yellow, but it loses in brilliancy on drying, and becomes shriveled. The pericarp is thin and tough, and at its base is a five-lobed calyx, greenish brown in color, terminating in a thick stem. The fruit proper is divided into two or three cells, which are separate and distinct at the lower portion, but which unite and form one at the top. The cells inclose a large number of yellow, wrinkled, kidney-shaped seeds, containing a fleshy endosperm, and a curved embryo.

**Constituents.**—Red peppers contain a fixed, bland oil, found in both pod and seed, but more abundantly in the latter, considerable resinous and mucilaginous material, a red coloring matter confined to the pod, and the active principle capsaicin, a crystalline alkaloid, to which much of the pungency is due.

*Capsaicin* is present in both seeds and pods, but is more abundant in the latter, particularly in the placentæ, being dissolved in the oil. Thresh\* first obtained the substance in an impure form, but Micko† prepared the pure substance and determined its formula,  $C_{18}H_{28}NO_3$ . According to Micko it forms white glistening plates, very soluble in alcohol, ether, chloroform, and benzol, less soluble in petroleum ether, difficultly soluble in hot, and insoluble in cold water; it is a weakly acid phenol-like substance with an intensely biting taste, a drop of a weak alkaline solution containing only 0.00005 mg. when placed on the tongue giving a burning

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\* Pharm. Jour. Trans., Nos. 315, 337, 376; Jahrb. Pharmakog., 1877.

† Zeits. Unters. Nahr. Genussm., 1, 1898, p. 818.



sensation lasting some time. Nelson \* has corroborated Micko's findings and developed a test for capsaicin.

*The Red Coloring Matter* is soluble in ether, petroleum ether, carbon bisulphide, and chloroform, but sparingly soluble in alcohol.

**Composition of Red Pepper.**—Winton, Ogden, and Mitchell † analyzed eight samples of whole chillies, representing three varieties, namely Zanzibar, Japan, and Bombay, the summarized results being as follows:

	Moisture.	Ash.			Ether Extract.	
		Total.	Soluble in Water.	Insoluble in HCl.	Volatile.	Non-volatile.
Maximum.....	7.08	5.96	4.93	0.23	2.57	21.81
Minimum.....	3.67	5.08	3.30	0.05	0.73	17.17
Average.....	5.73	5.43	3.98	0.15	1.35	20.15

	Alcohol Extract.	Reducing Matters as Starch. Acid Conversion.	Starch by Diastase Method.	Crude Fiber.	Nitrogen, $\times 6.25$ .	Total Nitrogen.
Maximum.....	27.61	9.31	1.46	24.91	14.63	2.34
Minimum.....	21.52	7.15	0.80	20.35	13.31	2.13
Average.....	24.35	8.47	1.01	22.35	13.67	2.18

The percentages of "starch by the diastase method" given in the above table represent errors of the process as neither cayenne nor paprika contain an appreciable amount of starch.

Doolittle and Ogden ‡ have made exhaustive analyses of known samples of Hungarian and Spanish red pepper, including determinations of non-volatile ether extract, and iodine number of this extract, which are of especial value in detecting added oil. A summary of their results is given on page 456.

Tolman and Mitchell § have analyzed samples of whole chillies, paprika, and pimiento obtained through official channels with results given on p. 457. They found that by sifting the total ash of African and Japan chillies could be lowered to a maximum of 6.16% and 5.82% and the sand to 0.85% and 0.53% respectively. In their examination of paprika

\* Jour. Ind. Eng. Chem., 2, 1910, p. 419.

† Ann. Rep. Conn. Exp. Sta., 1898, pp. 200-201.

‡ Jour. Am. Chem. Soc., 30, 1908, p. 1481.

§ Loc. cit.



ANALYSES OF HUNGARIAN AND SPANISH RED PEPPER (PAPRIKA AND PIMIENTO).

	Number of Samples.	Loss at 100° C.	Ash.			Alkalinity of Ash.*		Ether Extract.		Iodine No. of Non-volatile Ether Extract.	Reducing Matters by Acid Conversion.	Crude Fiber.	Nitrogen, X 6.25.
			Total.	Soluble in Water.	Insoluble in HCl.	Total.	Water-soluble.	Volatile.	Non-volatile.				
Whole Pods:	6	9.39	7.06	5.68	0.22	8.00	5.60	1.25	11.99	136.8	21.40	19.83	17.44
Hungarian: Maximum.....		7.26	5.63	4.67	0.05	6.22	4.07	0.17	7.42	130.3	19.56	15.10	14.06
Minimum.....		8.54	6.28	5.12	0.10	7.05	4.94	0.85	9.30	133.0	20.44	15.31	15.43
Average.....		8.58	6.79	5.79	0.06	7.90	5.70	1.18	10.39	136.1	16.52	15.37	16.87
Spanish: Maximum.....	2	8.28	5.24	4.59	0.05	6.10	4.10	1.18	10.39	134.5	16.52	15.37	14.62
Minimum.....		8.43	6.02	5.19	0.05	7.00	4.90	1.18	10.39	135.3	16.52	15.37	15.75
Average.....													
Pods (Seeds, placentae and stems removed):													
Hungarian: Maximum.....	7	10.86	6.90	6.10	0.08	8.60	6.10	1.10	6.69	138.0	24.52	23.61	15.37
Minimum.....		9.45	5.50	4.85	0.03	5.90	4.85	0.44	4.01	127.7	22.16	16.66	12.50
Average.....		10.37	6.03	5.44	0.05	6.92	5.53	0.80	5.08	133.1	23.90	19.50	16.33
Spanish: Maximum.....	3	10.35	7.68	6.68	0.20	8.90	6.90	1.40	4.76	133.0	19.96	15.19	14.06
Minimum.....		6.26	6.23	5.70	0.05	7.65	5.70	0.51	4.48	130.0	19.96	14.80	11.64
Average.....		7.69	7.17	6.22	0.11	8.45	6.35	0.95	4.62	131.0	19.96	15.00	13.05
Seeds and Placentae:													
Hungarian: Maximum.....	7	6.46	4.93	3.72	0.09	6.80	3.80	1.90	22.34	135.4	18.16	20.11	21.19
Minimum.....		5.00	3.06	1.72	0.05	3.40	1.50	0.95	17.66	130.2	17.36	17.29	16.56
Average.....		5.80	3.90	2.85	0.07	4.85	2.71	1.50	20.31	133.3	17.68	18.74	18.66
Spanish: Maximum.....	3	6.58	5.20	4.35	0.11	4.40	3.00	2.25	19.80	133.3	16.12	24.01	16.25
Minimum.....		5.19	3.41	2.23	0.04	3.90	1.80	1.56	18.99	130.7	16.12	19.48	15.50
Average.....		5.74	4.41	3.37	0.06	4.17	2.33	1.91	19.40	132.1	16.12	21.74	15.92
Stems:													
Hungarian, Average.....	7	6.55	11.32	8.36	0.44	14.07	8.46	0.48	1.94	15.88	.....	24.47	15.87
Spanish, Average.....	2	4.73	15.50	13.09	0.26	14.10	11.00	0.29	0.98	.....	.....	29.99	11.56

\* Cc.  $\frac{N}{10}$  HCl per gram of material, using methyl orange as indicator.



and pimiento they determined the percentage of the different parts as follows:

	Paprika (21 Samples).			Pimiento (18 Samples).		
	Shells.	Seeds and Placentæ.	Stems.	Shells.	Seeds and Placentæ.	Stems.
Maximum.....	63.7	43.2	9.4	58.1	37.4	10.9
Minimum.....	50.5	28.1	6.0	53.1	34.9	6.0
Average.....	56.4	36.1	7.5	55.3	36.0	8.7

Analyses were reported of each of the above separately as well as of the whole product and of the whole product less the stems as follows:

COMPOSITION OF RED PEPPER (TOLMAN AND MITCHELL).

	Number of Analyses.	Loss at 70° C., in vacuo.	Ash.			Ether Extract (Cont. Ext.).		Non-vol. Ether Ext. (Shaking Method).			Fiber.
			Total.	Insol. in HCl, (Sand).	Sand-free.	Volatile.	Non-volatile.	Per Cent.	Iodine No.	Refractive Index, 40° C.	
Mombassa Chillies.	26										
Maximum.....			8.41	3.03	6.41	1.72	19.00	.....	.....	.....	28.70
Minimum.....			5.34	0.44	4.73	0.28	15.88	.....	.....	.....	24.98
Average.....			6.31	1.24	5.07	0.81	17.26	.....	.....	.....	26.86
Japan Chillies.....	17										
Maximum.....			6.20	1.07	5.50	1.59	23.21	.....	.....	.....	25.96
Minimum.....			5.08	0.31	4.52	0.09	17.10	.....	.....	.....	22.82
Average.....			5.52	0.53	4.99	0.56	19.94	.....	.....	.....	24.25
Cherry Chillies.....	1		6.62	1.23	5.39	1.18	16.17	.....	.....	.....	26.20
Hungarian Paprika											
With stems.....	7										
Maximum.....		3.76	6.03	0.33	5.73	0.89	16.43	15.00	134.0	1.4854	22.76
Minimum.....		3.29	5.08	0.24	4.82	0.08	12.21	10.86	129.8	1.4758	20.69
Average.....		3.47	5.63	0.28	5.36	0.42	14.04	12.61	132.6	1.4806	21.93
Without stems.....	7										
Maximum.....		4.16	5.56	0.31	5.25	0.90	17.35	15.08	133.2	1.4834	23.18
Minimum.....		3.11	4.66	0.20	4.41	0.07	13.94	12.64	129.0	1.4756	20.47
Average.....		3.51	5.22	0.26	4.96	0.34	15.28	13.91	131.9	1.4799	21.56
Spanish Pimiento:											
With stems.....	7										
Maximum.....		5.98	7.86	0.48	7.54	0.69	12.58	10.81	137.3	1.4818	20.59
Minimum.....		4.31	6.98	0.29	6.69	0.10	11.30	9.81	136.0	1.4776	19.53
Average.....		5.06	7.39	0.35	7.04	0.47	11.87	10.34	136.7	1.4805	20.13
Without stems.....	8										
Maximum.....		5.09	7.35	0.40	6.98	0.60	13.34	11.30	137.2	1.4810	20.34
Minimum.....		4.52	6.60	0.24	6.26	0.25	11.58	9.80	134.5	1.4792	18.76
Average.....		4.83	6.98	0.32	6.66	0.44	12.47	10.67	136.1	1.4801	19.49

Boyles' \* analyses of red peppers appear in the following table. South Carolina capsicums were grown under the indirect supervision of the Bureau of Plant Industry from Hungarian paprika seed, but in that climate be-

\* *Loc. cit.*



came so hot as to be classed, when ground, with cayenne. He believes that the United States standards for cayenne should be revised as follows: Total ash increased to 7.5%, ash insoluble in 10% hydrochloric acid (sand) to 1.0%, fiber to 20%, and non-volatile ether extract lowered to 14%.

## COMPOSITION OF RED PEPPER (BOYLES).

	Number of Analyses.	Ash.		Ether Extract.		Fiber.*
		Total.	Insol. in HCl (Sand).	Volatile.*	Non-vol.*	
Mombassa Chillies..	7	.....	.....	(3)	(6)	(6)
Maximum.....		9.40	1.77	0.32	25.49	30.45
Minimum.....		4.36	0.35	0.15	15.75	22.63
Average.....		6.08	1.06	0.23	20.06	26.25
Japan Chillies.....	1	4.63	0.18	.....	22.50	24.02
So. Carolina Capsicums.....	17					
Maximum.....		7.75	1.20	1.85	15.70	30.48
Minimum.....		4.82	0.25	0.15	10.75	20.07
Average.....		5.98	0.78	0.60	13.92	25.48
Bombay Capsicums	35	.....	.....	(7)	(12)	(8)
Maximum.....		9.35	1.75	0.72	20.40	32.30
Minimum.....		5.56	0.14	0.25	12.35	25.00
Average.....		6.95	0.76	0.45	16.57	28.08
Japan Capsicums...	19	.....	.....	(3)	(5)	(5)
Maximum.....		6.84	1.17	0.40	17.03	26.64
Minimum.....		4.90	0.14	0.30	12.80	22.50
Average.....		6.05	0.39	0.35	15.56	23.84
Korean Capsicums..	4	.....	.....	(2)	(2)	(2)
Maximum.....		7.70	0.75	0.60	22.25	26.02
Minimum.....		6.20	0.20	0.45	19.77	25.85
Average.....		6.94	0.51	0.53	21.01	25.94
Niger Capsicums...	3	.....	.....	(2)		
Maximum.....		6.17	1.27	0.85	21.96	27.77
Minimum.....		5.27	0.60	0.25	18.22	22.82
Average.....		5.72	0.83	0.55	19.53	24.93
African Capsicums..	1	5.05	0.95	.....	19.45	28.76
Bombay Cherries...	2	.....	.....	(1)		
Maximum.....		5.67	0.82	0.30	17.55	29.20
Minimum.....		5.35	0.65	0.30	15.60	27.45
Average.....		5.51	0.74	0.30	16.57	28.33

\* Figures in parentheses are number of samples.

**Microscopical Structure of Red Pepper.**—Fig. 86, from Moeller, shows the appearance under the microscope of various elements of powdered red pepper. (1) is a sectional view through the outer portion of the fruit



shell or pod, showing the epidermis (*a*), and beneath this the collenchyma layer. The inner epidermis is shown at (2) with its cells thick-walled in places and inclosing brilliant, red oil drops of coloring matter. (3) represents the outer and (4) and (5) the inner epidermis in surface view. The outer epidermis of cayenne, which is the element of chief value in distinguishing this from paprika, is shown at (6).

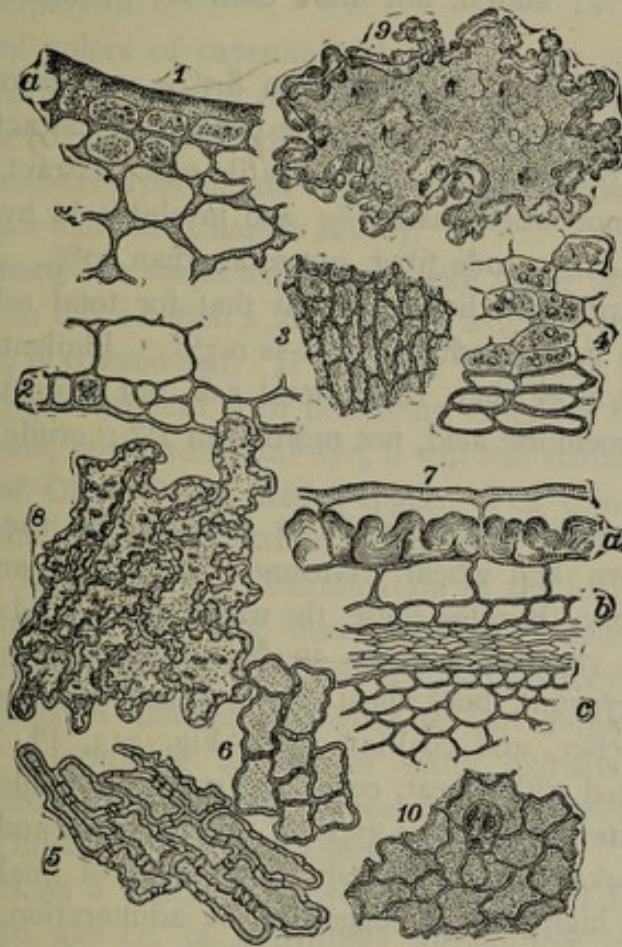


FIG. 86.—Powdered Red Pepper under the Microscope.  $\times 125$ . (After Moeller.)

A cross-section through the seed shell is shown at (7); *a* being the epidermis of the seed, *b* the parenchyma layer directly beneath, and *c* the tissues of the endosperm. (8) shows in surface view the peculiar seed epidermis, the appearance of which Moeller compares with that of intestines. At (9) is shown one of the isolated cells of this epidermis more highly magnified, while (10) shows the epidermis of the calyx.

Figs. 211 and 212, Pl. XXIII, show photomicrographs of powdered cayenne. In Fig. 211 is shown a large bit of the outer epidermis of the fruit pod, while in Fig. 212 appears a smaller portion of this same kind



of epidermis, and next to this the characteristic skin of the seed shell, with its striking markings suggestive of the convolutions of the intestines. Yellow or yellowish-red droplets of oily coloring matter are distributed through the field. Starch grains are absent.

**U. S. Standards.**—*Cayenne*: Non-volatile ether extract, not less than 15%; total ash, not more than 7%; ash insoluble in hydrochloric acid, not more than 1%; starch, not more than 1.5%; and crude fiber, not more than 28%.

*Paprika*: Total ash, not more than 8.5%; ash insoluble in hydrochloric acid not more than 1%; iodine number of extracted oil, between 125 and 136. *Rosenpaprika*: Non-volatile ether extract, not more than 18%; total ash not more than 6%; ash insoluble in hydrochloric acid, not more than 0.4%; crude fiber, not more than 23%. *Königspaprika*: same as rosenpaprika in limits, except that for total ash is 6.5%, and for ash insoluble in hydrochloric acid is 0.5%. *Pimiento*: non-volatile ether extract, not more than 18%; total ash, not more than 8.5%; ash insoluble in hydrochloric acid, not more than 1%; crude fiber, not more than 21%.

The most common adulterants of cayenne are the starches of the cereal grains, corn and wheat. Ground pilot bread and crackers are especially common. Besides these the writer has found in the routine examination of cayenne samples in Massachusetts, ginger, nutshells, turmeric, rice, gypsum, buckwheat, olive stones, mustard hulls, ground redwood, red ocher, and coal-tar dyes. Fig. 213, Pl. XXIV, shows a sample adulterated with wheat, corn, and cocoanut shells.

**Mineral Adulterants**, such as gypsum, and red ocher and other pigments, are all to be looked for in the ash by methods of qualitative analysis. An abnormally high ash is suggestive of adulteration. According to Vedrodi, the ash of genuine cayenne should not exceed 5.96. The presence of red ocher is rendered apparent by the high content of iron.

Salts of lead and mercury are rarely if ever now used for color.

**Ground Redwood.**—Numerous varieties of redwood are commonly used to intensify the color of cayenne, especially when otherwise highly adulterated with colorless materials, such as the starches. The redwood is sometimes used alone, and sometimes in mixture with turmeric. Both redwood and turmeric are readily recognized under the microscope.

Fig. 214, Pl. XXIV, shows a cayenne sample adulterated with corn starch and red sandalwood, a mass of the latter filling the center of the field. The wood fibers of the dyestuff, even when finely ground, are very striking under the microscope, showing a brick-red color.



**Detection of Coal-tar and Vegetable Colors.**—Oil-soluble coal-tar and vegetable colors may be tested for in cayenne and paprika by an adaptation of Martin's butter-color method, shaking the ether extract of the sample with the alcohol and carbon bisulphide mixture, page 557. The carbon bisulphide dissolves the oil and natural color, while the overlying alcohol layer holds in solution many of the artificial coloring matters that may be employed.

The natural colors of cayenne and paprika are sparingly soluble in alcohol, but readily soluble in carbon bisulphide. The separated alcohol is examined for colors by methods given elsewhere.

Tests for coal-tar dyes should also be made by Sostegni and Carpentieri's, or Arata's method (Chapter XVII).

Szigeti\* treats the suspected sample with water acidified with acetic acid, and boils in this solution a bit of wool, which, if carotin or a coal-tar dye be present, is colored red. If the color is carotin, it will be removed from the wool by treatment with petroleum ether, or by heating at 100° C. for some hours, but if a coal-tar dye, it will still remain fixed thereon.

**Detection of Olive Oil in Red Pepper.**—The color of paprika and pimiento is sometimes intensified by grinding with olive oil. This form of adulteration is detected by determination of the iodine number of the non-volatile ether extract. The usual method of determining the non-volatile ether extract having been found unsatisfactory for the purpose, Winton† suggested a method analogous to that employed by him and co-workers in determining alcohol in water extracts. The following details of the process, elaborated by Seeker,‡ have been adopted by the Association of Official Agricultural Chemists:

Dry 5 grams on a watch-glass over sulphuric acid for at least twelve hours. Measure 250 cc. of anhydrous alcohol-free ether into a dry graduated flask with the mark near the lower end of the neck, and brush the paprika into it. Place a mark on the neck of the flask at the meniscus, and allow to stand for one hour, shaking at twenty-minute intervals during that time. Bring the meniscus back to the mark either by cooling if the level has risen, or by adding absolute ether if it has fallen, and let settle. Pipette off 100 cc. of the supernatant liquid, filter through an 11-cm. close-textured paper into a tared, air-dry glass-stoppered 250-cc. Erlenmeyer flask previously counterpoised against a similar flask, wash

\* Zeits. landw. Versuchs. Oesterreich, 5, 1902, pp. 1208, 1222.

† U. S. Dept. of Agric., Br. of Chem., Bul. 122, 1909, p. 38.

‡ Ibid., Bul. 132, 1910, p. 114; 137, 1911, p. 81.



with a little absolute ether, and distil off the solvent until the ether ceases to come over. Lay the flask on its side in a water-oven, heat for thirty minutes, cool the open flask for at least thirty minutes in the air and weigh. Repeat this heating and weighing until the weight is constant to within 1 milligram, two heatings usually being sufficient, and calculate the per cent of ether extract. If more than  $1\frac{1}{2}$  hours' heating is required to obtain constant weight or if the ether extract becomes colorless it should be rejected, and a new determination started with freshly purified ether.

Dissolve the ether extract in the flask in 10 cc. of chloroform, add 30 cc. of Hanus solution, and proceed as described for the Hanus method. The iodine number thus determined should not be less than 125.

#### GINGER.

**Nature and Composition.**—Ginger as a spice is the ground root-stock of the *Zingiber officinale*, an annual herb of the family *Zingiberaceæ*, growing to a height of from 3 to 4 feet. It is a native of India and China, but is cultivated quite extensively in tropical America, Africa, and Australia.

The root is dug when the plant is a year old, and when the stem has withered. If the root, when freshly dug and scalded to prevent sprouting, is dried at once, it forms the so-called black ginger, of which Calcutta and African are the common varieties. When decorticated, the product is known in commerce as white ginger, the chief varieties being Jamaica, Cochin, and Japan. The best variety is Jamaica ginger. The scraped root is sometimes bleached to make it still whiter, or sprinkled with carbonate of lime.

In commerce whole or black ginger appears in "hands" 4 to 10 cm. long, and from 10 to 15 mm. in diameter. These usually have three or four various-sized, irregular branches, some short and thick, others elongated. The epidermis is gray or yellowish gray in color, more or less wrinkled, and beneath it is a reddish-brown layer. The inner portion of the dried root is white or yellowish. The root is hard, and of a compact, horny structure.

White or decorticated ginger appears in "hands" of smaller diameter than the black, and yields a lighter colored powder on grinding. Preserved ginger root is prepared by boiling the root in water, and curing with sugar or honey. Much of the preserved ginger comes from Canton.



The distinguishing features of ginger are its large content of starch, its volatile oil, and its resinous matter. Inasmuch as the epidermis contains a large amount of pungent resin, it is easy to see how the peeled or decorticated variety is inferior.

Oil of ginger is very aromatic, and of a greenish-yellow color. Its specific gravity ranges from 0.875 to 0.885. It is slightly soluble in alcohol. Of its composition little is known.

Richardson's analyses in full of five samples of whole ginger-root are as follows:

	Water.	Ash.	Volatile Oil.	Fixed Oil and Resin.	Starch.	Crude Fiber.	Albuminoids.	Undetermined.	Nitrogen.
Calcutta .....	9.60	7.02	2.27	4.58	49.34	7.45	6.30	13.44	1.01
Cochin .....	9.41	3.39	1.84	4.07	53.33	2.05	7.00	18.91	1.12
Unbleached Jamaica .....	10.49	3.44	2.03	2.29	50.58	4.74	10.85	15.58	1.74
Bleached Jamaica, London. ....	11.00	4.54	1.89	3.04	49.34	1.70	9.28	19.21	1.48
" " American .....	10.11	5.58	2.54	2.69	50.67	7.65	9.10	11.66	1.46

Summaries of Winton, Ogden, and Mitchell's analyses of eighteen samples of whole ginger, representing the common white and black varieties, as well as of two samples of exhausted ginger, are as follows:

	Moisture.	Ash.			Lime (CaO).	Ether Extract.	
		Total.	Soluble in Water.	Insoluble in HCl.		Volatile.	Non-volatile.
Ginger: Maximum .....	11.72	9.35	4.09	2.29	3.53	3.09	5.42
Minimum .....	8.71	3.61	1.73	0.02	0.20	0.96	2.82
Average .....	10.44	5.27	2.71	0.44	0.80	1.97	4.10
Exhausted ginger from English ginger-ale works .....	10.61	2.12	0.59	0.18	.....	1.61	3.86
Exhausted ginger from extract works..	8.02	5.05	3.55	1.50	.....	0.13	0.54

	Alcohol Extract.	Reducing Matters by Acid Conversion, as Starch.	Starch by Diastase Method.	Crude Fiber.	Nitrogen, $\times 6.25$ .	Cold-water Extract.	Total Nitrogen.
Ginger: Maximum .....	6.58	62.42	60.31	5.50	9.75	17.55	1.55
Minimum .....	3.63	53.43	49.05	2.37	4.81	10.92	0.77
Average .....	5.18	57.45	54.53	3.91	7.74	13.42	1.23
Exhausted ginger from English ginger-ale works .....	4.88	59.86	54.57	5.17	6.94	6.15	1.11
Exhausted ginger from extract works.	1.52	.....	.....	.....	.....	16.42	.....



McGill \* records the analyses of ninety-eight samples of ground ginger as sold in the Canadian market. Of thirty-two of these, pronounced pure on analyses, the following is a summary:

	Moisture or Loss on Drying at 100°.	Petroleum-ether Extract.	Cold-water Extract.	Ash.			
				Total.	Soluble.	Insoluble.	Alkalinity of Soluble Ash as K <sub>2</sub> O.
Maximum.....	12.00	6.13	15.48	7.84	3.15	3.99	.133
Minimum.....	9.50	2.78	14.04	3.67	2.28	1.96	.103

According to Vogl, the proportion of ginger ash varies quite widely according to the kind, but should never exceed 8%.

**Exhausted Ginger and Methods of Detection.**—There are two kinds of exhausted ginger commercially available for admixture with ground spice, as an adulterant. One is the product left after extraction with strong alcohol in the making of extract of Jamaica ginger, and the other the residue from extraction with either very dilute alcohol, or with water, in the manufacture of ginger ale. Ground, exhausted ginger is rarely substituted wholly for the pure variety, since, from its lack of pungency, the sophistication would be too apparent. It is rather used to mix with the latter in varying proportions, and as an adulterant of other spices.

Ginger that has been exhausted by extraction with alcohol has been deprived of most of its volatile oil, which is found in the "extract," while for the manufacture of ginger ale, a water extract, or at most a very dilute alcoholic extract is best adapted. Such a water extract does, as a matter of fact, remove much of the valued pungency, so that the residue, or exhausted ginger, is rather inert.

Either the alcohol- or the water-extracted variety of exhausted ginger, when present in considerable amount, would be apparent, one by the alcohol and ether extract, and the other by the abnormally low cold-water extract, and water-soluble ash.

*Dyer and Gilbard* † first called attention to the water-soluble ash as a reliable means of indicating exhausted ginger. Six samples of ginger of known purity were analyzed by them, their results being summarized as follows:

\* Dept. Int. Rev. Canada Bul. 48, pp. 10, 11.

† Analyst, 18, 1893, p. 197.



		Total Ash.	Water-soluble Ash.	Alcohol Extract, after Ether Extract.
Pure ginger (6 samples):	Highest .....	4.1	3.	3.8
	Lowest .....	3.1	1.9	2.1
	Average .....	3.8	2.7	2.8
Exhausted ginger (6 samples):	Highest .....	2.3	0.5	1.5
	Lowest .....	1.1	0.2	0.8
	Average .....	1.8	0.35	1.2

Allen and Moor\* pointed out the value of the cold-water extract as a help in detecting exhausted ginger, especially when taken in connection with the soluble ash, showing that the presence of this adulterant is assured, when the soluble ash is as low as 1% and the cold-water extract is less than 8%.

**Determination of Cold-water Extract.**—*Winton, Ogden, and Mitchell's Method.*†—Four grams of the ground sample are placed in a 200-cc. graduated flask, and the latter is filled to the mark with water, and shaken at half-hour intervals during eight hours, after which it is allowed to stand at rest for sixteen hours in addition. The contents are then filtered, and 50 cc. of the filtrate evaporated to dryness in a platinum dish. It is then dried at 100° to constant weight and weighed.

**Microscopical Structure of Ground Ginger.**—Fig. 87, from Moeller, shows elements of ginger root, from which the epidermis has not been removed. A bit of the large-celled cork (or dead protective tissue of the epidermis) is shown in surface view at (1); at (2) is shown in cross-section the parenchyma in which the starch is contained, *h* being an oil-cell; (3) shows the parenchyma in longitudinal section, with bast fibers. Fragments of spiral ducts are shown at (4), and starch grains at (5). (6) is a cross-section in the extreme interior of the root.

The most prominent feature of powdered ginger is the starch grains (5), which Moeller compares in shape to tied sacks.

Fig. 228, Pl. XXVII, is a photomicrograph of pure, ground ginger, mounted in water, showing the starch grains inclosed in the cells of the parenchyma. Fig. 231 shows the starch grains alone. The granules of ginger starch are ellipsoidal, and as a rule very clear and transparent, being for the most part entirely devoid of either hilum or concentric rings. Occasionally granules are to be found, however, with faint concentric

\* Analyst, 19, 1894, p. 194.

† Conn. Agric. Exp. Sta. Rep., 1898, 190.



markings, and even with an apparent hilum. The characteristic form of the ginger starch granule is more or less egg-shaped, with a small protuberance near one end. This protuberance serves to readily distinguish the starch granules of ginger from those of wheat, with which ginger

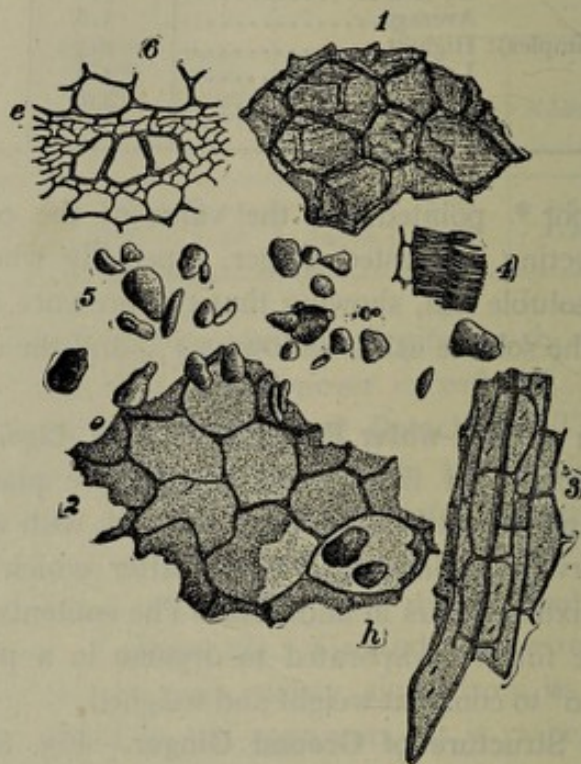


FIG. 87.—Powdered Ginger under the Microscope.  $\times 125$ . (After Moeller.)

is frequently adulterated. While wheat granules are of various sizes, the grains of ginger starch are as a rule much more uniform.

**U. S. Standards.**—*Ginger*: Starch, not less than 42%; crude fiber, not more than 8%; lime (CaO), not more than 1%; cold-water extract, not less than 12%; total ash, not more than 7%; ash insoluble in hydrochloric acid, not more than 2%; ash soluble in cold water, not less than 2%. *Jamaica ginger*: cold-water extract, not less than 15%, otherwise as for ginger. *Limed ginger*: calcium carbonate, not more than 4%; total ash, not more than 10%; otherwise as for ginger.

**Adulteration.**—Besides exhausted ginger, the common adulterants reported in powdered ginger are turmeric, wheat, corn, rice, and sawdust. Sawdust of soft wood was a not uncommon adulterant, and care should be taken to distinguish between the wood fiber natural to the ginger root, and that of the foreign variety. A careful study should be made of finely ground, soft-wood sawdust, with its long spindle cells and lateral



pores, as shown in Fig. 266, Pl. XXXVII, and the wood fiber of the genuine ginger root. A large admixture of sawdust would materially increase the percentage of crude fiber.

Fig. 234, Pl. XXIX, shows a sample of ginger adulterated with corn and wheat. Fig. 232 shows a mass of wheat bran in an adulterated sample.

Fig. 233 shows ginger adulterated with turmeric.\*

### TURMERIC.

**Nature and Composition.**—Turmeric, while largely used as an adulterant of other spices (especially of ginger and mustard), possesses some value as a condiment in itself, forming, for instance, the chief ingredient of curry powder.† Turmeric (*Curcuma longa*) belongs to the same family (*Zingiberaceæ*) as ginger, having a perennial rootstock, and an annual stem. It is a native of the East Indies and Cochin-China. Its chief ingredients are starch, a volatile oil, a yellow coloring matter (curcumin), cellulose, and gum.

Curcumin ( $C_{14}H_{14}O_4$ ) is insoluble in cold water, but readily soluble in alcohol. It is extracted from powdered turmeric by boiling the latter with water, filtering, and extracting the residue with boiling alcohol. The alcohol solution is filtered, evaporated, and the residue extracted with ether. The ether extract contains the curcumin, together with a small amount of volatile oil.

Curcuma oil is an orange-yellow, slightly fluorescent liquid, its specific gravity being 0.942.

The following analyses of turmeric were made in the writer's laboratory:

Variety.	Moisture.	Total Ash.	Ash Soluble in Water.	Ash Insoluble in HCl.	Total Nitrogen.	Protein. N $\times$ 6.25.	Total Ether Extract.
China. ....	9.03	6.72	5.20	0.11	1.73	10.81	10.86
Pubna. ....	9.08	8.52	6.14	....	0.97	6.06	12.01
Alleppi. ....	8.07	5.99	4.74	....	1.56	9.75	10.66
Average.....	8.73	7.07	5.36	....	1.42	8.88	11.17

\* This photomicrograph is very disappointing, in that it fails to show the intense yellow of the central mass of turmeric.

† Curry powder consists of a mixture of turmeric, cayenne, and various pungent spices.



Variety.	Volatile Ether Extract.	Non-volatile Ether Extract.	Alcohol Extract.	Crude Fiber.	Reducing Matter by Acid Conversion, as Starch.	Starch by Diastase Method.
China. ....	2.01	8.84	9.22	4.45	48.69	40.05
Pubna. ....	4.42	7.60	7.28	5.84	50.08	29.56
Alleppi. ....	3.16	7.51	4.37	5.83	50.44	33.03
Average.....	3.19	7.98	6.96	5.37	49.73	34.21

**Microscopical Structure of Turmeric.**—Moeller's representation of characteristics of powdered turmeric is reproduced in Fig. 88. The

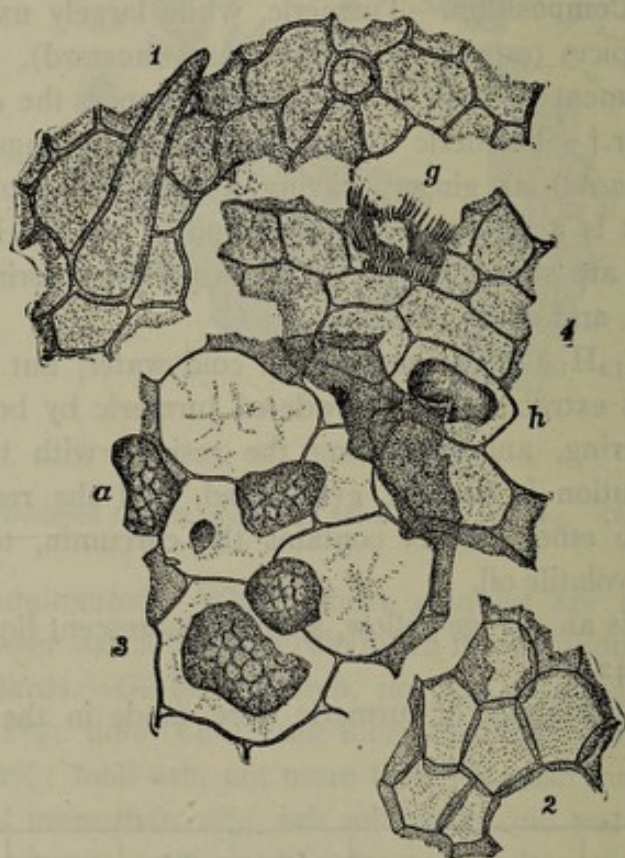


FIG. 88.—Powdered Turmeric under the Microscope.  $\times 125$ . (After Moeller.)

epidermis is shown at (1) with one of the numerous, one-celled hairs that grow from it, also the scar left after one of the hairs has been removed; (2) shows in plan view the cork immediately under the epidermis. The tender-celled parenchyma is shown in cross-section at (3), and in longitudinal section at (4). In some of the cells of the parenchyma are found dark-yellow lumps of resin (*h*), and vascular ducts (*g*), but by far the most numerous and striking contents of the parenchyma-cells are the bright-



yellow masses of "paste balls" (3a) and the starch granules, one of which is shown in (3). See also Plate XIII. The starch grains in the water-mounted powder show under the microscope in masses, usually of a deep-yellow color, unless very finely rubbed out, when they appear for the most part in fragments.

The whole starch granule appears somewhat in the form of a clam-shell, with very distinct markings. When fragments of the starch granules are carefully examined, these distinct markings are so strongly characteristic, even in the smallest pieces commonly found in the powdered sample, as to nearly always serve to identify them. See Fig. 171, Plate XIII.

**Turmeric as an Adulterant.**—Turmeric is a material especially adapted by its deep-yellow color to intensify mustard and ginger, especially when these spices are adulterated with the lighter-colored cereal starches, hence formerly it was used in these spices, both with and without other adulterants.

It was also frequently used in small quantities in adulterated cayenne, mace, and various spices, to counteract the colors of other dyestuffs, such as ground redwood, which in itself would sometimes be too intense if used alone.

#### MUSTARD.

**Nature and Composition.**—Mustard is the seed of the mustard plant, an annual belonging to the family *Cruciferae*, and to the genus *Sinapis*, or *Brassica*, as it is now generally known. The mustards include wild and cultivated species all with yellow flowers and lyrate leaves.

The common species are black, sometimes called brown, mustard (*B. nigra*), brown or Serepta mustard (*B. Besseriana*), white or yellow mustard, (*B. alba*), and Indian mustard (*B. juncea*). The seeds of charlock (*B. arvensis*), growing wild in the grain and flax fields of the Northwest, together with brown mustard, are separated from the grain by ingenious machines and constitute the so-called wild mustard of commerce.\*

The seeds of all varieties are globular, those of the black mustard being smaller than those of brown, and both smaller than those of white mustard. As seen under the lens the surface of black, brown, and Indian mustard is reticulated, while that of white mustard and charlock is smooth. Most of the seeds of charlock are of a deep black color.

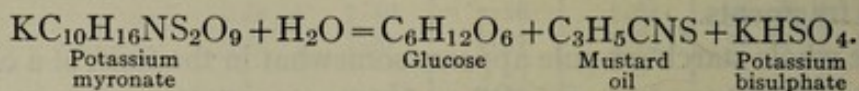
Both black and white mustard contain from 27 to 38% of fixed oil, a soluble ferment known as *myrosin*, and a sulphocyanate of sinapin. Mustard seeds contain no starch, and very little volatile oil as such. Black

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\* Jour. Ind. Eng. Chem., 7, 1915, p. 684.

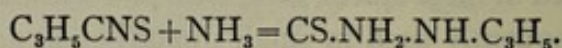


mustard seed contains *sinigrin*, or myronate of potash (not found in the white seed), which, when moistened with water, forms by hydrolysis the volatile oil of black mustard, otherwise known as allyl isothiocyanate, in accordance with the following equation:



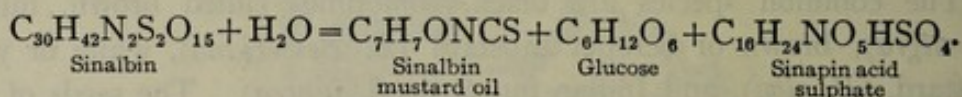
*Mustard Oil* (volatile) is a colorless, or slightly yellow, highly refractive liquid of a very strong odor, and capable of blistering the skin when brought in contact with it. It is optically inactive. Its specific gravity varies between 1.016 and 1.030. It boils between 148° and 156°. It turns reddish brown by exposure to light.

Volatile oil of black mustard forms thiosinamine with ammonia, as follows:



Thiosinamine is soluble in hot water, from which it crystallizes in tufts of monoclinic crystals, having a melting-point of 74° C. It is precipitated by silver nitrate, mercuric chloride, and Mayer's solution.

White mustard differs from the black in containing a sulphur compound, *sinalbin*,  $\text{C}_{30}\text{H}_{42}\text{N}_2\text{S}_2\text{O}_{15}$ . This is a glucoside. Sinalbin by hydrolysis forms an oil of white mustard, in a somewhat similar manner to the potassium myronate of black mustard, and according to the following equation:



*Sinalbin Mustard Oil* cannot be obtained by the distillation of white mustard, being sparingly volatile with steam.

Sinalbin mustard oil somewhat resembles that from black mustard, being quite as pungent, but less strong in odor when cold. It is soluble in dilute alkali.

Fixed oil of mustard is a bland, tasteless, and nearly odorless oil, its specific gravity at 15° varying between the limits of 0.914 to 0.918. It is said to be used to some extent as an adulterant of table oils, being separated by pressure from the crushed mustard seeds before the latter are ground into "flour." The chief use of mustard oil is in mixture with other oils as an illuminant.

**MUSTARD FLOUR.**—In the process of preparing the ground spice commonly known as mustard "flour," the seeds are first crushed and sepa-



rated by winnowing from the hulls, the latter being incapable of the fine grinding necessary to produce a smooth flour. The yellow hulls are, however, found in the cheaper grades of ground mustard, and both varieties of hull are frequently used in the wet mustard preparations, sold in bottled form. In order to produce an even, dry powder, free from lumps, it is necessary to remove a large portion of the fixed oil, which is indeed of no value in the final product, and this is done by subjecting the crushed material to hydraulic pressure, during which process the mustard is molded together into thin, hard plates, called "mustard cake." This is then broken up and reduced to fine powder by pounding.

Richardson's\* analyses of whole-seed flour, prepared by himself without the removal of the fixed oil, are as follows:

	Water.	Ash.	Volatile Oil.	Fixed Oil.	Starch.	Crude Fiber.	Albuminoids.	Undetermined.	Nitrogen.
White seed .....	5.57	4.29	.97	33.56	.00	5.40	28.88	21.33	4.62
White flour. ....	3.33	5.23	1.84	34.83	.00	9.05	25.56	20.16	4.09
Seed husk. ....	6.17	4.99	.55	28.12	.00	9.50	23.44	27.23	3.75
California yellow. ....	4.83	5.96	1.27	31.96	.00	8.50	31.13	16.35	4.98
California brown. ....	4.11	4.88	1.35	36.63	.00	16.18	24.69	12.16	3.95
English yellow. ....	3.11	4.07	2.06	31.51	.00	6.90	30.25	22.10	4.84
Trieste brown. ....	4.62	5.61	.63	39.55	.00	10.84	25.88	18.87	4.14

Winton and Mitchell made no full analyses of mustard seed of known purity, but the following is a summary of analyses of 18 samples of commercial mustards, sold in packages in Connecticut, and not found to be adulterated:

	Total Ash.	Ether Extract.		Reducing Matters by Acid Conversion, as Starch.	Starch by Diastase Method.	Crude Fiber.	Nitrogen $\times 6.25$ .
		Volatile.	Non-volatile.				
Maximum.....	7.35	1.90	28.10	6.12	2.08	4.87	43.56
Minimum.....	4.81	0.00	17.14	1.85	0.28	1.58	35.63
Average.....	5.99	0.56	20.61	4.33	1.07	2.58	39.57

The following analyses of 5 samples of mustard flour, 6 samples of mustard hulls, and 6 samples of whole mustard, were made in the author's laboratory in 1903:

\* U. S. Dept. of Agric., Div. of Chem., Bul. 13, part 2.



	Moisture.	Total Ash.	Water-soluble Ash.	Ash Insoluble in HCl.	Total Ether Extract.	Volatile Ether Extract.	Non-volatile Ether Extract.	Alcohol Extract.	Total Nitrogen.	Volatile Oil.	Crude Fiber.	Reducing Matters by Acid Conversion.	Reducing Matters by Diastase.	On Dry Fat-free Substance.					Reduced Material by Diastase.
														Alcohol Extract.	Total Nitrogen.	Total Ash.	Crude Fiber.	Reduced Material by Acid Conversion.	
Mustard "flour" as prepared commercially:*																			
English brown. ....	5.55	5.58	.27	.08	17.46	0.0	17.46	25.31	6.37	1.35	3.28	11.80	.71	32.88	8.47	7.25	4.26	15.43	.93
California brown. ....	7.23	4.00	.23	.13	20.04	0.0	20.04	19.22	6.75	1.50	2.27	4.87	.23	26.64	9.36	6.70	3.15	6.75	.32
German brown. ....	0.50	5.25	.09	.50	16.28	0.0	16.28	21.98	7.13	1.05	2.45	5.03	.22	20.71	9.01	7.07	3.30	7.59	.30
Av. of brown flours. ....	7.43	5.24	.10	.24	18.10	0.0	18.10	22.17	6.75	1.33	2.67	7.47	.30	20.71	9.08	7.04	3.57	9.02	.52
German yellow. ....	7.47	4.73	.18	.35	12.65	0.0	12.65	24.21	7.44	.....	1.87	6.12	.23	20.03	9.20	5.85	2.31	7.57	.28
California yellow. ....	5.09	4.60	.22	.29	25.95	0.0	25.95	20.78	6.21	.....	2.21	5.75	.00	30.13	9.00	6.81	3.20	8.34	.00
Av. of yellow flours. ....	6.28	4.66	.20	.32	19.30	0.0	19.30	22.49	6.83	.....	2.04	5.04	.12	30.03	9.10	6.33	2.75	7.95	.14
Average of all varieties of flour. ....	6.96	5.03	.19	.27	18.50	0.0	18.50	22.30	6.78	.....	2.42	6.85	.28	29.84	9.09	6.75	3.30	9.13	.37
Mustard hulls as removed in preparation of flour:†																			
English brown. ....	6.83	5.03	.05	.14	13.81	0.0	13.81	14.21	3.80	0.77	10.00	0.00	1.20	17.80	4.83	6.34	13.74	12.52	1.51
No. 2 brown. ....	8.67	4.43	1.20	.23	10.51	0.0	10.51	14.15	4.04	0.62	11.34	14.05	3.56	17.52	5.00	5.48	14.28	17.38	4.40
Av. of brown hulls. ....	7.75	4.73	1.11	.10	12.16	0.0	12.16	14.18	3.97	0.70	11.12	11.98	2.38	17.70	4.92	5.91	14.01	14.95	2.95
No. 1 yellow. ....	6.46	4.50	2.33	.05	7.03	0.0	7.03	11.07	3.03	.....	16.46	20.40	6.11	12.70	3.58	5.15	19.02	23.58	7.06
No. 2 yellow. ....	5.36	4.65	1.91	.22	6.63	0.0	6.63	10.46	3.22	.....	17.60	20.00	4.21	11.80	3.66	5.28	20.10	22.73	4.77
California yellow. ....	9.12	4.50	1.78	.05	7.79	0.0	7.79	8.51	3.60	.....	16.08	19.90	3.74	10.31	4.44	5.52	19.34	23.05	4.40
German yellow. ....	8.46	4.66	1.80	.04	6.17	0.0	6.17	8.07	2.90	.....	18.95	17.35	2.91	9.45	3.40	5.46	22.20	20.30	3.40
Av. of yellow hulls. ....	7.35	4.65	1.95	.09	6.90	0.0	6.90	9.53	3.19	.....	17.29	19.41	4.24	11.11	3.74	5.35	20.16	22.64	4.93
Average of all samples of hulls. ....	7.48	4.65	1.67	.12	8.66	0.0	8.66	11.07	3.45	.....	15.20	16.96	3.62	13.31	4.14	5.56	18.11	20.23	4.27
Whole ground mustard seeds:†																			
Bari brown. ....	5.88	4.07	.46	.22	37.81	0.0	37.81	13.70	4.10	0.94	4.41	7.34	1.76	24.33	7.28	7.23	7.83	13.03	3.13
California brown. ....	6.49	3.84	.45	.21	35.30	0.0	35.30	14.26	4.40	0.90	4.21	6.94	1.78	24.54	7.73	6.61	7.24	11.94	3.06
Av. of brown seeds. ....	6.18	3.95	.45	.21	36.60	0.0	36.60	13.98	4.29	0.96	4.30	7.14	1.77	24.43	7.50	6.92	7.53	12.48	3.00
German yellow. ....	6.69	4.34	.63	.56	27.19	0.0	27.19	17.75	5.09	0.00	4.87	9.35	1.45	26.85	7.70	6.61	7.36	14.44	2.10
Dutch yellow. ....	5.93	4.83	.73	.33	30.84	0.0	30.84	14.98	4.12	0.00	6.53	10.00	1.82	23.60	6.51	7.64	10.33	15.91	2.87
English yellow. ....	6.43	4.37	.52	.16	27.45	0.0	27.45	16.31	3.96	0.00	4.85	8.42	.92	24.62	6.00	6.61	7.34	12.74	1.39
California yellow. ....	6.82	4.05	.57	.42	28.64	0.0	28.64	16.11	4.72	0.00	5.28	9.60	1.15	24.06	7.31	6.28	8.18	14.88	1.78
Av. of yellow seeds. ....	6.47	4.39	.61	.37	28.53	.....	28.53	16.29	4.47	.....	5.41	9.36	1.33	25.03	6.88	6.78	8.30	14.49	2.05
Average of all six samples of seeds. ....	6.37	4.25	.56	.32	31.22	.....	31.22	15.50	4.41	.....	5.04	8.62	1.48	24.83	7.09	6.83	8.05	13.82	2.40

\* Freed from hulls and with a portion of the fixed oil removed.

† These hulls necessarily have a little of the inner seed adhering thereto.

‡ Ground to a fine paste with full content of oil and hulls.



Piesse and Stansell give the following composition of mustard ash:

	White Seeds.		Brown Seeds.
	Yorkshire.	Cambridge.	Cambridge.
Potash. ....	21.29	18.88	21.41
Soda. ....	0.18	0.21	0.35
Lime. ....	13.46	9.34	13.57
Magnesia. ....	8.17	10.49	10.04
Iron oxide. ....	1.18	1.03	1.06
Sulphuric acid. ....	7.06	7.16	5.56
Chlorine. ....	0.11	0.12	0.15
Phosphoric acid. ....	32.74	35.00	37.20
Silica. ....	1.00	1.12	1.41
Sand. ....	1.82	1.95	1.38
Charcoal. ....	12.82	15.14	7.57
	99.85	100.48	99.70

**Determination of Potassium Myronate, Sinapin Thiocyanate, and Myrosin.**—*Leeds and Everhart Method.*\*—Dry 10 grams of the sample at 105° C., remove the fat with absolute ether, and extract the potassium myronate and sinapin sulphocyanate from the residue in a continuous extractor with a mixture of equal parts of alcohol and water. Evaporate the extract in a tared dish, and dry the combined sulphur compounds at 105° C. to constant weight. Incinerate at a temperature sufficiently high to transform the potassium bisulphate, resulting from the decomposition of the myronate, into the neutral sulphate. Multiply the weight of the ash by 4.77, thus obtaining the weight of potassium myronate. This, deducted from the total weight of the dried alcoholic residue, gives that of the sinapin sulphocyanate.

Remove the alcohol from the residue after the alcoholic extraction, as above described, and treat with 0.5% sodium hydroxide solution, thus dissolving the myrosin. Filter, nearly neutralize the filtrate with dilute hydrochloric acid, add 50 cc. of Ritthausen's copper sulphate solution, and nearly neutralize with dilute sodium hydroxide solution. Collect the heavy green precipitate of copper myrosin on a tared filter, dry at 110° C., and weigh. Ignite, weigh again, and deduct the weight of the ash from the total weight, thus obtaining the weight of the myrosin.

**Determination of Volatile Mustard Oil.**—*Roeser Method.*†—Mix 5 grams of the sample with 60 cc. of water and 15 cc. of 60% alcohol, and

\* Zeits. anal. Chem., 21, 1882, p. 389.

† Jour. pharm. chim, [6], 15, 1902, p. 361.



let stand for 2 hours. Distil into a flask containing 10 cc. of ammonia, and, after about two-thirds of the solution have been distilled off, mix the ammoniacal distillate with 10 cc. of tenth-normal silver nitrate solution, and allow the mixture to stand for 24 hours, after which make up with water to 100 cc. Filter, and treat 50 cc. of the filtrate with 5 cc. of tenth-normal potassium cyanide solution. Titrate the excess of cyanide with the tenth-normal silver nitrate, using as an indicator a 5% solution of potassium iodide, made slightly ammoniacal.

Calculate the percentage of mustard oil, containing 93% of allyl isothiocyanate, (*P*), by the following formula: \*

$$P = \frac{A \times 0.004957 \times 2 \times 100}{0.93 \times 5} = 0.2132A,$$

in which *A* = the number of cc. of N/10 silver nitrate required for the final titration and 0.004957 = the weight of allyl isothiocyanate corresponding to 1 cc. of N/10 silver nitrate.

To obtain the results in terms of allyl isothiocyanate, as recommended by Boutron,† omit 0.93 from the above formula or employ the factor 0.1983 instead of 0.2132.

*The Kuntze Method*, like that of Godamer and others, employs ammonium thiocyanate for the titration. The method, as applied to mustard oil,‡ was adopted by the committee on the ninth revision of the United States Pharmacopœia and, as applied to mustard seed, by the Bureau of Chemistry.§ In details of distillation, it is the same as the Roeser method, except that 5 grams of the sample are macerated for 2 hours at 37° C. with 100 cc. of water, 20 cc. of 95% alcohol are used, and the distillation is begun at once and continued until 60 cc. have passed over. To the distillate 20 cc. of N/10 silver nitrate are added and, after standing over night, the mixture is heated to boiling to cause the silver

\* The conversion factor given in former editions of this work, taken from an abstract of Roeser's paper published in the Analyst (27, 1902, p. 197), was nearly three times too high. Attention to this error was directed by Mr. M. C. Albrech, chemist of the R. T. French Co., mustard makers, Rochester, N. Y., and Mr. A. E. Paul, of the U. S. Food Laboratory, Chicago. This same erroneous factor is given in the fourth edition of Allen's Commercial Organic Analysis and other works, and appears to have vitiated the results of several investigations. In the present edition the analytical results by Leach and by Winton and Bornmann, as well as the factor itself, have been corrected. A. L. W.

† Bul. sci. pharm., July, 1912; Ann. chim. anal., 18, 1913, p. 61.

‡ Arch. pharm., 246, 1908, p. 58; Allen's Coml. Org. Anal., 4 ed., 7, 1913, p. 110.

§ Service and Regulatory Announcements, 20, 1907, p. 59.



sulphide to flock, cooled, made up to 100 cc., shaken, and filtered. Fifty cc. of the filtrate are mixed with 5 cc. of concentrated nitric acid, titrated with N/10 ammonium thiocyanate solution, using 5 cc. of 10% ferric ammonium sulphate solution as indicator.

**Microscopical Characteristics of Powdered Mustard.**—The principal features of powdered black mustard are represented in Fig. 89. The

seed shell or hull is shown in cross-section at (1), *a* being the polygonal-celled epidermis, *b* a layer of palisade-shaped cells, and *c* a thin pigment layer, the brown coloring matter of which is colored blue by iron salts; *d* is the aleurone layer and obscure parenchyma, and *e* the small-celled tissue of the cotyledons, containing fixed oil and albumen.

(2) shows in surface view the various layers of the seed shell, the letters of reference corresponding to those of (1).

(3) shows in surface view a bit of the extreme outer mucilaginous layer of the seed-hull.

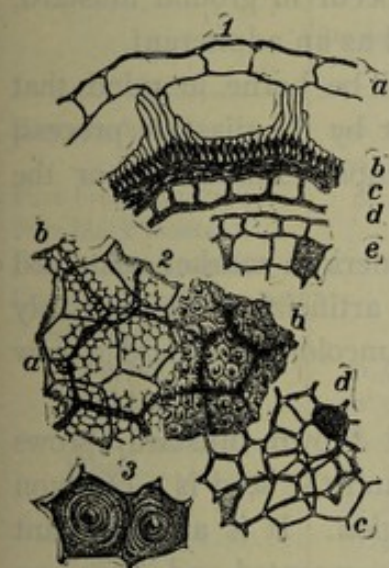


FIG. 89. — Powdered Mustard under the Microscope.  $\times 125$ . (After Moeller.)

Fig. 247, Pl XXXII, shows the appearance in water-mount of pure ground

mustard. This is a photomicrograph of the ground hulled seed without the extraction of the oil, and should not be taken as a standard for commercial mustard "flour," from which, as a rule, a large portion of the oil has been removed. The cellular tissue of the mustard shows in the form of granular masses of loose, fine gray texture; the globular bodies are oil drops. Here and there through the field of ordinary ground mustard are to be seen patches of the yellowish layer of the seed skin of the brown mustard, a mass of which is shown in Fig. 248, with dark-brown spots distributed regularly through it. This is the layer shown at (2) *b*, Fig. 89. The hull of the yellow seed, also common in powdered mustard, is similar in appearance, having dark-brown spots, but with nearly colorless or gray cell walls, instead of yellow.

Patches of the outer hull layer represented by (3) in Fig. 89 are also very common in the commercial mustard flour. Mustard contains no starch.



**U. S. Standards** for mustard flour are as follows: Starch, by diastase method, should not exceed 1.5%, and total ash should not exceed 6%. Mustard seed should not contain more than 5% of total ash nor more than 1.5% of ash insoluble in hydrochloric acid; black mustard seed and related types yield not less than 0.6% of volatile mustard oil.

**Adulteration of Mustard.**—It is difficult to draw the line between the amount of mustard hulls which may naturally occur in ground mustard, and the excess amount which is sometimes added as an adulterant.

In determining starch in mustard, it should be borne in mind that mustard hulls have considerable reducing matter by the diastase process, although no true starch is evident by microscopic examination or the iodine tests.

At one time much of the mustard in the American market contained cereal flour, gypsum, or other makeweights, artificial colors, notably turmeric, being used to cover the fraud. Pure uncolored mustard is now everywhere obtainable.

**Wild Mustard**, consisting of charlock and brown mustard, grows luxuriantly in the grain fields of the Northwest and the seed is a common impurity of the uncleaned wheat from that region. It is an important constituent of wheat screenings, from which it is separated and placed on the market under such names as "Dakota mustard," "Domestic mustard," etc. The brown mustard has been shown by Kate Barber Winton\* to be *B. Besseriana* and not as has been generally believed *B. juncea*. The product also contains other weed seeds, notably those of the mustard family, and also a certain amount of broken wheat.

The following table by Winton and Bornmann† gives botanical analyses and results of determinations of volatile mustard oil in samples of pure mustards and wild mustard separated from screenings. In addition to percentages of volatile oil by Roeser's method are given figures obtained by calculation from the botanical analyses by the following formula:

$$V = 0.0082B + 0.0005C,$$

in which *V* is percentage of volatile oil, *B* is percentage of brown mustard, and *C* is percentage of charlock.

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\* Winton's Microscopy of Vegetable Foods, 2 ed., New York, 1916, 183.

† Jour. Ind. Eng. Chem., 7, 1915, p. 684.



## BOTANICAL ANALYSES AND VOLATILE OIL CONTENT OF CULTIVATED AND WILD MUSTARD SEED.

	AS SEPARATED.			CALCULATED FREE OF FOREIGN SEEDS.			
	Botanical Analysis.			Botanical Analysis.		Volatile Oil.	
	Charlock.	Brown Mustard.	Foreign Seeds.	Charlock.	Brown Mustard.	Actual Determination.	Calc. from Botanical Analysis.
Pure charlock.....	100	0	0	100	0	0.05	
" " .....	100	0	0	100	0	0.09	
Pure brown mustard....	0	100	0	0	100	0.98	
Pure black mustard.....	.....	.....	.....	.....	.....	1.57	
Pure white mustard.....	.....	.....	.....	.....	.....	0.05	
Commercial wild mustard							
Separated from—							
Flaxseed.....	0.5	99.5	0	0.5	99.5	0.82	
Barley.....	90.5	4.7	4.8	95.1	4.9	0.08	0.09
" .....	88.0	10.7	1.3	89.1	10.9	0.13	0.14
Wheat.....	85.2	9.0	5.8	90.4	9.6	0.12	0.13
" .....	82.1	12.9	5.0	86.4	13.6	0.14	0.16
" .....	87.5	8.5	4.0	91.2	8.8	0.11	0.12
" .....	75.7	18.2	6.1	80.6	19.4	0.19	0.20
" .....	47.8	35.8	16.4	57.3	42.7	0.39	0.38
" .....	57.3	27.6	15.1	67.5	32.5	0.32	0.30
" .....	73.5	24.5	2.0	75.0	25.0	0.31	0.24
" .....	86.6	2.5	10.9	97.2	2.8	0.06	0.07
Unknown.....	89.0	9.0	2.0	90.8	9.2	0.15	0.12
" .....	66.2	28.5	5.3	69.9	30.1	0.31	0.28
" .....	87.4	18.1	0.8	88.1	11.9	0.16	0.14

Mustard flour is often made from wild mustard or a mixture of mustard seeds containing wild mustard. If the latter consists in large part of charlock, the product is doubtless inferior, since this seed has a rank flavor; on the other hand, if wild mustard shows a preponderance of brown mustard, it is well adapted for use as a spice.

Charlock is identified by the presence in the palisade cells of a black substance which on heating in various acid reagents (such as chloral hydrate, glycerol, or zinc chloride acidified with hydrochloric acid, syrupy phosphoric or citric acid), becomes bright carmine. A satisfactory reagent is a solution of 16 grams of chloral hydrate in 10 cc. of water and 1 cc. of concentrated hydrochloric acid. Mount 10 mg. of the material in a drop of the reagent, heat gently and examine under a lens.



**Detection of Coloring Matter.**—Turmeric is best detected by the microscope (see pages 468 and 469). Oil-soluble coal-tar dyes should be tested for as in the case of cayenne. Nitro colors, such as naphthol yellow (Martius yellow) and naphthol yellow S, are detected by dyeing tests, with subsequent examination of dyed fabric according to directions given in Chapter XVII.

**PREPARED MUSTARD.**—This product consists of a mixture of ground mustard seed or mustard flour with salt, spices, and vinegar. The U. S. standards require that it should contain not more than 24% of carbohydrates calculated as starch, not more than 12% of crude fiber, and not less than 5.6% of nitrogen.

Most of the product consumed in the United States is of domestic manufacture, although until the passage of the federal food law it was customary to designate it German or French mustard, or label it in a foreign language.

**Composition and Adulteration.**—The common admixtures are wheat flour, maize flour, and other starchy matter, mustard hulls, sugar, chemical preservatives, and artificial colors.

Of 28 brands examined in Connecticut in 1905 by Winton and Andrew,\* 13 contained cereal flour (wheat or corn), 4 salicylic acid, and 25 artificial color (turmeric, nitro-color or azo-color). A summary of the analyses of those brands free from cereal flour and those containing it follows:

	In the Material as Sold.										
	Water.	Acidity Calculated as Acetic Acid.	Total Solids.	Total Ash.	Common Salt.	Ash other than Salt	Protein.	Crude Fiber.	Reducing Matters by Acid Conversion, as Starch.	Nitrogen- free Extract.	Fat.
Prepared mustard free from cereal flour:											
Maximum .....	83.68	3.66	23.67	4.79	3.69	1.31	6.12	1.68	2.92	6.11	7.23
Minimum .....	73.01	2.74	13.32	2.60	1.78	0.82	3.62	0.77	1.83	4.21	2.12
Average .....	78.59	3.05	18.36	3.38	2.32	1.06	4.71	1.17	2.40	4.98	4.12
Prepared mustard containing cereal flour:											
Maximum .....	85.63	3.54	27.70	4.21	3.39	1.16	6.38	1.59	13.69	15.35	3.25
Minimum .....	70.44	1.86	9.89	2.28	1.51	0.48	1.53	0.22	2.12	3.82	0.76

\* Ann. Rep. Conn. Exp. Sta., 1905, p. 123.



	In the Dry, Fat, and Salt-free Material.				
	Ash.	Protein.	Crude Fiber.	Reducing Matters by Acid Conversion, as Starch.	Nitrogen-free Extract.
Prepared mustard free from cereal flour:					
Maximum .....	10.66	43.94	14.12	24.37	44.76
Minimum .....	7.35	32.01	7.77	16.82	34.98
Average .....	8.94	39.44	9.89	20.11	41.73
Prepared mustard containing cereal flour:					
Maximum .....	9.68	33.89	18.44	59.22	66.42
Minimum .....	4.84	21.37	0.45	24.51	41.79
Whole mustard seed (analyses by the author. See page 472):					
Maximum .....	7.64	48.31	10.33	15.91	48.55
Minimum .....	6.28	37.50	7.24	11.94	37.84
Average .....	6.83	44.31	8.05	13.82	40.81

The following methods for the analysis of prepared mustard were used by Winton and Andrew, and afterwards adopted by the Association of Official Agricultural Chemists:

**Determination of Solids, Ash, and Salt** is carried out in one portion of 5 grams of the thoroughly mixed material, following the usual methods. The salt is calculated from the percentage of chlorine.

**Determination of Ether Extract.**—Place 10 grams of the material and about 30 grams of sand in a capsule, and dry on a water bath with stirring. Grind the dried residue and extract with anhydrous ether in the usual manner.

**Determination of Reducing Matters by Acid Conversion.**—Treat the material directly, without previously washing, as described on page 425.

**Determination of Fiber.**—Treat 8 grams of the material (equivalent to about 2 grams of dry matter) as described on page 286, except that (1) the boiling 1.25% acid is added directly to the material without previous extraction, taking care to introduce it in small portions and shake thoroughly until all lumps are broken up, and (2) the fiber after collecting on the weighed paper is washed twice with alcohol and finally with ether until all fat is removed. If these precautions are not followed the results will be high.

**Determination of Protein.**—Nitrogen is determined by the Kjeldahl or Gunning method, and the result multiplied by 6.25.

**Detection of Dyes and Preservatives.**—See chapters XVII and XVIII.



## NUTMEG AND MACE.

**Nature and Production.**—Both nutmeg and mace occur in the fruit of several varieties of trees of the genus *Myristica*, especially of *Myristica fragrans* or *Myristica moschata*, belonging to the family *Myristicaceæ*. The nutmeg tree is a native of the Malay archipelago, and grows from 20 to 30 feet high, somewhat resembling the orange tree in appearance. It does not produce flowers till its eighth or ninth year, after which it bears fruit constantly for many years. The fruit is a globular, pendant drupe, about 5 cm. in diameter, of a yellowish-green color, the pericarp of which, when ripe, splits in two, showing within it the seed, completely surrounded by a fleshy, fibrous aril, or covering of a crimson color. This covering, when dried, furnishes the mace of commerce, while the kernel of the hard, brown seed is the nutmeg.

The seed as separated from the fruit is surrounded by a thick tegument, marked with depressions corresponding to the lobes of the aril or mace, and by a second thin, inner envelope, closely adhering to the seed. The whole seed is dried in the sun for about two months, or by the aid of heat, the tegument becoming separated from the kernel, and, by breaking with a hammer, is readily removed. The kernels are then commonly washed in milk of lime, and again dried, or they are sometimes treated with dry, powdered, air-slaked lime. Liming is alleged to prevent sprouting and ward off the attacks of insects. The so-called brown nutmegs of commerce are those which have not been treated or coated with lime.

**NUTMEGS.**—True nutmegs, the seed kernel of *M. fragrans*, are spheroidal, sometimes nearly spherical, from 20 to 25 mm. long and 15 to 18 mm. in diameter. The outer surface is somewhat furrowed. A cross-section of the kernel shows the grayish-brown, starchy endosperm, mottled with the dark-brown, resinous veins of the perisperm. These veins on pressure with the finger nail present an oily appearance. Near the end of the nutmeg which is attached to the stem, is a small cavity, in which is the undeveloped embryo with two cotyledons.

Macassar, or long nutmegs, the seed kernel of *M. argentea*, are more elongated than true nutmegs and are inferior in flavor.

Nutmeg contains a considerable amount of fixed oil, a volatile oil, starch, and albuminous matter. Its volatile oil is colorless, and is soluble in three parts of strong alcohol. The specific gravity of nutmeg oil varies between 0.865 and 0.920, and its specific rotary power  $(\alpha)_D = 14$  to 28.



Richardson's analyses of three samples of nutmeg are as follows:

	Water.	Ash.	Volatile Oil.	Fixed Oil or Fat.	Starch, etc.	Crude Fiber.	Albuminoids.	Nitrogen.
Whole limed. ....	6.08	3.27	2.84	34.37	36.98	11.30	5.16	.83
Ground limed. ....	4.19	2.22	3.97	37.30	40.12	6.78	5.42	.87
Ground. ....	6.40	3.15	2.90	30.98	41.77	9.55	5.25	.84

König gives the following minimum and maximum composition of nutmeg:

	Minimum.	Maximum.
Water. ....	4.2	12.2
Albuminoids. ....	5.2	6.1
Volatile oil. ....	2.5	4.0
Fat. ....	31.0	37.3
Carbohydrates. ....	29.9	41.8
Cellulose. ....	6.8	12.0
Ash. ....	2.2	3.3

Winton, Ogden, and Mitchell analyzed four samples of nutmeg of known purity, the following being maximum and minimum results:

	Moisture.	Ash.			Ether Extract.	
		Total.	Soluble in Water.	Insoluble in HCl.	Volatile.	Non-volatile.
Maximum. ....	10.83	3.26	1.46	0.01	6.94	36.94
Minimum. ....	5.79	2.13	0.82	0.00	2.56	28.73

	Alcohol Extract.	Reducing Matters by Acid Conversion, as Starch.	Starch by Diastase.	Crude Fiber.	Nitrogen $\times 6.25$ .	Total Nitrogen.
Maximum. ....	17.38	25.60	24.20	3.72	7.00	1.12
Minimum. ....	10.42	17.19	14.62	2.38	6.56	1.05

**Microscopical Structure of Nutmeg.** (Fig. 90.)—The thin-walled cells of the parenchyma of the endosperm or albumen are shown at (1), with starch grains. Simple and compound granules of the starch are shown at (2). Aleurone grains appear as shown at (3), and (4) represents in surface view the epidermis, or brown seed coat, with its numerous layers of flat cells. Powdered nutmeg under the microscope in water-mount shows most commonly a sponge-like, loose meshwork of bruised



or broken cellular tissue, with many starch granules, and occasional fragments of the epidermis.

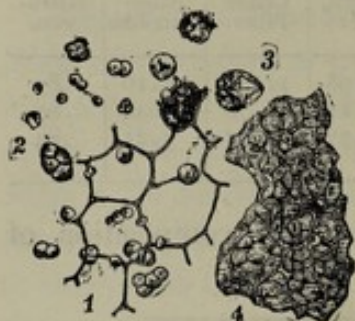


FIG. 90.—Powdered Nutmeg under the Microscope.  $\times 125$ . (After Moeller.)

Fig. 240, Pl. XXX, is a photomicrograph of a water-mounted sample of pure nutmeg. The starch granules of nutmeg are different from other starches in appearance, being almost circular as a rule, quite uniform in size (averaging 0.006 mm. in diameter), and having very distinct central hyla.

**The U. S. Standards** for nutmegs are as follows: Non-volatile ether extract should be not less than 25%; total ash should not exceed 5%; ash insoluble in hydrochloric acid should not exceed 0.5%; crude fiber should not exceed 10%.

**Adulteration of Nutmeg.**—Nutmegs are usually sold whole, since the housewife much prefers to grate the whole nutmeg, rather than to use the ground material. It is hence less liable to adulteration than the other spices, though of late more of the ground nutmeg is being sold in packages. Samples of ground nutmeg have been found in Massachusetts adulterated with wheat and nutshells. One sample was found to contain at least 25% of ground cocoanut shells.

Nutmegs which have become mouldy, or have been eaten out by insects, have been imported for grinding, as sound nutmegs are not readily reduced to a powder. Such a product is obviously unfit for consumption.

An inferior variety is known as the Macassar nutmeg. This lacks much of the agreeable pungency of the better grades.

**MACE.**—The crimson-colored aril that surrounds the nutmeg kernel within the pericarp, as above described (p. 480), has many narrow, flattened lobes. In the process of drying to form the mace of commerce, it loses its brilliant red color, and turns a yellowish brown. When dried, it is brittle, somewhat translucent, and of a pungent odor. Whole mace appear on the market in the form of flat membranous masses, 3 to 4 cm. long.

Macassar mace has a characteristic wintergreen taste. Bombay mace is lacking in spicy flavor.

Mace contains no starch as such, but a modified form of starch known as amyloextrin. This is a carbohydrate,  $C_{36}H_{62}O_{31} + H_2O$ , which produces with iodine a red coloration. Mace has a large amount of fixed oil, as well as considerable resinous and albuminous matter, and a volatile oil which much resembles that of nutmeg.



The specific gravity of volatile oil of mace is rather higher than that of nutmeg oil. Its specific rotary power,  $(\alpha)_D = 10$  to  $20$ .

König's figures for the composition of mace are as follows:

	Minimum.	Maximum.
Water .....	4.9	17.6
Protein ( $N \times 6.25$ ) .....	4.6	6.1
Volatile oil .....	4.0	8.7
Fat .....	18.6	29.1
Carbohydrates .....	41.2	44.1
Cellulose .....	4.5	8.9
Ash .....	1.6	4.1
Alcoholic extract .....	45.1	55.7

Richardson gives the following as the results of analyses of three samples made by him:

	Water.	Ash.	Volatile Oil.	Resin.	Undetermined.	Crude Fiber.	Protein ( $N \times 6.25$ ).	Nitrogen.
Whole mace .....	5.67	4.10	4.04	27.50	41.17	8.93	4.55	.73
Ground mace. ....	4.86	2.65	8.66	29.08	35.50	4.48	6.13	.98
Ground mace. ....	10.47	2.20	8.68	23.33	34.68	6.88	5.08	.81

Winton, Ogden, and Mitchell's analyses of four samples of pure Banda or Penang mace, as well as of Bombay and Macassar mace, are summarized as follows:

	Moisture.	Ash.			Ether Extract.	
		Total.	Soluble in Water.	Insoluble in HCl	Volatile.	Non-volatile.
True mace: Maximum....	12.04	2.54	1.33	0.21	8.65	23.72
Minimum ....	9.78	1.81	1.06	0.00	6.27	21.63
Average.....	11.05	2.01	1.13	0.07	7.58	22.48
Macassar. ....	4.18	2.01	1.11	0.03	5.89	53.54
Bombay (adulterant) .....	0.32	1.98	1.37	0.07	4.65	59.81

	Alcohol Extract.	Reducing Matters by Acid Conversion, as Starch.	Starch by Diastase.*	Crude Fiber	Nitrogen $\times 6.25$ .	Total Nitrogen.
True mace: Maximum....	24.76	34.42	30.43	3.85	7.00	1.12
Minimum ....	22.07	26.77	23.12	2.94	6.25	1.00
Average.....	23.11	31.73	27.87	3.20	6.47	1.03
Macassar. ....	32.89	10.39	8.78	4.57	7.00	1.12
Bombay (adulterant) .....	44.27	16.20	14.51	3.21	5.06	0.81

\* The figures in this column do not express starch, but amylo-dextrin, which like starch may be determined by the diastase method.



**Microscopical Structure of Mace.**—Fig. 91 shows characteristics of mace, (1) being a cross-section through it, (2) a surface view of the

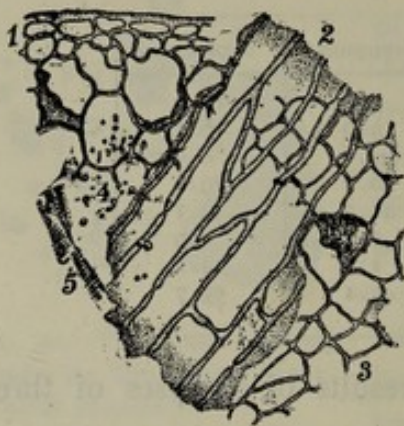


FIG. 91.—Powdered Mace under the Microscope.  $\times 125$ . (After Moeller.)

epidermis, showing its elongated, often nearly rectangular cells, and (3) the large-celled parenchyma, in which are numerous oil globules. The contents of the parenchyma cells are for the most part colorless, consisting of protein, fat, and granules of amylodextrin, which are shown at (4). At (5) are shown fragments of vascular tissue.

The water-mounted powder of pure mace shows no highly colored fragments, but as a mass, is white or grayish, and of loose texture. Occasional pale, yellowish, lumpy masses appear, and pale-brown fragments of the seed coating. The amylodextrin granules (which are colored red-brown by solution of iodine) are very small.

**U. S. Standards.**—Mace should contain not less than 20 nor more than 30% of non-volatile ether extract; nor more than 3% of total ash; nor more than 0.5% ash insoluble in hydrochloric acid; nor more than 10% of crude fiber.

**Adulteration.**—Turmeric and cereal starches have been detected in mace, but by far the most common adulterant is the so-called false, or wild mace, otherwise known as Bombay mace.

The non-volatile ether extract of both Bombay and Macassar mace is twice as high as that of true mace, and at room temperature the fixed oil of Bombay mace is a thick and viscous fat, while that of true and other maces is a thin oil.

The refractive indices of the fixed oils of various species of pure, as well as of Bombay mace, as determined by Lythgoe follow:



		$n_D$ at 35° C.
Banda Mace	(1) .....	1.4848
" "	(2) .....	1.4747
" "	(3) .....	1.4829
Batavia Mace	(1) .....	1.4893
" "	(2) .....	1.4975
Papua Mace	(1) .....	1.4816
" "	(2) .....	1.4795
West Indian Mace	(1) .....	1.4766
Bombay Mace	(1) .....	1.4615
" "	(2) .....	1.4633

The microscope indicates at once when Bombay mace is present in a sample. The oil glands situated in the outer layers of Bombay mace are strongly colored and contain a reddish resinous substance, while the glands of the more interior layers have balsam-like contents of bright yellow color. Both the red and yellow lumps are visible in water mounts, but a 5% potassium hydroxide solution colors them a brilliant blood-red, making possible an approximate percentage estimation of Bombay mace in true mace.

*Hefelmann's Test for Bombay Mace*\* consists in saturating a strip of filter-paper with an alcoholic solution of the mace, and removing the excess of liquid by pressure between filter-paper. On treating with a drop of dilute sodium or potassium hydroxide solution, a red color is produced in presence of the wild mace.

*Waage's Test*.†—One part of the mace is extracted with ten parts of alcohol, and potassium chromate solution is added to the extract. If Bombay mace is present, the solution becomes red, and the precipitate, which is at first yellow, becomes red on standing. True mace gives a yellow solution and precipitate, and the latter does not change greatly on standing.

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\* Pharm. Zeit., 36, 1891, p. 122.

† Pharm. Centbl., 33, 1892, p. 372.



## CHAPTER XIII.

### OILS AND FATS.

**Constituents.**—The oils and fats are essentially the glycerol salts or triglycerides of the fatty acids. Free fatty acids, lecithin (page 35), and sterols (cholesterol and phytosterol, page 521) are among the minor constituents. Vegetable oils owe their yellow color to carotin or related substances. Butter fat also contains carotin derived indirectly from feeds. The greenish color of certain grades of olive and other oils is due to chlorophyll.

Mono- and di-glycerides are prepared synthetically, but do not exist in natural oils or fats.

**Solubilities of Oils and Fats.**—The edible members of the group are insoluble in water, and are almost insoluble in cold 95% alcohol, though they are somewhat soluble in absolute alcohol. They are readily soluble in ether, petroleum ether, chloroform, carbon tetrachloride, various chloro-compounds of ethylene and ethane (especially trichloro-ethylene), acetone, amyl alcohol, oil of turpentine, and carbon bisulphide.

**Fatty Acids.**—Following, compiled from Lewkowitsch,\* are the fatty acids whose glycerides occur in edible oils and fats, together with their melting- and boiling-points so far as these have been determined.

ACIDS OF EDIBLE OILS AND FATS.

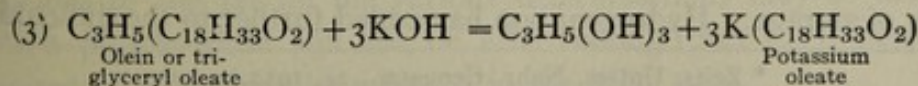
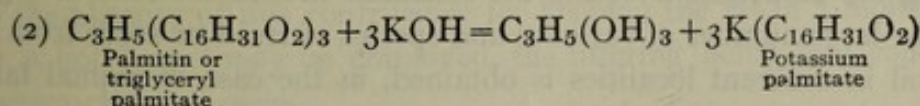
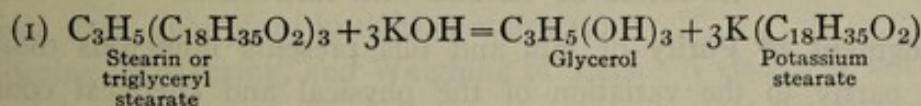
Name.	Formula.	Melting-point.	Boiling-point.	Occurrence in Oils and Fats.
Acetic Series.....	$C_nH_{2n}O_2$			
Butyric.....	$C_4H_8O_2$	$-6.5^\circ$	$162.3^\circ$	Butter.
Caproic.....	$C_6H_{12}O_2$	.....	202-203	Butter, cocoanut, palm nut.
Caprylic.....	$C_8H_{16}O_2$	16.5	236-237	Butter, cocoanut, palm nut.
Capric.....	$C_{10}H_{20}O_2$	31.3	268-270	Butter, cocoanut, palm nut.
Lauric.....	$C_{12}H_{24}O_2$	43.6	176	Cocoanut, palm nut.
Myristic.....	$C_{14}H_{28}O_2$	53.8	196.5	Cocoanut, palm nut, sesame, butter.
Palmitic.....	$C_{16}H_{32}O_2$	62.6	215	Nearly all oils and fats.

\* Chemical Technology and Analysis of Oils, Fats, and Waxes, 5th ed., London, I, 1913, p. 348.



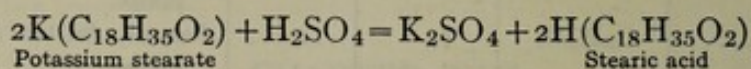
Name.	Formula.	Melting-point.	Boiling-point.	Occurrence in Oils and Fats.
Stearic.....	$C_{18}H_{36}O_2$	69.3°	232.5	Fats, most oils, except olive and maize.
Arachidic.....	$C_{20}H_{40}O_2$	77	.....	Peanut, butter (trace), rape, cocoa.
Behenic.....	$C_{22}H_{44}O_2$	83-84	.....	
Lignoceric.....	$C_{24}H_{48}O_2$	81	.....	Peanut.
Oleic Series.....	$C_nH_{2n-2}O_2$			
Hypogæic.....	$C_{16}H_{30}O_2$	33	236	Peanut.
Oleic.....	$C_{18}H_{34}O_2$	14	232.5	Nearly all fats and oils.
Iso-oleic.....	$C_{18}H_{34}O_2$	44-45	.....	
Rapic.....	$C_{18}H_{34}O_2$	.....	.....	Rape, mustard.
Erucic.....	$C_{22}H_{42}O_2$	33-34	264	Rape, mustard.
Linolic Series.....	$C_nH_{2n-4}O_2$			
Linolic.....	$C_{18}H_{32}O_2$	under -18	.....	Linseed, olive, cottonseed, peanut, sesame, maize, cocoa, poppy seed, soy bean, sunflower.
Linolenic series.....	$C_nH_{2n-6}O_2$			
Linolenic.....	$C_{18}H_{30}O_2$	.....	.....	Linseed, poppy seed, soy bean.
Clupanodonic Series..	$C_nH_{2n-8}O_2$			
Clupanodonic.....	$C_{18}H_{28}O_2$	.....	.....	Whale, cod-liver, fish.

**Saponification of Fats and Oils.**—By this term is meant the decomposition of the glycerides composing the fats or oils, whereby the tri-atomic alcohol glycerol and the fatty acids or their alkali soaps are separated. The saponification process is commonly applied in carrying out many determinations of value on fats and oils, such as those of the soluble and insoluble fatty acids, the Reichert value, etc. As commonly carried out, the tri-glycerides are first split up into glycerol and the soluble soaps of the fatty acids by the action of caustic alkali, usually in solution in alcohol. This part of the process in the case of a given oil, composed, for example, of stearin, olein, and palmitin, is illustrated as follows:





These "soaps," or potassium salts of the fatty acids, are further decomposed by the action of sulphuric acid into the free fatty acids and potassium sulphate, in the case of potassium stearate, as follows:



The result obtained in the determination of saponification number—the number of milligrams of potassium hydroxide required to saponify 1 gram of fat—is inversely proportional to the average molecular weight of the glycerides present.

**Hydrogenation of Oils.**—Recently the hardening of oils by hydrogenation, employing nickel (or less often platinum or palladium) as a catalyst, has become of commercial importance. By this process olein and other unsaturated glycerides are more or less completely converted into stearin as is shown by the lowering of the iodine number and the change of the physical constants. Not only are vegetable oils hardened by hydrogenation but also whale oil and other fish oils which may be transformed from inedible products into bland and tasteless fats. Hardened oils are used in lard substitutes in place of natural stearin.

Various oxides and salts of nickel have been used in place of the metal in hydrogenation, but the actual catalyst in all such cases, although for some years a matter of dispute, has been shown without question to be the metal.

The treatment, unless slight, renders useless the Halphen test for cottonseed oil and the hexabromide test for fish oils, but does not usually interfere with the Baudouin test for sesame oil. Bömer\* finds that neither cholesterol nor phytosterol is changed.

The table of results by Bömer\* on page 489 shows the effects of hydrogenation on the chemical and physical constants of certain oils.

#### ANALYSIS OF EDIBLE OILS AND FATS.

**Judgment of Purity** of fats and oils presents numerous difficulties owing partly to the variation of the physical and chemical constants. Among the influences affecting the constants are, in the case of vegetable oils, the large number of varieties and species of fruits or seeds from which each oil in different localities is obtained, in the case of animal fats, the breed and feed, and in both cases the method of refining, age, and conditions of storage. Hydrogenation has added further to the difficulties.

\* Zeits. Unters. Nahr. Genussm., 24, 1912, p. 107.



## EFFECTS OF HYDROGENATION ON THE CONSTANTS OF OILS.

	Hardness.	Color.	Melting Point. Deg. C.	Solidi- fying Point. Deg. C.	Refract- ometer Reading at 40° C.	Acid Number (Mgs. KOH per gram).	Saponi- fication Number.	Iodine Number.
Peanut oil								
Natural.....	Fluid	Yellow	—	—	56.8	1.1	191.1	84.4
Hardened.....	Soft	White	44.2	30.2	52.3	1.3	188.3	56.5
".....	Medium *	White	46.1	32.1	50.5	0.9	188.4	54.1
".....	Hard †	White	53.5	38.8	49.0	1.2	189.0	42.2
Sesame oil								
Hardened.....	Hard †	White	62.1	45.3	38.4‡	4.7	188.9	25.4
Cottonseed oil.....								
Hardened.....	Medium *	Yellow	38.5	25.4	53.8	0.6	195.7	69.7
Cocoanut oil								
Natural.....	Soft	White	25.6	20.4	37.4	0.3	255.6	11.8
Hardened.....	Medium *	White	44.5	27.7	35.9	0.4	254.1	1.0
Whale oil								
Hardened.....	Hard †	Yellow	45.4	33.7	49.1	1.1	193.0	46.8

\* Consistence of lard.

† Consistence of tallow.

‡ Determined at 50° C.

It is often difficult to name the adulterant or estimate the extent of adulteration. In some cases a large number of tests must be made before one can intelligently form an opinion. It should be borne in mind that skilful manufacturers may adulterate the edible oils and fats with mixtures intended to confuse the chemist, and yield on analysis constants that are entirely misleading. Information may often be gained by carefully noting the color, taste, odor, and appearance of the sample.

**Rancidity** should not be confounded with acidity, although rancid oils usually are high in acids. Lewkowitsch holds that fatty acids are liberated by the action of moisture in the presence of enzymes. If in addition the oil is exposed to air and light, the fatty acids are acted on, causing rancidity, which is detected by taste and smell, although chemically little understood. As a rule rancidity develops more readily in liquid oils in which olein predominates than in solid fats. To avoid changes samples should be kept in a dark, cool place in tight containers.

**Filtering, Measuring, and Weighing of Fats.**—A steam- or hot-water-jacketed funnel as represented in Fig. 92 is convenient for filtering fats, or, in the absence of this contrivance for keeping the fat in a molten condition, a hot funnel may be employed, the filtering being best conducted in a warm closet or oven.

Portions of the fat for the various determinations may be measured off with a pipette while still hot, or, after cooling (over ice if necessary), the desired amounts may be removed in the solid state.



**Specific Gravity.**—The specific gravity of liquid oils is most conveniently taken either at room temperature or at 15.5°, being always best referred to the latter. Either the hydrometer, Westphal balance, Sprengel tube, or pycnometer is employed, according to the degree of accuracy required. If taken at any other temperature than 15.5°, say

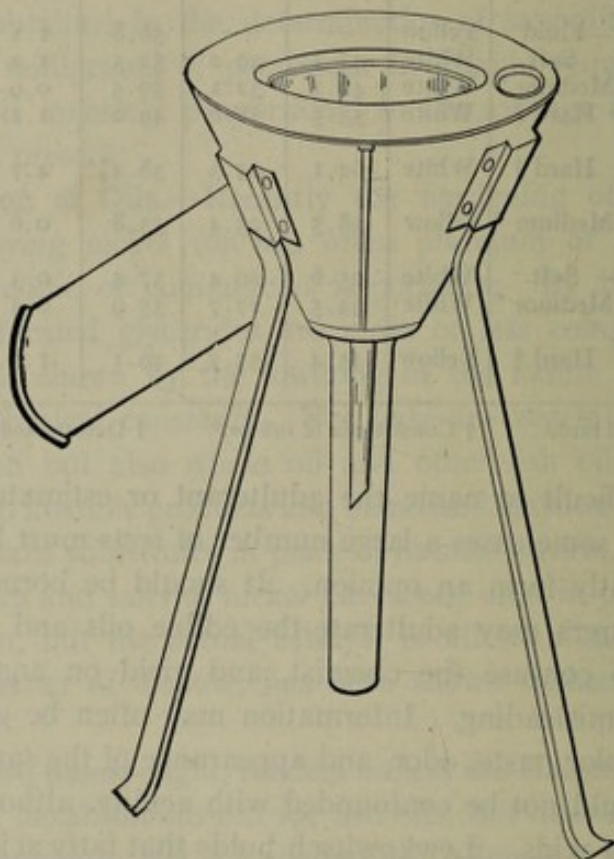


FIG. 92.—Jacketed Funnel for Hot Filtration.

at room temperature,  $T$ , the specific gravity may be computed at 15.5° by the formula

$$G = G' + K(T - 15.5),*$$

in which  $G$  is the specific gravity at 15.5°,  $G'$  the specific gravity at  $T^\circ$ , and  $K$  a factor varying with the different oils as follows:

FACTORS FOR CALCULATING SPECIFIC GRAVITY.

Oil.	Correction for 1° C.	Observer.
Cod-liver oil. ....	0.000646	A. H. Allen
Lard oil. ....	.000658	C. M. Wetherill
Olive oil. ....	.000629	C. M. Stillwell
Peanut oil. ....	.000655	A. H. Allen
Rape oil. ....	.000620	"
Sesame oil. ....	.000624	"
Cottonseed oil. ....	.000629	"

\* Allen, Com. Org. Anal., 4 Ed.; Vol. II, p. 49.



Unless the most accurate work is necessary, it is sufficient to assume in all cases  $K=0.00064$ , in which case the formula becomes  $G=G'+0.00064(T-15.5)$ .

In the case of solid fats, it is most convenient to take the specific gravity of the melted fat. This may be done at any temperature above the melting-point by either of the instruments above described, or at the temperature of boiling water by the Westphal balance or pycnometer. The figures thus obtained may be compared with those for water determined in the same apparatus, either at the same temperature or at  $15.5^{\circ}$ .

When the pycnometer is used, it is immersed in a water-bath, the temperature of which is well above the melting-point of the fat, say  $35^{\circ}$  or  $40^{\circ}$  or  $100^{\circ}$ . While still immersed nearly to the neck, it is carefully filled with the melted fat and kept in the bath till the fat has acquired the same temperature, usually about 15 minutes. If the pycnometer is provided with a thermometer stopper, this will serve to indicate the temperature; otherwise a separate thermometer is inserted in the bath. The pycnometer is then removed, cleaned, dried, and cooled to the room temperature, at which it is weighed. The factors employed in the above formula for calculation of specific gravity of solid fats at  $15.5^{\circ}$  are as follows:

FACTORS FOR CALCULATING SPECIFIC GRAVITY.

Fat.	Correction for $1^{\circ}$ C.
Cocoa butter.....	0.000717
Tallow.....	.000673
Lard.....	.000650
Butter fat.....	.000617
Cocoanut stearin.....	.000674
Cocoanut oil.....	.000642
Palm nut oil.....	.000657

Either the Westphal balance or the hydrometer may be used directly on the melted fat, carefully recording the temperature and calculating as above.

For making the determination with the Westphal balance at the temperature of boiling water, the melted fat is contained in a vessel immersed in a boiling water-bath, and kept sufficiently long to acquire that temperature, which is carefully noted.

**Calculation of Proportions of Two Known Oils in Mixture.\***—This may be roughly accomplished from the specific gravity of the mixture and of the oils known to compose it.

\* Villiers et Collin, *Les Substances Alimentaires*, Paris, 1900, p. 646.



Let  $G$  = specific gravity of mixture,  
 $D$  and  $D'$  = specific gravity of the two oils,  
 and  $X$  = % oil of specific gravity  $D$ .

Then

$$X = \frac{100(G - D')}{D - D'}.$$

#### SPECIFIC GRAVITY OF EDIBLE OILS AND FATS.

Oil.	Specific Gravity, $\frac{15.5^{\circ}}{15.5^{\circ}}$
Rape oil.....	0.913-0.917
Olive oil.....	0.915-0.918
Lard oil.....	0.915-0.918
Mustard oil.....	0.914-0.919
Sesame oil.....	0.921-0.924
Peanut oil.....	0.917-0.926
Cottonseed oil.....	0.922-0.926
Sunflower oil.....	0.924-0.926
Maize oil.....	0.921-0.927
Poppyseed oil.....	0.924-0.927
Soy oil.....	0.922-0.928
Linseed oil.....	0.931-0.941

Fat.	Specific Gravity, $\frac{100^{\circ}}{15.5^{\circ}}$
Mutton tallow.....	0.858-0.860
Cacao butter.....	0.858
Oleo stearin.....	0.858-0.862
Beef tallow.....	0.860-0.863
Oleo oil.....	0.860-0.863
Lard.....	0.859-0.864
Cottonseed stearin.....	0.864-0.868
Cocoanut stearin.....	0.870
Palm kernel stearin.....	0.870
Butter fat.....	0.865-0.870
Palm kernel oil.....	0.859-0.873
Cocoanut oil.....	0.863-0.874

**Determination of Viscosity** in the case of edible oils, is of less importance than in the case of lubricating oils, and gives little insight into the nature or purity of the sample. Its application in the detection of oleo-margarine in butter is discussed by Lewkowitsch.\* Descriptions of viscosimeters are given by the same author.

\* Loc. cit., p. 348.



**Determination of Refractive Index**, and the reading on the arbitrary scale of the butyro-refractometer, express in two different and interchangeable terms the refraction value, a useful and easily determined constant of fats and oils.

For the routine examination of fats and oils the butyro-refractometer is more convenient than the Abbé refractometer, and the readings obtained by the former instrument are less cumbersome than refractive indices.

These instruments and details with regard to their manipulation are described in Chapter VI.

The readings on the scale of the butyro-refractometer may be readily transformed into refractive indices and *vice versa* by table or by means of the Leach and Lythgoe slide rule (page 93). Lythgoe's\* table on pages 494 and 495 is useful as showing readings on the butyro-refractometer of edible oils and fats at various temperatures.

#### REFRACTION OF EDIBLE OILS AND FATS.

Oil.	Butyro Scale, 25°.	Refractive Index, 25°.
Lard oil.....	54-60	1.4620-1.4660
Olive oil.....	60-64	1.4659-1.4680
Peanut oil.....	65-67.5	1.4691-1.4707
Cottonseed oil.....	66-70	1.4698-1.4723
Rape oil.....	68-70	1.4708-1.4723
Mustard oil.....	64-71	1.4688-1.4729
Sesame oil.....	68-71	1.4710-1.4729
Maize oil.....	71-72	1.4729-1.4734
Sunflower oil.....	72	1.4735
Soy oil.....	71-73	1.4729-1.4742
Poppy seed oil.....	70-75	1.4723-1.4754
Linseed oil.....	81-87	1.4789-1.4824

Fat.	Butyro Scale, 40°.	Refractive Index, 40°.
Cocoonut stearin.....		
Cocoonut oil.....	33-36	1.4474-1.4495
Palm kernel stearin.....		
Palm kernel oil.....	36-39	1.4495-1.4517
Butter fat.....	40.5-46 *	1.4527-1.4566 †
Oleo stearin.....	40-48	1.4524-1.4580
Cacao butter.....	46-48	1.4566-1.4580
Beef tallow.....	46-49	1.4566-1.4586
Mutton tallow.....	49	1.4586
Oleo oil.....	47-50 ‡	1.4573-1.4593 §
Lard.....	48.5-52.5	1.4583-1.4609
Cottonseed stearin.....		

\* 48.7-54.2 at 25°. † 1.4582-1.4621 at 25°. ‡ 55.2-58.2 at 25°. § 1.4627-1.4647 at 25°.

\* Tech. Quart., 16, 1903, p. 222.



## CALCULATED READINGS ON BUTYRO-REFRACTOMETER OF EDIBLE OILS AND FATS.

Temp. C.	Cocoanut Oil.	Butter.*	Beef Stearin.	Cacao Butter.	Beef Tallow.	Lard Stearin.	Beef Oleo.	Lard.†	Lard Oil.
45.0	31.6	41.5	41.9	43.7	44.1	44.9	45.0	48.2	
44.5	31.9	41.8	42.2	44.0	44.3	45.1	45.3	48.4	
44.0	32.2	42.0	42.4	44.2	44.6	45.5	45.6	48.7	
43.5	32.4	42.3	42.6	44.5	44.8	45.7	45.9	49.0	
43.0	32.7	42.6	42.9	44.8	45.1	46.0	46.1	49.3	
42.5	32.9	42.9	43.2	45.0	45.4	46.3	46.4	49.6	
42.0	33.2	43.1	43.5	45.3	45.6	46.5	46.7	49.9	
41.5	33.5	43.4	43.7	45.6	45.8	46.8	47.0	50.1	
41.0	33.7	43.7	44.0	45.9	46.1	47.0	47.3	50.4	
40.5	34.0	43.9	44.2	46.1	46.3	47.3	47.6	50.7	
40.0	34.3	44.2	44.5	46.4	46.6	47.6	47.8	51.0	51.6
39.5	34.5	44.5	.....	46.6	46.8	47.8	48.1	51.3	51.8
39.0	34.8	44.8	.....	46.8	47.1	48.1	48.4	51.6	52.1
38.5	35.0	45.0	.....	47.1	47.4	48.4	48.7	51.9	52.4
38.0	35.3	45.3	.....	47.4	47.6	48.6	48.9	52.1	52.6
37.5	35.5	45.6	.....	47.6	47.8	48.9	49.2	52.4	52.8
37.0	35.8	45.9	.....	47.9	48.1	49.2	49.5	52.7	53.1
36.5	36.1	46.1	.....	48.2	48.3	49.4	49.8	53.0	53.4
36.0	36.3	46.4	.....	48.5	48.6	49.7	50.0	53.3	53.7
35.5	36.6	46.7	.....	48.7	48.8	50.0	50.3	53.6	54.0
35.0	36.9	47.0	.....	49.0	49.1	50.2	50.6	53.9	54.2
34.5	37.1	47.2	.....	.....	.....	.....	50.9	54.2	54.5
34.0	37.4	47.5	.....	.....	.....	.....	51.2	54.4	54.7
33.5	37.6	47.8	.....	.....	.....	.....	51.5	54.7	55.0
33.0	37.9	48.1	.....	.....	.....	.....	51.7	55.0	55.3
32.5	38.1	48.3	.....	.....	.....	.....	52.0	55.3	55.6
32.0	38.4	48.6	.....	.....	.....	.....	52.3	55.6	55.9
31.5	38.6	48.9	.....	.....	.....	.....	52.6	55.9	56.1
31.0	38.9	49.2	.....	.....	.....	.....	52.8	56.1	56.4
30.5	39.2	49.5	.....	.....	.....	.....	53.1	56.4	56.7
30.0	39.5	49.8	.....	.....	.....	.....	53.4	56.7	57.0
29.5	39.7	50.0	.....	.....	.....	.....	53.7	57.0	57.2
29.0	40.0	50.3	.....	.....	.....	.....	53.9	57.3	57.5
28.5	40.3	50.5	.....	.....	.....	.....	54.1	57.6	57.8
28.0	40.5	50.8	.....	.....	.....	.....	54.4	57.8	58.1
27.5	40.8	51.1	.....	.....	.....	.....	54.7	58.1	58.3
27.0	41.0	51.4	.....	.....	.....	.....	55.0	58.4	58.6
26.5	41.3	51.6	.....	.....	.....	.....	55.2	58.7	58.9
26.0	41.5	51.9	.....	.....	.....	.....	55.5	59.0	59.2
25.5	41.8	52.2	.....	.....	.....	.....	55.8	59.3	59.5
25.0	42.0	52.5	.....	.....	.....	.....	56.1	59.6	59.8

\* Butter readings by Zeiss.

† Lard readings by Hefelmann.



## CALCULATED READINGS—(Continued).

Temp. C.	Olive Oil.	Peanut Oil.	Cotton- seed Oil.	Rape- seed Oil.	Sesame Oil.	Yellow Mustard Oil.	Black Mustard Oil.	Sun- flower Oil.	Corn Oil.	Poppy- seed Oil.
35.0	57.0	59.8	61.8	62.1	62.3	63.0	64.2	64.5	65.0	65.5
34.5	57.2	60.0	62.1	62.4	62.5	63.3	64.5	64.8	65.3	65.8
34.0	57.4	60.3	62.3	62.7	62.8	63.6	64.8	65.1	65.6	66.1
33.5	57.7	60.6	62.5	63.0	63.1	63.9	65.1	65.4	65.9	66.4
33.0	58.0	60.9	62.8	63.3	63.4	64.1	65.3	65.7	66.2	66.7
32.5	58.3	61.1	63.0	63.6	63.7	64.4	65.6	66.0	66.5	67.0
32.0	58.5	61.4	63.2	63.8	64.0	64.7	65.9	66.3	66.8	67.3
31.5	59.0	61.7	63.6	64.1	64.3	65.0	66.2	66.6	67.1	67.6
31.0	59.2	62.0	64.0	64.4	64.6	65.3	66.5	66.9	67.4	67.9
30.5	59.5	62.2	64.2	64.7	64.9	65.6	66.8	67.2	67.7	68.2
30.0	59.9	62.5	64.5	65.0	65.1	65.8	67.0	67.5	68.0	68.5
29.5	60.1	62.8	64.9	65.3	65.4	66.1	67.3	67.7	68.2	68.7
29.0	60.3	63.1	65.1	65.6	65.7	66.4	67.6	68.0	68.5	69.0
28.5	60.6	63.3	65.3	65.9	66.0	66.7	67.9	68.3	68.8	69.3
28.0	60.9	63.6	65.7	66.1	66.2	66.9	68.1	68.6	69.1	69.6
27.5	61.1	63.9	66.0	66.4	66.5	67.2	68.4	68.9	69.4	69.9
27.0	61.5	64.2	66.5	66.7	66.8	67.5	68.7	69.2	69.7	70.2
26.5	62.0	64.4	67.0	67.0	67.1	67.8	69.0	69.5	70.0	70.5
26.0	62.2	64.7	67.3	67.3	67.0	68.0	69.2	69.8	70.3	70.8
25.5	62.4	65.0	67.5	67.6	67.7	68.3	69.5	70.1	70.6	71.1
25.0	63.0	65.3	67.9	67.8	67.9	68.6	69.8	70.4	70.9	71.4
24.5	63.3	65.5	68.2	68.1	68.2	68.9	70.1	70.7	71.2	71.7
24.0	63.6	65.8	68.5	68.4	68.5	69.2	70.4	71.0	71.5	72.0
23.5	63.9	66.1	68.8	68.7	68.8	69.5	70.7	71.3	71.8	72.3
23.0	64.2	66.4	69.1	69.0	69.1	69.7	70.9	71.6	72.1	72.6
22.5	64.5	66.6	69.4	69.3	69.4	70.0	71.2	71.9	72.4	72.9
22.0	64.8	66.9	69.7	69.7	69.7	70.3	71.5	72.2	72.7	73.2
21.5	65.1	67.1	70.0	70.0	70.0	70.6	71.8	72.5	73.0	73.5
21.0	65.4	67.4	70.3	70.3	70.3	70.9	72.1	72.8	73.3	73.8
20.5	65.7	67.7	70.6	70.6	70.5	71.2	72.4	73.1	73.6	74.1
20.0	66.0	68.0	70.9	70.8	70.8	71.4	72.6	73.4	73.9	74.4
19.5	66.3	68.2	71.2	71.1	71.1	71.7	72.9	73.6	74.1	74.6
19.0	66.6	68.5	71.5	71.4	71.4	72.0	73.2	73.9	74.4	74.9
18.5	66.9	68.8	71.8	71.7	71.7	72.3	73.5	74.2	74.7	75.2
18.0	67.2	69.1	72.1	72.0	72.0	72.6	73.8	74.5	75.0	75.5
17.5	67.5	69.3	72.4	72.3	72.3	72.9	74.1	74.8	75.3	75.8
17.0	67.8	69.6	72.7	72.6	72.5	73.1	74.3	75.1	75.6	76.1
16.5	68.1	69.9	73.0	72.9	72.8	73.4	74.6	75.4	75.9	76.4
16.0	68.4	70.2	73.3	73.2	73.1	73.7	74.9	75.7	76.2	76.7
15.5	68.7	70.5	73.6	73.5	73.4	74.0	75.2	76.0	76.5	77.0
15.0	68.9	70.8	73.8	73.8	73.7	74.3	75.5	76.3	76.8	77.3



**Determination of Melting-point and Solidifying Point.**—A piece of small glass tubing is drawn out to a capillary open at both ends, and this is inserted into a beaker of the fat, melted at a temperature slightly above its fusing-point. A portion of the melted fat being drawn up into the capillary, the latter is removed and the fat allowed to solidify spontaneously. After an interval of not less than twelve hours, the capillary is attached by a rubber band to the stem of a delicate thermometer (preferably capable of being read to tenths of a degree), the portion of solidified fat being opposite the thermometer bulb. A test-tube containing water is held in the neck of a flask in such a manner as to be immersed in water contained in the flask, as shown in Fig. 93, the flask being held on the ring of a stand, with wire gauge interposed between flask and flame. The thermometer with attached capillary is then held immersed

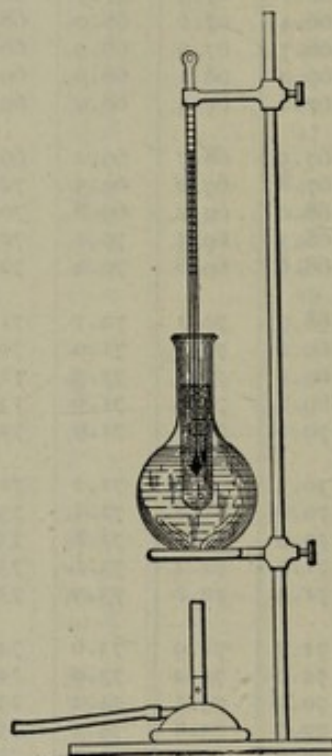


FIG. 93.

FIG. 93.—Apparatus for Determining Melting-point. Capillary tube with enclosed fat shown on the right, enlarged.

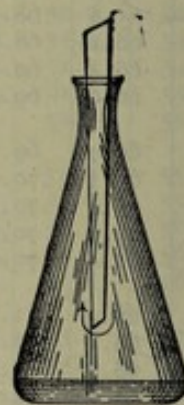


FIG. 94.

FIG. 94.—Reichert Flask with Card Inserted for Quick Evaporation.

in the water of the test-tube and below the level of the water in the flask, as shown. The water in the flask is then heated *very gradually*, so that the rise of temperature as shown by the thermometer does not exceed  $0.5^{\circ}\text{C}$ . per minute, the exact temperature at which fusion of the fat occurs being recorded as the melting-point.



The flame is then removed, and the temperature at which the fat solidifies is noted as the solidifying-point.

# MELTING AND SOLIDIFYING-POINTS OF EDIBLE OILS AND FATS.

Oil.	Melting-point.	Solidifying-point.
Linseed oil.....	.....	-25°
Poppy seed oil.....	.....	-18
Sunflower oil.....	.....	-19 to -16
Mustard oil.....	.....	-17 to -15
Maize oil.....	.....	-12 to -10
Soy oil.....	.....	-15 to -8
Cottonseed oil.....	.....	-5 to 0
Sesame oil.....	.....	-6 to -4
Peanut oil.....	.....	0 to +3
Rape oil.....	.....	-10 to +10
Olive oil.....	.....	-6 to +10
Lard oil.....	.....	

Fat.	Melting-point.	Solidifying-point.
Cocconut oil.....	20-28°	14-23°
Butter fat.....	28-36	19-24
Cocconut stearin.....	29	21-26
Palm kernel oil.....	23-30	20-27
Cacao butter.....	28-35	21-27
Palm kernel stearin.....	31-32	28
Oleo oil.....	30-39	
Lard.....	36-46	27-30
Cottonseed stearin.....	26-40	16-33
Beef tallow.....	42-49	27-35
Mutton tallow.....	44-49	32-41
Oleo stearin.....	44-54	40-50

The mean of two or three determinations is usually taken as the true melting and solidifying-points.

**Reichert-Meissl Process for Volatile Fatty Acids.**—This process has undergone various modifications from time to time. Reichert originally used 2.5 grams of fat, but Meissl, who improved the process, used 5 grams, so that the Reichert-Meissl number is now expressed on the basis of 5 grams of fat. The method is conveniently carried out as follows:

Five grams of the fat are transferred to a dry, clean Erlenmeyer flask of about 300 cc. capacity, 10 cc. of 95% alcohol are added, and 2 cc. of



sodium hydroxide solution (prepared by dissolving 100 grams of sodium hydroxide in 100 cc. of water). The flask with its contents is then heated on a water-bath with a funnel in the neck, which satisfactorily replaces the return-flow condenser originally prescribed. The heating is continued with occasional shaking till saponification is complete. This

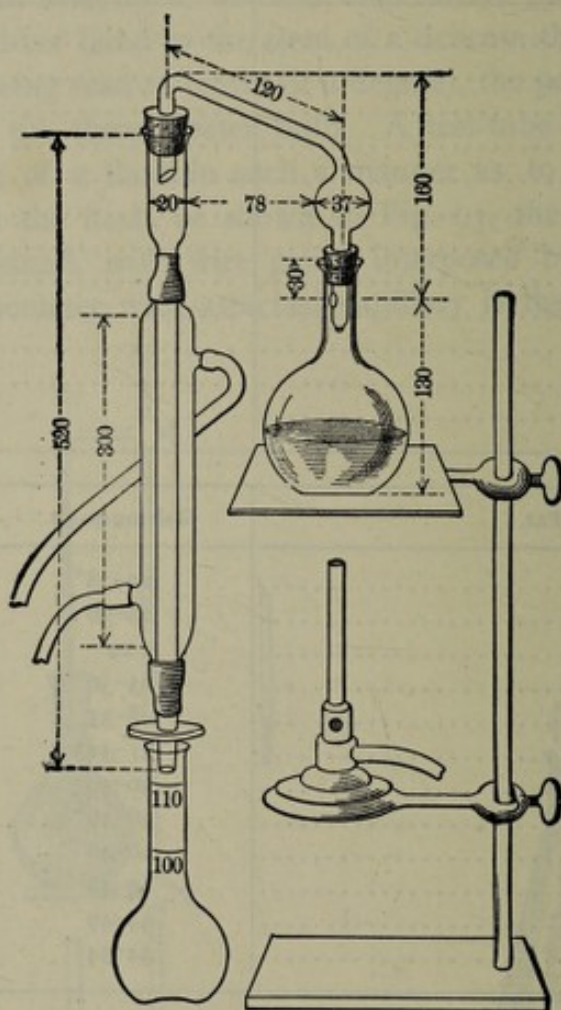


FIG. 95.—Apparatus for Reichert-Meissl and Polenske Distillation.

stage of the process is indicated by the appearance of the solution, which is then perfectly clear and free from fat globules.

The condenser-funnel being removed, the contents of the flask are next evaporated by continued heating over the bath to dryness. This may be hastened by inserting a card in the neck of the flask, as shown in Fig. 94, thus starting a circulatory movement to the air through the flask.

The dry soap thus formed is then dissolved by warming on the water-bath with 135 cc. of added water, shaking the flask occasionally. After



cooling, 5 cc. of dilute sulphuric acid (200 parts sulphuric acid in 1 liter of water) are added, and the fatty acid emulsion formed is melted by heating the flask on the water-bath, the flask being corked during the heating. The fatty acids are completely melted when they form an oily layer on the surface of the solution.

Scraps of pumice stone joined by platinum wires are next placed in the flask to prevent bumping, and the flask is properly connected with the condenser for distilling, as shown in Fig. 95. A flask graduated at 110 cc. is used as a receiver, the funnel placed therein being provided with a loose tuft of absorbent cotton to serve as a filter. The distillation is conducted by so grading the heat that the receiving flask is filled with the distillate in about thirty minutes.

Finally the entire distillate is titrated with decinormal sodium hydroxide, using 0.5 cc. of a solution of phenolphthalein as an indicator. The number of cubic centimeters of decinormal alkali required to neutralize the acidity of the distillate from 5 grams of the fat in the manner described expresses what is known as the Reichert-Meissl number.

*Leffmann and Beam's Modification.\**—Five grams of the fat placed in the flask are treated with 20 cc. of a solution of soda in glycerin (20 cc. of a 50% solution of sodium hydroxide in 180 cc. of glycerin), heating the flask till the contents are completely saponified. The solution becomes perfectly clear, showing complete saponification in about five minutes, after which 135 cc. of water are added to the clear soap solution, at first drop by drop to prevent foaming; 5 cc. of the dilute sulphuric acid are then added, and the distillation conducted at once without first melting the fatty acids.

**Polenske Number.†**—This number represents the volatile fatty acids insoluble in water, and is of value in detecting cocoanut and palm kernel oils in butter and other fats. The details of apparatus and manipulation here described should be closely adhered to in order to secure comparable results. The Reichert-Meissl, Polenske, and Jensen-Kirschner numbers may be determined in one weighed portion of the fat. The method of saponification is that devised by Leffmann and Beam.

Place 5 grams of the clear filtered fat in a 300-cc. Jena flask, add 20 grams of glycerine and 2 cc. of a 50% solution of sodium hydroxide. Heat the flask on a wire gauze until the contents are completely saponified, which requires about 5 minutes, and is indicated by the clearing up of the liquid. While still hot add 90 cc. of boiled water, at first drop by drop

\* Analyst 16, 1891, p. 153.

† Polenske, Zeits. Unters. Nahr. Genussm., 7, 1904, p. 274. Fritsche, *ibid.*, p. 193.



to prevent foaming, and shake until the soap is dissolved. The solution should be completely clear and almost colorless. Rancid or oxidized fats that yield a brown soap solution should not be examined.

To the soap solution, warmed to  $50^{\circ}$ , add 50 cc. of dilute sulphuric acid (25 cc. : 1 liter) and 0.5 gram of granulated pumice stone with grains 1 mm. in diameter, then connect with the distilling apparatus shown in Fig. 95. Distil over a 0.5-mm. mesh copper gauze,\* using a Bunsen flame so regulated as to give a distillate of 110 cc. in 19-20 minutes, and a stream of water that will cool the distillate to about  $20-23^{\circ}$ . The room should have a temperature of about  $18-22^{\circ}$ . As soon as 110 cc. have come over, replace the flask by a 25-cc. measuring cylinder.

Without mixing the distillate place the flask for 10 minutes in water at  $15^{\circ}$ , so that the 110-cc. mark is about 3 cm. below the surface of the water. After the first 5 minutes, gently move the neck of the flask in the water so that the fatty acids floating on the surface come in contact with the glass, noting at the end of 10 minutes the condition of these acids. If the butter is pure, the floating acids are either solid or form a half solid turbid mass, according as the Reichert-Meissl number is high or low; if it is adulterated with 10% or more of cocoanut oil, they form transparent oil drops. Stopper the 110-cc. flask, mix by inverting 4 or 5 times, avoiding violent shaking, filter through an 8-cm. dry filter fitted close to the funnel, titrate 100 cc. of the liquid with N/10 barium hydroxide solution, and multiply by 1.1, thus obtaining the Reichert-Meissl number.

After the last drop of distillate has passed through the filter, wash with three 15-cc. portions of water, each of which has previously been used to rinse the condenser tube, the measuring cylinder, and the 110-cc. flask. Then repeat this treatment, using 15-cc. portions of neutral 90% alcohol. Titrate the united alcoholic washings with tenth-normal barium hydroxide solution, using phenolphthalein as indicator. The number of cc. required is the Polenske number.

The following results illustrate the value of the method:

	Reichert-Meissl Number.	Polenske Number.
31 samples of butter (Polenske).....	23.3-30.1	1.5-3.0
4 samples of cocoanut oil (Polenske).....	6.8-7.7	16.8-17.8
Oleomargarine (Arnold).....	0.5	0.53
Lard (Arnold).....	0.35	0.5
Tallow (Arnold).....	0.55	0.56

\* Lewkowitsch recommends a circular piece of asbestos 12 cm. in diameter with a hole in the center 5 cm. in diameter and warns against overheating. He was unable to secure uniform heating with gauze.



**Jensen-Kirschner Number.\***—This number ("Kirschner value"), which is a measure of the butyric acid content, is recommended by Bolton and Revis † as the only available means of detecting butter in oleomargarine containing cocoanut oil. They give tentatively Jensen-Kirschner numbers 20–26 for butter as corresponding to Polenske numbers 1.6–3.2, also Jensen-Kirschner numbers 1.6–1.9 for cocoanut oil and 1.1 for palm oil.

Add to the 100 cc. of distillate, neutralized for the determination of the Reichert-Meissl number (page 500), 0.5 gram of powdered silver sulphate, allow to stand for an hour with occasional shaking, filter, pipette 100 cc. of the filtrate into a distillation flask, add 35 cc. of water, 10 cc. of 2.5% sulphuric acid, and a long piece of aluminum wire, then distil as in the Reichert-Meissl-Polenske method, collecting 110 cc. of distillate in 20 minutes. Titrate 100 cc., correct for a blank determination, and calculate the Jensen-Kirschner number by the following formula:

$$K = \frac{121X(100 + Y)}{10,000}$$

in which  $X$  = cc. of  $N/10$  alkali used in the Jensen-Kirschner titration, corrected for blanks, and  $Y$  the cc. of  $N/10$  alkali used in the Reichert-Meissl titration.

**Determination of Soluble and Insoluble Fatty Acids.—Jones Method.‡**

—*Soluble Acids.*—Five grams are weighed out and transferred to an Erlenmeyer flask of the same size and in the same manner as that used for the Reichert-Meissl process. Fifty cc. of alcoholic potash solution are added (40 grams of potassium hydroxide in 1 liter of 95% redistilled alcohol) and the flask, provided with a return-flow condenser, is heated on the water-bath till saponification is complete, as evidenced by the clear solution free from fat globules. The alcoholic solution of potash is preferably measured from a pipette, from which it is allowed to drain for a noted interval of time, say thirty seconds.

After complete saponification, the condenser is removed and the alcohol is evaporated by further heating. One or more blanks are prepared at the same time, using the same 50-cc. pipette for measuring, and

\* Zeits. Unters. Nahr. Genussm., 9, 1905, p. 65.

† Analyst, 36, 1911, p. 333; 37, 1912, p. 183. Fatty Foods, Phila., 1913, p. 120.

‡ Analyst, 3, 1878, p. 19.



applying the same time limit for draining the pipette. The blanks are first titrated, after evaporation, with half-normal hydrochloric acid, using phenolphthalein as an indicator. Then add to the flask containing the fatty acids 1 cc. more of the half-normal acid than is found necessary to neutralize the alkali in the blanks, after which heat the flask again with a funnel in the neck till the fatty acids have completely separated in a layer on top of the solution. Then cool the flask in ice water till the fatty acids are solidified, after which decant the liquid portion through a filter, previously dried in the oven and weighed, into a liter flask, keeping the solid mass of fatty acids intact. Next add 200 or 300 cc. of hot water to the flask containing the fatty acids, and again melt over the water-bath till they collect as before on top, having again inserted the funnel to act as a condenser, and occasionally shaking the contents of the flask during heating. Cool as before in ice water, after which again decant the liquid from the solid mass through the same filter into the liter flask. Repeat this process of washing, melting, cooling, and decanting three times, receiving all the wash water through the same filter in the same flask. Make up the washings with water to the liter mark, and, after mixing, two portions of 100 cc. each are titrated with tenth-normal sodium hydroxide, using phenolphthalein for an indicator. Each reading is multiplied by ten to represent the total volume, and the figure thus obtained represents the number of cubic centimeters of tenth-normal alkali necessary to neutralize the acidity of the soluble fatty acids, together with the excess of half-normal acid used, amounting to 1 cc. This 1 cc. of half-normal acid corresponds to 5 cc. of tenth-normal alkali, hence 5 cc. are to be deducted from the total number of cubic centimeters required for the titration, the corrected figure thus obtained being multiplied by the factor 0.0088, which gives the weight of soluble fat acids in the 5 grams of the sample, calculated as butyric acid.

*Hehner Method.\*—Insoluble Acids.*—Transfer the fatty acids left in a cake in the flask from the separation of the soluble acids, to a weighed glass evaporating dish, using strong alcohol to wash them out thoroughly. Dry the filter used in the separation, transfer it to an Erlenmeyer flask, and thoroughly wash it with strong alcohol, transferring all the washings to the dish. The alcohol is then evaporated by placing the dish on the water-bath, after which it is dried for 2 hours in the air-oven at 100°, cooled

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\* Zeits. anal. Chem., 16, 1877, p. 145.



in the desiccator, and weighed. After once heating for 2 hours, cooling and weighing, heat again for half an hour, cool, and weigh. If a considerable loss in weight is found, heat for an additional half-hour. It is best, however, to avoid too prolonged heating, lest oxidation of the fatty acids should produce an increase in weight.

#### INSOLUBLE FATTY ACIDS OF EDIBLE OILS AND FATS.

Mustard oil.....	96.2-95.1
Cottonseed oil.....	96 -95
Corn oil.....	96 -93
Lard.....	96 -93
Peanut oil.....	95.8
Sesame oil.....	95.7
Beef tallow.....	95.6
Mutton tallow.....	95.5
Poppyseed oil.....	95.2-94.9
Rape oil.....	95.1
Sunflower oil.....	95
Olive oil.....	95
Cocoa butter.....	94.6
Cocoanut oil.....	90 -88.6
Butter.....	89.8-86.5

**Saponification Number.**—*Koettstorfer's Method.*—By the saponification number is meant the number of milligrams of potassium hydroxide necessary to completely saponify 1 gram of the fat. Between 1 and 2 grams of the fat are transferred in the usual manner (see p. 489) to an Erlenmeyer flask, and 25 cc. of the alcoholic potash solution (40 grams of potassium hydroxide free from carbonates in 1 liter of 95% alcohol redistilled after standing for some time with potassium hydroxide) are added with a graduated pipette, which is allowed to drain for a noted period of time, say 30 seconds. The determination should preferably be made in duplicate. Conduct the saponification as in the case of the soluble fatty acids by heating on the water-bath. After saponification, remove from the bath, cool, and titrate with half-normal hydrochloric acid, using phenolphthalein as an indicator. Titrate also several blanks in which 25 cc. of the alcoholic potash solution are measured out with the same pipette as before, and allow to drain for the same amount of time. Subtract the number of cubic centimeters of half-normal acid necessary to neutralize the alkali in the case of the saponified fat from that necessary to neutralize the blank, multiply the result by 28.06, and divide the product by the number of grams of fat taken.



## SAPONIFICATION NUMBER OF EDIBLE OILS AND FATS

Oil.	Saponification No.
Mustard oil.....	170-178
Rape oil.....	168-179
Sesame oil.....	188-193
Sunflower oil.....	188-194
Maize oil.....	189-194
Soy oil.....	189-194
Cottonseed oil.....	191-195
Olive oil.....	185-196
Peanut oil.....	186-196
Linseed oil.....	190-196
Poppyseed oil.....	189-197
Lard oil.....	190-198

Fat.	Saponification No.
Mutton tallow.....	192-195
Cottonseed stearin.....	195
Oleo stearin.....	192-197
Beef tallow.....	193-200
Cacao butter.....	192-202
Oleo oil.....	198-202
Lard.....	193-203
Butter.....	220-241
Palm kernel stearin.....	242
Palm kernel oil.....	242-255
Cocoanut stearin.....	251-257
Cocoanut oil.....	246-268

**The Iodine Absorption Number.**—This determination is based on the well-known property of the unsaturated fatty acids to absorb a fixed amount of iodine under given conditions of time, strength of reagent, etc.

*Hübl's Method.\**—The following reagents are necessary:

(1) *Iodine Solution*, made by dissolving 26 grams of pure iodine in 500 cc. of 95% alcohol, and, separately, 30 grams of mercuric chloride in 500 cc. of the same strength of alcohol. Filter the latter solution, if necessary, and mix the two together, allowing the mixture to stand at least 12 hours before using. As the solution loses strength rapidly, it should not be used in accurate work after it is 24 hours old.

(2) *Decinormal Thiosulphate Solution*, made by dissolving 24.8 grams of the freshly powdered, chemically pure salt in water, and making up to 1 liter.

\* Dingler's Polyt. Jour., 25, 1884, p. 281.

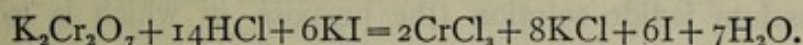


(3) *Starch paste*, prepared by boiling 1 gram of starch in 200 cc. of water for ten minutes, then cooling.

(4) *Potassium Iodide Solution*, made by dissolving 150 grams of the salt in water, and making up the volume to 1 liter.

(5) *Potassium Bichromate Solution* for standardizing the thiosulphate, made by dissolving 3.874 grams of chemically pure potassium bichromate in distilled water, and making up the volume to 1 liter.

The sodium thiosulphate solution is standardized as follows: 20 cc. of the potassium bichromate solution are introduced into a glass-stoppered flask together with 10 cc. of potassium iodide and 5 cc. of strong hydrochloric acid. Then slowly add from a burette the sodium thiosulphate solution, till the yellow color of the solution has nearly disappeared, after which a little of the starch paste is added, and the titration carefully continued to just the point of disappearance of the blue color. The reaction which takes place is as follows:



The equivalent of 1 gram of iodine in terms of the thiosulphate solution is found by multiplying the number of cubic centimeters of the latter solution required for the above titration by 5.

If, for example, 16.4 cc. of the thiosulphate solution are required for 20 cc. of the bichromate solution, then 1 gram of iodine is equivalent to  $16.4 \times 5 = 82.0$  cc. of sodium thiosulphate solution, or 1 cc. of the thiosulphate solution  $= \frac{1}{82} = 0.0122$  gram of iodine. 1 cc. of exactly decinormal thiosulphate is theoretically equivalent to 0.0127 gram of iodine.

The thiosulphate solution may also be standardized by means of iodine. A short tube closed at one end is tared, together with another tube of such a size as to fit over the first. Into the inner tube are introduced about 0.2 gram of resublimed iodine and the tube heated until the iodine melts, after which it is closed by the second tube and the whole cooled in a desiccator and weighed. The iodine is dissolved in 10 cc. of 10% potassium iodide solution, the solution diluted with water, and the thiosulphate solution added with constant stirring until only a yellow color remains. Starch paste is then added, and the titration continued until the blue color disappears.

*Manipulation.*—Place 0.4 to 1 gram of the solid fat, or from 0.2 to 0.4 gram of oil, in a glass-stoppered flask or bottle of 300 cc. capacity.



In the case of oils, this may conveniently be done by difference, weighing first a small quantity of the oil in a beaker with a short piece of glass tubing to serve as a pipette, transferring a number of drops of the oil from the beaker to the bottle, and again weighing the beaker and contents. The number of drops of oil required for the desired weight is first ascertained experimentally.

The material may also be conveniently and accurately weighed in small, flat bottomed cylinders of glass about 10 mm. in diameter and 15 mm. high, which may be made by cutting off so-called "shell vials." Fats are introduced while melted, the weight being taken after cooling. The cylinder and fat are transferred together by means of forceps to the glass-stoppered bottle.

Dissolve the oil in 10 cc. of chloroform, and after solution has taken place, add 30 cc. of the iodine solution, shake, and set in a dark place for three hours, shaking occasionally. The excess of iodine should be at least as much as is absorbed. When ready for the titration, add 20 cc. of the potassium iodide solution (the purpose of which is to keep in solution the mercuric iodide formed, which would otherwise precipitate on dilution) and 100 cc. of distilled water. Titrate the excess of iodine by the thiosulphate solution, which is slowly added from a burette till the yellow color has nearly disappeared, then add a little starch paste, and finally thiosulphate solution drop by drop until the blue color of the iodized starch is dispelled. Near the end of the reaction the flask should be stoppered and vigorously shaken, in order that all the iodine may be taken up, and sufficient thiosulphate should be added to prevent a reappearance of any blue color in five minutes.

Two blanks are conducted at the same time and in similar flasks or bottles, in exactly the same manner as in the case of the above titration, except that the fat is omitted. This is to get the true value of the iodine solution in terms of the thiosulphate solution.

Suppose, for example, in the case of the blanks, 30 cc. of the iodine solution required in one instance, 46.2 cc. of sodium thiosulphate solution and in the other 46.4 cc. The mean is 46.3. Suppose 30.7 cc. of thiosulphate solution were required for the excess of iodine remaining over and above that absorbed by 0.5 gram of the fat in the above process. Then the thiosulphate equivalent to the iodine absorbed by the fat would be  $46.3 - 30.7 = 15.6$  cc., and the per cent of iodine absorbed would be

$$\frac{15.6 \times 0.0122 \times 100}{0.5} = 38.06.$$



## IODINE NUMBER OF EDIBLE OILS AND FATS.

Oil.	Iodine No.
Lard oil.....	67- 88
Olive oil.....	77- 95
Peanut oil.....	83-105
Rape oil.....	94-105
Sesame oil.....	103-117
Cottonseed oil.....	104-117
Mustard oil.....	92-122
Maize oil.....	116-130
Sunflower oil.....	120-135
Soy oil.....	121-143
Poppyseed oil.....	132-143
Linseed oil.....	170-202

Fat.	Iodine No.
Cocoanut stearin.....	4- 6.6
Palm kernel stearin.....	8
Cocoanut oil.....	8- 9.5
Palm kernel oil.....	13- 18
Oleo stearin.....	8- 27
Butter fat.....	26- 38
Cacao butter.....	32- 41
Beef tallow.....	35- 45
Mutton tallow.....	32- 50
Oleo oil.....	40- 50
Lard.....	54- 70
Cottonseed stearin.....	88-104

The Hübl method was long almost universally used for estimating the per cent of iodine absorbed, but is open to serious objections, chief of which are the tendency of the iodine solution to lose strength, and the length of time required to insure saturation of the oil with the iodine.

The Wijs and Hanus modifications obviate these defects, the former being quite generally used in Europe and to a considerable extent in America, and the latter being the official method of the A. O. A. C.

Tolman and Munson\* have shown that with oils and fats having iodine numbers below 100, the three methods give practically identical figures, while with oils having high iodine numbers, the Wijs and Hanus modifications give higher results than the Hübl method, but are doubtless more nearly correct. Their results follow:

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\* Jour. Am. Chem. Soc., 25, 1903, p. 244.



Number of Analyses.		Hübl's Number (3 hours).	Wijs's Number (30 min.).	Hanus' Number (30 min.).	Difference between Wijs and Hübl.	Difference between Hanus and Hübl.
1	Cocoanut oil.....	8.93	9.05	8.60	+0.12	-0.33
2	Butter— minimum .....	34.8	35.9	35.4	+1.1	+0.6
	maximum. ....	35.3	36.2	35.3	+0.9	+0.0
1	Oleo oil. ....	42.6	43.5	43.3	+0.9	+0.7
4	Olcomargarine—minimum .....	52.5	52.9	52.0	+0.4	-0.5
	maximum. ....	66.3	66.0	64.8	-0.3	-1.5
2	Lard oil— minimum .....	69.3	70.5	69.8	+1.2	+0.5
	maximum. ....	73.7	74.5	73.9	+0.7	+0.2
36	Olive oil— minimum .....	79.2	79.9	80.6	+0.7	+1.4
	maximum. ....	89.8	91.4	90.0	+1.6	+0.2
	average .....	84.0	85.3	84.6	+1.3	+0.6
3	Peanut oil— minimum .....	94.5	95.2	94.1	+0.7	-0.1
	maximum. ....	107.7	109.5	107.7	+1.8	+0.0
5	Mustard oil— minimum .....	98.4	104.3	103.8	+5.9	+5.4
	maximum. ....	113.0	118.2	116.8	+5.2	+3.8
2	Rape oil— minimum .....	100.2	104.1	102.8	+3.9	+2.6
	maximum. ....	101.3	105.7	105.2	+4.4	+3.8
1	Sunflower oil .....	106.4	109.2	107.2	+2.8	+0.8
3	Cottonseed oil— minimum .....	103.8	105.3	105.2	+1.5	+1.4
	maximum. ....	106.2	107.3	107.8	+1.1	+1.6
1	Sesame oil. ....	106.4	107.0	106.5	+0.6	+0.1
3	Corn oil— minimum .....	119.0	122.2	119.6	+3.0	+0.4
	maximum. ....	123.3	129.2	126.0	+5.8	+2.7
2	Poppyseed oil— minimum .....	133.4	135.2	132.9	+1.8	-0.5
	maximum. ....	134.9	139.1	138.4	+4.2	+3.5

*Hanus Modification.\*—Reagents.—Iodine Solution.*—Dissolve 13.2 gms. of pure iodine in 1 liter of pure glacial acetic acid (99%), and to the cold solution add 3 cc. of bromine, or sufficient to practically double the halogen content when titrated against the thiosulphate solution, but with the iodine slightly in excess.

*Decinormal Thiosulphate Solution, Starch Paste, and Potassium Iodide Solution,* as in Hübl's method.

*Method of Procedure.*—Proceed as in Hübl's method, substituting 30 cc. of the Hanus iodine reagent for that of Hübl, stirring the solution before adding the water, and, instead of adding 20 cc. of the potassium iodide solution, use only 15 cc. The excess of iodine should be at least 60% of that added.

\* Zeits. Unters. Nahr. Genussm., 4, 1901, p. 913.



Only half an hour is required for full saturation of the oil by the iodine in the Hanus method, as against three hours in the Hübl. In case of the non-drying oils and fats, the reaction takes place in from eight to fifteen minutes, though it is best to let the flask set for half an hour at least, in all cases. With oils having an iodine number in excess of 100, Tolman and Munson recommend one hour's standing.

On account of the high coefficient of expansion of acetic acid, care should be taken that the temperature is the same when the iodine solution is measured for the blank and for the determination, as otherwise a serious error may be introduced.

*Wijs Modification.\*—Reagents.—Iodine Solution.*—Dissolve 13.2 grams of pure iodine in 1 liter of pure glacial acetic acid, and pass through the larger portion of this solution a current of carefully washed and dried chlorine gas † until the solution is practically decolorized. Finally add enough of the original solution of iodine in acetic acid to restore the iodine color, so that there is a slight excess of iodine.

*Hunt's Modified Iodine Solution.*—Dissolve 10 grams of iodine trichloride in 1 liter of pure glacial acetic acid, and finally add and dissolve 10.8 grams of pure iodine.

*Other Reagents,* as in the Hübl and Hanus methods.

*Method of Procedure.*—Proceed as in the Hanus method, observing the same precautions, the only difference being in the use of the Wijs iodine reagent.

Wijs recommends the following periods of time for absorption of the iodine: For non-drying oils and fats, such as peanut, olive, and cocoanut oils, butter fat, lard, and other animal fats, 15 minutes; for semi-drying oils, such as cottonseed, rape, sesame, corn, and mustard, 30 minutes; for drying oils, such as sunflower and poppyseed, 1 hour.

**The Bromine Index or Bromine Absorption Number.**—The measure of the amount of bromine absorbed by the oils and fats is a useful factor. By the bromine index is understood the weight of bromine which is absorbed by 1 gram of a given oil. The bromine index of various oils has been determined as follows:

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\* Ber. d. chem. Ges., 31, 1898, p. 750.

† The chlorine is conveniently prepared by treatment of bleaching powder with dilute sulphuric acid, using gentle heat, and washing the gas by passing through strong sulphuric acid.



	Bromine Index.	Observer.
Poppyseed.....	0.835	Levallois
Mustard.....	0.763	Girard
Sesame.....	0.695	Levallois
Cottonseed.....	0.645	"
Rape.....	0.632	Girard
Peanut.....	0.530	Levallois
Olive.....	0.500 to 0.544	"

The following methods are at present seldom used:

*Method of Levallois.\**—Five grams of the oil are saponified with alcoholic potash in a 50-cc. graduated flask by the aid of a gentle heat. At the end of the saponification and after cooling, the flask is filled to the mark with alcohol, and, after shaking, 5 cc. are removed by means of a pipette and transferred to a flask. A slight excess of hydrochloric acid is added to set free the fatty acids, and from a burette a standardized solution of bromine water is run in till with constant shaking a permanent yellow color persists. The bromine is previously standardized with potassium iodide and sodium thiosulphate. The weight of bromine fixed by 1 gram of the fat is then calculated.

*Mill's Method.—Modified.†*—Dissolve 0.1 gram of the filtered and dried fat in 50 cc. of carbon tetrachloride or chloroform in a 100-cc. stoppered bottle. From a burette a standard solution of bromine in carbon tetrachloride, approximately tenth-normal (8 grams to a liter), is slowly added to the oil solution till, after fifteen minutes, a permanent coloration remains. The amount of bromine absorbed is calculated by comparing with the color similarly produced in a blank experiment, or an excess of bromine solution may be run in and the solution titrated back with a standard solution of thiosulphate, using potassium iodide and starch.

**Thermal Tests.**—The rise in temperature produced by the action of certain reagents on various oils and fats, when applied in a definite manner, has been found to be of considerable value, especially in the case of sulphuric acid and of bromine.

\* Villiers et Collin, *Les Substances Alimentaires*, p. 680.

† Jour. Soc. Chem. Ind., 2, 1883, p. 435; 3, 1884, p. 366. See also McIlhiney, Jour. Am. Chem. Soc., 16, 1894, p. 275; 21, 1899, p. 1084.



**The Maumené Test,\*** or thermal reaction with sulphuric acid, is most readily carried out in a beaker of say 150 cc. capacity, which is set into a larger beaker or vessel of any kind, the space between the two being packed with felt or cotton waste. The inner beaker is removed, and into it is weighed 50 grams of the oil. It is then replaced and the packing adjusted, if necessary, after which the temperature of the oil is noted with a thermometer. From a burette containing the strongest sulphuric acid of the same temperature as the oil, 10 cc. are run into the beaker, at the same time stirring the mixture of acid and oil with the thermometer. The temperature rises somewhat rapidly, and remains for an appreciable time at its maximum point, which should be noted. The difference in degrees centigrade between the initial temperature of the oil and the maximum temperature of the mixture expresses the Maumené number.

With certain oils, as cottonseed, considerable frothing ensues when concentrated acid is employed, making an accurate determination of the Maumené number somewhat difficult. In this case it is better to employ a somewhat weaker acid, and to express results in terms of what is called the "specific temperature reaction." This is the result obtained by dividing the rise of temperature in the case of the oil by the rise of temperature in the case of water, using the same strength of acid, and multiplying the quotient by 100. Indeed, it is of importance in all cases to compare results on oils with those obtained by carrying out the same test on water.

**Bromination Test.**—This test depends upon the avidity with which the oils and fats absorb bromine, the rise in temperature caused by the reaction being measured in this case rather than the actual *amount* of bromine absorbed, as in the case of the iodine absorption. Indeed, there is such a close relation between the iodine number and the heat of bromination, that when one is determined the other may be calculated quite closely by multiplying by a factor. In view of the fact that the heat of bromination is much more readily determined than the iodine number, it is often convenient to calculate the latter from the former, the result in the case of the edible oils and fats being quite sure to fall within the limits of variation of the iodine number of different oils of the same class. The bromination test was devised by Hehner and Mitchell,† who employed a vacuum jacketed tube for a calorimeter in which to make the test. Various modifications have been suggested both in the

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\* Maumené, Compt. Rend., 35, 1852, p. 572.

† Analyst, 20, 1895, p. 146.



apparatus employed and in the manner of diluting the oil and applying the reagent. The calorimeter employed by Gill and Hatch,\* Fig. 96, is conveniently made and is very satisfactory. It consists of a long, narrow, flat-bottomed tube, held by a cork in a small beaker, in such a manner that it is surrounded by an air jacket. The small beaker is set into one of larger size, the space between the two being packed with cotton waste. Five grams of the oil or fat are dissolved in 25 cc.

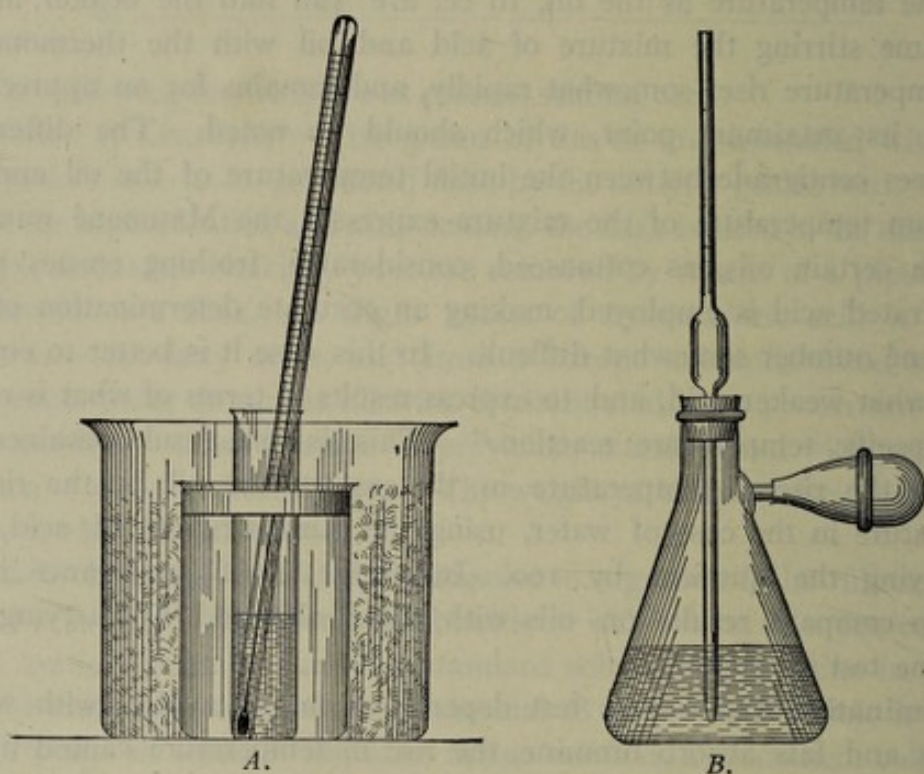


FIG. 96.

A. Gill and Hatch's Calorimeter for the Bromination Test with Oils.

B. Wiley's Pipette for Measuring Bromine in Chloroform.

of chloroform or carbon tetrachloride, and 5 cc. of this solution (containing 1 gram of the oil) are transferred by a pipette to the inner tube of the above calorimeter, being careful not to let it flow down the sides of the tube. The temperature of the oil is then taken by a thermometer graduated to  $0.2^{\circ}$ . The bromine reagent, which should be freshly prepared, is made up by measuring from a burette one part by volume of bromine into four parts of chloroform or carbon tetrachloride. The reagent is transferred to a measuring-flask devised by Wiley,† consisting of a side-necked filter-flask provided with a per-

\* Jour. Am. Chem. Soc., 21, 1899, p. 27. Gill, Oil Analysis, p. 50.

† Jour. Am. Chem. Soc., 18, 1896, p. 378.



forated rubber stopper into which the stem of a 5-cc. pipette is fitted, Fig. 96. A bulb on the side-neck serves to fill the pipette. This pipette, filled to the mark with the bromine reagent (which should be at the same temperature as the oil solution in the calorimeter), is first covered by the finger and removed, and its contents of 5 cc. allowed to flow down the sides of the inner tube of the calorimeter and mingle with the oil without stirring. The rise in temperature is very quick, and the highest point is noted. The difference between the highest and the initial temperature constitutes the heat-of-bromination number.

This number, in the case of Gill and Hatch's calorimeter, is somewhat lower than when a vacuum jacketed tube is employed, and differs somewhat with the diluent of the oil and bromine. In spite of these variations and that due to the personal equation, concordant results may be obtained with the various oils, when the method is carried out under precisely the same conditions. The analyst should carefully work out the test several times with a particular oil till the results agree, and should then with equal care determine the iodine number of the same oil. The iodine number, divided by the heat-of-bromination number, gives the factor which is to be employed under the same conditions for calculating one constant from the other. In the case of Hehner and Mitchell's work with the vacuum tube, measuring 1 cc. of undiluted bromine into 1 gram of oil dissolved in 10 cc. of chloroform, it was found that the factor to be used in calculating the iodine number was 5.5.

The following are some of the results on edible oils obtained by Hehner and Mitchell:

Oil.	Heat of Bromination.	Iodine Number.	Calculated Iodine Number.
Lard .....	10.6	57.15	58.3
Butter .....	6.6	37.07	36.3
Olive oil.....	15	80.76	82.5
Corn oil. ....	21.5	122	118.2
Cottonseed oil.....	19.4	107.13	106.7

As in the case of the Maumené test with sulphuric acid (wherein the rise in temperature of sulphuric acid and water is taken as a standard), it is convenient to employ some standard for the bromination test, whereby varying results due to difference in apparatus, etc., may be compared.

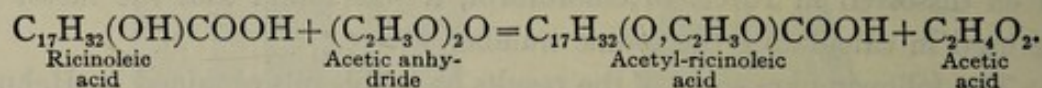
In this case Gill and Hatch found that sublimed camphor may be prepared sufficiently pure to be used for such a standard. Applying the bromination test with their calorimeter, as above described, to 5 cc. of a



solution of  $7\frac{1}{2}$  grams of camphor in 25 cc. of carbon tetrachloride, an average rise in temperature of  $4.2^{\circ}$  was obtained, and the specific temperature reaction is calculated for each oil by dividing the heat of bromination by this number. Furthermore, by dividing the iodine number of several oils by this specific temperature reaction, the factor to be employed for the calculation of the iodine number was found to be 17.18, as in the following cases:\*

Oil.	Specific Temperature Reaction.	Iodine Number.	
		Calculated.	Found.
Prime lard .....	3.705	63.8	63.8
No. 1 lard.....	4.096	70.3	73.9
Olive. ....	4.762	81.8	82.0
Cottonseed .....	5.667	97.3	103.0
Corn. ....	6.381	109.5	107.8

**The Acetyl Value.**—On heating fats with acetic anhydride they become “acetylated”; i.e., the hydrogen atom of their alcoholic hydroxyl group is exchanged for the acetic acid radicle, in accordance, for example, with the following reaction:



By the actyl value is meant the number of milligrams of potassium hydroxide necessary to neutralize the acetic acid formed by the saponification of 1 gram of the acetylated fat.

*The Lewkowitsch method* of procedure follows:† 10 grams of the oil are boiled with twice that weight of acetic anhydride for 2 hours in a flask with a return-flow condenser, and the mixture is then transferred to a large beaker containing 500 cc. of water, and boiled for 2 hours. To prevent bumping, a current of carbon dioxide is slowly passed through it during the boiling, introduced through a finely drawn, bent glass tube reaching nearly to the bottom of the beaker. The mixture on standing separates into two layers, of which the lower, or aqueous layer, is si-

\* Gill, Oil Analysis, p. 128.

† Jour. Soc. Chem. Ind., 16, 1897, p. 503.



phoned off, and the oily layer boiled with fresh portions of water, which are in turn siphoned off, the operation being repeated till the wash water tests free from acid by litmus paper.

The acetylated fat is then separated and dried by filtering through a dry paper at  $100^{\circ}$  in an oven. If desired, the process may be carried out quantitatively, weighing the acetylated fat on a tared paper or in a tared dish as in the case of the insoluble acids, page 502.

About 5 grams of the acetylated fat are weighed into a flask, and saponified with alcoholic potash in precisely the same manner as for the determination of the saponification number. Evaporate the alcohol and dissolve the soap in water. One of two methods may be carried out for freeing the acetic acid for titration, one by distillation and the other by filtration. In either case the water used must be boiled until free from carbon dioxide.

For the former or distillation process, acidify the aqueous solution of the soap with 1 : 10 sulphuric acid, and distil in a current of steam until 600 to 700 cc. of distillate are obtained. The distillate should be received in a funnel with a loose cotton plug, so as to filter it free from insoluble acids mechanically carried over. The filtrate is titrated with tenth-normal sodium hydroxide, using phenolphthalein as an indicator. The number of cubic centimeters of alkali used is multiplied by 5.61, and the product divided by the number of grams of acetylated fat taken. The result is the acetyl value.

If the filtration process is used (which is more rapid and should give concordant results with the distillation process), the exact amount of alcoholic potash used in the saponification should be accurately measured in carrying out the former part of the test, and the exact number of cubic centimeters of standard acid corresponding to the amount of alkali employed should be added to the aqueous soap solution. The mixture should be gently warmed, and the fatty acids will gather in a layer at the top. These are filtered off and washed, till free from acid, with boiling water. The filtrate is titrated with tenth-normal sodium hydroxide, and the acetyl value calculated as in the distillation process.

#### ACETYL NUMBER OF EDIBLE OILS AND FATS.

Lard .....	2.6
Cacao Butter.....	2.8
Linseed Oil. ....	4.0
Palm Kernel Oil.....	1.9 - 8.4



Butter Fat.....	1.9- 8.6
Beef Tallow.....	2.7- 8.6
Peanut Oil.....	9.1
Olive Oil.....	10.6
Cocanut Oil.....	0.9-12.3
Rape Oil.....	14.7
Cottonseed Oil.....	21.0-25.0

*Holland*\* has simplified the process and adopted as the rational acetyl number the milligrams of potassium hydroxide required for the saponification of the acetyl assimilated by 1 gram of the fat on acetylation (not 1 gram of the acetylated fat) thus making the number analogous to the other common fat constants. His process is as follows:

Heat 5 grams of the sample with 10 cc. of acetic anhydride for 1-1.5 hours in a 300-cc. Erlenmeyer flask under a reflux condenser on a boiling water-bath. Add sufficient ceresine to form a solid disk on cooling (0.4-0.5 gram for butter, less for hard fats, more for oils), heat on the water-bath with rotation until the mass is homogeneous, add carefully 150 cc. of boiling water and heat further with occasional agitation to remove occluded acetic acid. Cool in water, decant off the solution without disturbing the cake onto a dense ether-extracted filter. Repeat the treatment with water about 6 times or until the final filtrate gives a decided color with 2-3 drops of N/10 alkali and phenolphthalein.

Drain in a cool place, return to the flask small particles adhering to the filter, add 50 cc. of alcoholic potash (50 cc. saturated solution to 1000 cc. of alcohol), 50 cc. of alcohol, a few glass beads, and boil under a reflux condenser on a water-bath for 60 minutes, or to complete saponification. Place in water at 60° C. and titrate with N/2 hydrochloric acid using as indicator phenolphthalein or preferably 1 cc. of a solution of alkali blue (6B) prepared by digesting 1 gram with 100 cc. of alcohol for several days at room temperature in a stoppered flask, with occasional shaking, and filtering. Boil and retitrate if necessary. Conduct blanks without ceresine. Calculate the saponification number of the fat after acetylation from these data.

The acetyl number (in the new sense) is the difference between the saponification numbers of the fat before and after acetylation.

**The Valenta Test.**†—This depends upon the solubility of the oil in

\* Jour. Ind. Eng. Chem., 6, 1914, p. 484.

† Dingler's polyt. Jour. 252, 1884, p. 296.



glacial acetic acid. Pour from 3 to 5 cc. of the oil into a test-tube, and add an equal volume of glacial acetic acid (specific gravity 1.0562). Place a thermometer in the tube and warm gently till the oil goes into solution. Then allow the mixture to cool, and observe the temperature at which the solution begins to appear turbid.

Castor oil and oil of the olive kernel are soluble in glacial acetic acid at ordinary temperatures, rape, mustard seed, and other cruciferous oils are partly insoluble even in the boiling acid, while the other edible oils and fats become turbid at temperatures between 23° C. and the boiling-point of acetic acid. Tables have been prepared by Valenta, Allen, and others, showing the turbidity temperatures for different oils and fats, but as these figures are far from concordant, the analyst will do well to establish his own standards.

Thompson and Ballantyne\* found that the amount of free fatty acids and the specific gravity of the acetic acid exert a marked influence on the results. Fryer and Weston† dry the oil by hot filtration through cotton.

**The Elaïdin Test** was first suggested by Poutet in 1819 and was formerly much used. It is based on the conversion by nitrous oxide of liquid olein into the solid elaïdin, a crystalline compound isomeric with olein, while other common glycerides remain liquid under treatment with this reagent. By the consistency of the final product, when subjected under certain conditions to the action of nitrous oxide, some idea as to the character of the oil may be gained.

*Manipulation.*—To carry out the test according to Poutet (modified), weigh 5 grams of the oil into a beaker, add 7 grams of nitric acid (specific gravity 1.34) and about 0.5 gram of copper wire. Place the beaker in water at 15° and stir thoroughly with a glass rod in such a manner as to make an intimate mixture of the oil and the evolved nitrous oxide gas. After the wire has been dissolved, add another piece of about the same size and again stir vigorously. Set aside for about 2 hours, at the end of which, in the case of pure olive, almond, peanut, or lard oil, it will have been changed into a solid white mass.

Nearly all the seed oils, especially cottonseed and mustard, are turned into a pasty or buttery mass.

Another modification of Poutet's tests‡ consists in mixing 10 grams

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\* Jour. Soc. Chem. Ind., 10, 1891, p. 233.

† Analyst, 43, 1918, 3.

‡ Used in the Paris municipal laboratory.



of the oil, 5 grams of nitric acid (specific gravity 1.38), and 1 gram of mercury in a test-tube, shaking for 3 minutes and allowing to stand 20 minutes, when it is again shaken.

The behavior of various oils after that time on further standing is as follows:

	Solidified after
Olive oil .....	60 minutes
Peanut oil. ....	80 "
Sesame oil.....	185 "
Rape oil .....	186 "

Archbutt \* prepares the reagent previous to use by dissolving 18 grams of mercury in 15.6 c.c of nitric acid (sp. gr. 1.42) and uses 8 grams for 96 grams of the oil, shaking every 10 minutes for 2 hours.

**Determination of Free Fatty Acids.**—Weigh 5–20 grams of the oil or fat into a flask, add 50 cc. of neutralized 95% alcohol, warm in the case of fats until melted, shake thoroughly, and titrate with N/10 alkali, using phenolphthalein as indicator.

The result may be reported in terms of percentage of oleic acid (each cubic centimeter of tenth-normal alkali is equivalent to 0.0282 gram of oleic acid) or as the "acid number," by which is meant the number of cubic centimeters of tenth-normal alkali necessary to saturate the free acid in 1 gram of the fat or oil.

**Constants of the Free Fatty Acids.**—Often much information as to the character of an oil or fat may be obtained by determining such constants of its fatty acids as the melting- and solidifying-point, the iodine number, etc.

To prepare the fatty acids or if soluble acids are present the insoluble fatty acids, for examination, saponify a quantity of the oil or fat with alcoholic potash, evaporate the alcohol, and dissolve the soap in hot water. Decompose the soap by the addition of an excess of hydrochloric or sulphuric acid, heat till the fatty acids rise in a layer to the top of the liquid, cool, remove the fatty acids, boil up again with water, and repeat until the mineral acid is removed. The melting-point, iodine number, etc., are determined as with the oil or fat itself.

**Solidifying-point of the Fatty Acids, or Titer Test.**—*Modified Wolfbauer Method.*†—Saponify 75 grams of fat in a metal dish with 60 cc.

\* Jour. Soc. Chem. Ind., 5, 1886, 304.

† A. O. A. C. Method, U. S. Dept. of Agric., Bur. of Chem. Bul. 107, p. 135.



of 30% sodium hydroxide (36° Baumé) and 75 cc. of 95% by volume alcohol or 120 cc. of water. Boil to dryness, with constant stirring to prevent scorching, over a very low flame, or over an iron or asbestos plate. Dissolve the dry soap in a liter of boiling water, and if alcohol has been used, boil for forty minutes in order to remove it, adding sufficient water to replace that lost in boiling. Add 100 cc. of 30% sulphuric acid (25° Baumé) to free the fatty acids, and boil until they form a clear, transparent layer. Wash with boiling water until free from sulphuric acid, collect in a small beaker, and place on the steam bath until the water has settled and the fatty acids are clear; then decant them into a dry beaker, filter, using a hot-water funnel, and dry twenty minutes at 100° C.

When dried, cool the fatty acids to 15 or 20° C. above the expected titer, and transfer to the titer tube, which is 25 mm. in diameter and 100 mm. in length (1 by 4 inches), and made of glass about 1 mm. in thickness. Place in a 16-ounce saltmouth bottle of clear glass, about 70 mm. in diameter and 150 mm. high (2.8 by 6 inches), fitted with a cork, which is perforated so as to hold the tube rigidly when in position. Suspend the thermometer, graduated to 0.10° C., so that it can be used as a stirrer, and stir the mass slowly until the mercury remains stationary for thirty seconds. Then allow the thermometer to hang quietly, with the bulb in the center of the mass, and observe the rise of the mercury. The highest point to which it rises is recorded as the titer of the fatty acids.

Test the fatty acids for complete saponification as follows:

Place 3 cc. in a test tube and add 15 cc. of alcohol (95% by volume). Bring the mixture to a boil and add an equal volume of ammonium hydroxide (0.96 sp. gr.). A clear solution should result, turbidity indicating unsaponified fat. The titer must be made at about 20° C. for all fats having a titer above 30° C. and at 10° C. below the titer for all other fats.

The thermometer must be graduated in tenth degrees from 10° to 60°, with a zero mark, and have an auxiliary reservoir at the upper end, also one between the zero mark and the 10° mark. The cavity in the capillary tube between the zero mark and the 10° mark must be at least 1 cm. below the 10° mark, the 10° mark to be about 3 or 4 cm. above the bulb, the length of the thermometer being about 15 inches over all. The thermometer is annealed for 75 hours at 450° C., and the bulb is of Jena normal 16<sup>iii</sup> glass, moderately thin, so that the thermometer will be quick acting. The bulb is about 3 cm. long and 6 mm. in diameter. The stem of the thermometer is 6 mm. in diameter and made of the best



thermometer tubing, with scale etched on the stem, the graduation to be clear cut and distinct, but quite fine.\*

**Unsaponifiable Matter.**—As will be seen by reference to the table on page 529, the unsaponifiable matter in pure edible oils and fats is comparatively insignificant in amount, consisting largely of cholesterol or phytosterol. A high content of unsaponifiable matter is indicative of adulteration, pointing to the presence of mineral or coal-tar oils, or to paraffin.

**Determination of Unsaponifiable Matter.**†—Weigh 7 to 10 grams of the fat or oil in a 250-cc. flask, and saponify by boiling with 25 cc. of alcoholic potassium hydroxide and 25 cc. of alcohol under a return-flow condenser. After saponification, add 30 to 40 cc. of water, and bring to the boiling-point. Cool and transfer the contents from the flask to a separatory funnel, washing out the flask first with a small amount of 50% alcohol, and finally with 50 cc. of petroleum ether (B.P. 40°–70°), adding both washings to the separatory funnel. Shake the latter thoroughly, but avoid if possible forming an emulsion. If the latter persists in forming, add a volume of water equal to that of the soap solution, which will sometimes break it up. After separation of the petroleum ether layer, draw off the underlying soap solution into a beaker, and wash the petroleum ether two or three times with 50% alcohol, which is drawn off and added to the soap solution. The petroleum ether is then run into a tared Erlenmeyer flask, and the soap solution extracted twice more with fresh portions of petroleum ether, washing the ether each time with 50% alcohol as before and then transferring the ether to the tared flask. The petroleum ether is then removed by placing the flask on the water-bath, bumping being prevented by means of a spiral of platinum wire weighed with the flask. Finally remove all traces of remaining ether by blowing hot air through the flask, or, in the absence of mineral oils (some of which are volatile), dry in the water-oven to constant weight, cool in a desiccator, and weigh.

**Cholesterol and Phytosterol.**—These are monatomic alcohols, and combine with the fatty acids forming esters. Both respond to the same reactions, and are separated by the same process from the oils and fats in which they occur. Phytosterol was long thought to be the same as cholesterol, and some confusion seems to have arisen from the fact that early writers purport to have found cholesterol in vegetable oils, when

\* Tolman, U. S. Dept. of Agric., Bur. of Chem., Bul. 90, p. 75.

† Hönig and Spitz, Jour. Soc. Chem. Ind., 1891, 1039.



in reality the substance was phytosterol. The latter first distinguished from cholesterol by Hesse, who named it.

*Cholesterol* ( $C_{26}H_{44}O$ ) crystallizes in white, nacreous, monoclinic laminae, having a melting-point of  $145^{\circ}$  and specific gravity 1.067. Its reaction is neutral, it is devoid of taste or smell, insoluble in water, sparingly soluble in cold, but readily soluble in boiling alcohol, and soluble in ether, chloroform, methyl alcohol, benzene, and oil of turpentine. It sublimes unchanged at  $200^{\circ}$ , but at higher temperatures decomposes.

Commercial cholesterol is obtained from wool oil and is known as lanolin, being used largely in medicine as a basis for ointment.

Cholesterol occurs also in the yolk of eggs, in many animal secretions, and in most animal oils and fats.

It separates in laminated, transparent crystals from a mixture of 2 volumes alcohol and 1 volume ether, and in the form of anhydrous needles from chloroform.

*Phytosterol* ( $C_{26}H_{44}O, H_2O$ ) is most abundantly found in the leguminous seeds, and is prepared commercially from these, especially from peas and lentils. It is a constituent of most vegetable oils.

It crystallizes in slender, glittering plates from chloroform, ether, and petroleum ether, and from alcohol in tufts of needles. In solubility it much resembles cholesterol, but its melting-point from  $132^{\circ}$  to  $134^{\circ}$  is lower.

**Determination of Cholesterol and Phytosterol.**—*Method of Forster and Reichmann.*\*—50 grams of the oil or fat are boiled for five minutes in a flask connected with a reflux condenser with two successive portions of 75 cc. of 95% alcohol, and in each case the alcoholic solution is separated by means of a separatory funnel. The combined alcoholic solutions are then boiled in a flask provided with a funnel in the neck, till one-fourth of the alcohol is evaporated, and then poured into an evaporating dish and brought to dryness. The residue is then extracted with ether, and the ether solution is evaporated to dryness, taken up again with ether, filtered, evaporated once more, and dissolved in hot 95% alcohol, from which it is allowed to crystallize. Cholesterol or phytosterol will crystallize out under these conditions, and may be weighed.

**Distinguishing between Cholesterol and Phytosterol.**—It is sometimes of importance to determine which of these substances is present in an oil, or whether indeed both occur. Confirmatory proof as to the presence of vegetable in animal oils may, for instance, be established by

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\* Analyst, 22, 1897, p. 131.



showing whether the unsaponifiable residue in the sample contains cholesterol or phytosterol or both. Hehner\* has made use of this test in determining the presence of cottonseed oil in lard.

The most ready means of distinguishing between cholesterol and phytosterol is furnished by the marked difference between the form of the crystals, the manner of crystallization of the two substances, and the melting points of the acetates.

**Separation and Crystallization of Cholesterol and Phytosterol.—***Bömer's Method.*†—Saponify 100 grams of the fat by heating in a liter Erlenmeyer flask on a boiling water bath with 200 cc. of alcoholic potash solution (200 grams of potassium hydroxide + 1 liter of alcohol). The flask should be provided with a perforated rubber stopper, through which passes a glass tube 700 cm. long, which serves as a reflux condenser. During the first part of the heating shake often and vigorously until the solution is clear, after which continue the heating one-half to one hour longer with occasional shaking.

While still warm, transfer to a separatory funnel of about 1.5 liters capacity, rinsing the flask with 400 cc. of water. When cool, add 500 cc. of ether, shake vigorously for one-half to one minute, opening the cock repeatedly, and allow to stand for two to three minutes until the liquids separate. Remove the ether solution to a flask, and distil off the ether, using a few pieces of pumice stone to prevent bumping. Shake the soap solution two to three more times in the same manner with 200 to 250 cc. of ether, add the ether solution after each shaking to the residue in the distilling flask, and distil off the ether.

Usually a small amount of alcohol remains in the flask after removal of the ether, which may be removed by heating on a boiling water bath in a blast of air. To saponify any remaining fat, add 20 cc. of the alcoholic potash solution, and heat for five to ten minutes as before. Transfer to a small separatory funnel, rinse with 40 cc. of water, cool and shake with 150–200 cc. of ether from one-half to one minute, allow to stand two to three minutes, and draw off the lower layer. Wash the ether solution three times with 10–20 cc. of water, filter, to remove drops of water, into a small beaker, and remove the ether by cautious evaporation on the water bath, thus obtaining the crude cholesterol or phytosterol.

The unsaponifiable residue, which may be weighed after drying, in the case of animal fats shows beautiful radiating crystals, and consists

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\* Ibid., 13, 1888, p. 165.

† Zeits. Unters. Nahr. Genussm., 1, 1898, p. 31.



largely of cholesterol, while in the case of vegetable fats it consists largely of phytosterol. Dissolve the residue in 4-20 cc. of absolute alcohol with the aid of heat, and allow to crystallize slowly in a shallow dish.

The crystallization in the case of cholesterol alone begins from the margin of the liquid and gradually extends inward toward the center, forming a uniformly bright, thin, colorless film over the whole surface. This film is best removed with a knife or spatula and pressed between filter-paper. The film will be seen, even megascopically, to be composed of large, glossy plates with a silk-like luster. After the removal of the first film a second will form similar to the first, but composed as a rule of smaller crystals. These are removed in like manner, dried between filters, and added to the first in a glass. After the second crop, the mother liquid is thrown away. The crystals are then redissolved in absolute alcohol, and again allowed to separate out, being repeatedly recrystallized till the melting-point is constant. In lard and most fats the crystals were found pure by Bömer after the second crystallization.

Phytosterol is crystallized with greater difficulty, especially when derived from seed oils, on account of the presence of pigments and other foreign matter. The first procedure is the same as above described for cholesterol, the crystals being allowed to separate slowly out of a solution in absolute alcohol. Unlike cholesterol, no film is formed on the surface, but needles (sometimes 1 cm. in length) are gradually eliminated, beginning at the margin and extending inward mostly at the bottom. In concentrated solutions, fine needles would be uniformly deposited through the liquid. These are best separated from the mother liquid by filtration, as they are not easily taken out with a knife. They may be washed on the filter with small amounts of absolute alcohol for microscopical examination, or repeatedly recrystallized, as in the case of cholesterol, till the melting-point is constant.

1. *Cholesterol Crystals*.—When crystallized separately under above conditions, cholesterol crystals viewed under the microscope show generally rhomboidal forms of plates, as in Fig. 97, but sometimes with a reentering angle. The plates are often grown together in masses. The most characteristic forms are found from the first crystallization or from the first film removed. Sometimes quadrilateral crystals predominate among the plates, often also the other shapes shown are found most numerous.

2. *Phytosterol Crystals*.—Pure phytosterol crystallizes in needles or narrow plates, arranged commonly in star form or in bunches. The



most common forms are shown in Fig. 98, best conditions as to shape of crystals being obtained from slow crystallization, in which case the needles are finer and more regular.

The crystals are commonly in the form of long, narrow plates, thin and slender, often pointed at both ends. Sometimes the points are lacking, or the ends are beveled. The more frequently they are recrystallized, the larger and more varied are the crystal forms. The

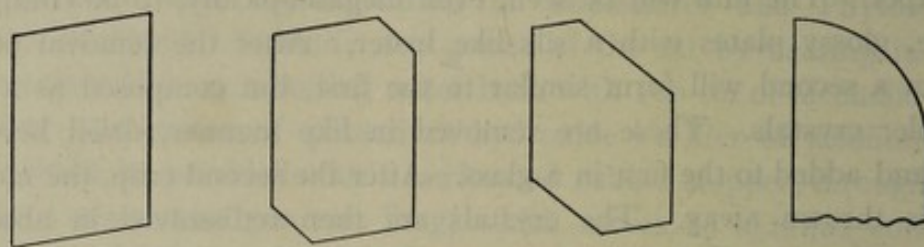


FIG. 97.—Cholesterol Crystals under the Microscope. (After Bömer.)

broad, hexagonal and quadrilateral plates shown are products of recrystallization; the shorter forms are rarely met with. Sometimes various forms are found side by side in the same crystallization.

Phytosterol crystals, from a second or third recrystallization, sometimes grow together in bunches resembling at first glance to the naked eye the cholesterol masses. They never do this in the first crystallization, whereas in the case of cholesterol the growing together in masses is very characteristic of the first crystallization.

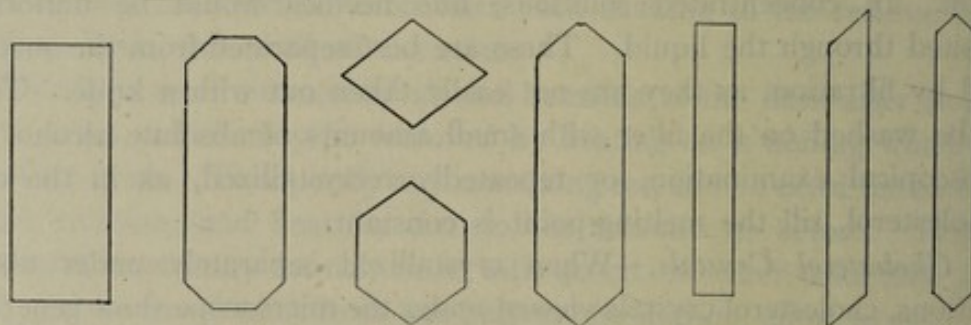


FIG. 98.—Phytosterol Crystals. (After Bömer.)

Thus for purposes of distinguishing between the two the product of the first crystallization is best observed.

3. *Crystals of Mixed Cholesterol and Phytosterol.*—In mixtures of the two they do not crystallize separately, but when in nearly equal proportion, or with phytosterol predominating, the crystals much resemble phytosterol. Even when cholesterol predominates to the extent of 20 parts to 1 of phytosterol, the mode of crystallization leans most toward



that of phytosterol, though the needles are of different shape. Such a mixture, for instance, does not form in a film like cholesterol, but, like phytosterol, comes out in needle-like bunches. The needles, however, are more often like those shown in Fig. 99 when viewed under the micro-

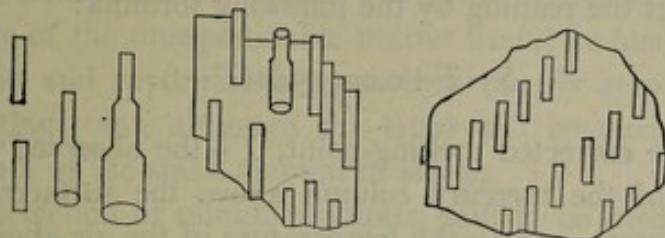


FIG. 99.—Characteristic Forms of Crystallization of Mixed Cholesterol and Phytosterol (After Bömer.)

scope, showing needles for the most part squarely cut off at the ends, and sometimes placed end to end, and of varying diameter, giving the appearance of a spy-glass. When cholesterol predominates over phytosterol 50 to 1, the plates resemble those of cholesterol.

**Bömer's Phytosterol Acetate Test for Vegetable Fats.\***—Dissolve the crude cholesterol or phytosterol, or the mixture of the two, obtained by Bömer's method, as described on page 522, in the smallest possible amount of absolute alcohol, and allow to crystallize. Examine under the microscope the first crystals that separate, comparing with the cuts and descriptions given in the preceding section. Remove the alcohol completely by evaporation on the water bath, add 2 to 3 cc. of acetic anhydride, cover with a watch glass, and boil for one-fourth minute on a wire gauze; then remove the watch glass, and evaporate the excess of acetic anhydride on the water bath. Heat the residue with sufficient absolute alcohol to dissolve the esters, and add enough more to prevent immediate crystallization on cooling. Cover until the room temperature is reached and allow to crystallize.

After one-half to one-third of the liquid has evaporated and the greater part of the esters have crystallized, transfer the crystals to a small filter by the aid of a small spatula, rinsing with two portions of 2 to 3 cc. of 95% alcohol. Return the crystals to the crystallizing dish, dissolve in 5 to 10 cc. of absolute alcohol, and again allow to crystallize. After the greater part of the crystals have separated, collect on a filter as before. Repeat the recrystallization several times (5 to 6 is usually sufficient), determining the melting point of the crystals after each recrystallization beginning with the third.

\* Zeits. Unters. Nahr. Genussm., 4, 1901, p. 1070.



If after the last crystallization the corrected melting-point of the crystals is above  $116^{\circ}$ , the presence of a vegetable fat or oil is indicated, if it is  $117^{\circ}$  or higher the proof may be regarded positive.

The standard thermometer used should be graduated to tenths of a degree. Correct the reading by the following formula:

$$S = T + 0.000154n(T - t)$$

in which  $S$  = the corrected melting-point,  $T$  = the observed melting-point,  $n$  = the length of the mercury column above the surface of the liquid, expressed in degrees, and  $t$  = the temperature of the air about the mercury column as determined by a second thermometer.

Bömer states that by this method the analyst can detect in edible animal fats 1 to 2 per cent of oils rich in phytosterol (cottonseed, peanut, sesame, rape, hemp, poppy, and linseed), and 3 to 5 per cent of oils containing smaller amounts of this constituent (olive, palm, palm kernel, and probably cocoanut). He found the corrected melting-point of cholesterol acetate to be  $114.3^{\circ}$  to  $114.8^{\circ}$  and of phytosterol acetate,  $125.6^{\circ}$  to  $137.0^{\circ}$ , according to the source.

*Klosterman Digitonin Method* \* *Modified by Kühn, Benger, and Werwerinke.*†—To the fatty acids, separated from 50 grams of the sample in the usual manner, contained in a beaker, add 25 cc. of a 1% solution of digitonin (crystalline) in 95% alcohol, and heat at  $70^{\circ}$  C. for 30–45 minutes with occasional stirring until the precipitate of digitonides separates. Add 15–20 cc. of chloroform, filter, using suction, wash successively with chloroform and with ether until all the fat is removed, and dry. Boil the dried precipitate with 3–5 cc. of acetic anhydride for 5 minutes, add while hot 4 volumes of 50% alcohol, and allow the crystals of sterol acetate to separate. Filter, recrystallize from ether, and determine the melting-point as above described.

Numerous experiments, made both in Europe and America, show that feeding milch cows and swine with oil cakes does not introduce phytosterol into either the fat of the milk or the lard, although both fats may respond to the Halphen test, or give abnormally high Polenske numbers as a result of feeding with cottonseed or cocoanut cake respectively, and although the lard (not the butter fat) may respond to the Baudouin test, owing to feeding with sesame cake. (See pages 552 and 579).

\* Zeits. Unters. Nahr. Genussm., 26, 1913, p. 433.

† Ibid., 28, 1914, p. 369; 29, 1915, p. 321.



**Paraffin**, sometimes used as an adulterant of fats, is included in the unsaponifiable matter determined as described on page 520. If present in an amount sufficient to indicate substitution for a more valuable fat the hot solution of the soap will be cloudy, showing often oily drops, and the percentage of unsaponifiable matter will be far in excess of normal. The characteristics of the unsaponifiable matter useful in further identification are the iodine and saponification numbers (nearly zero), the refraction, and the melting-point, although the latter will be modified in a degree by the presence of the small amount of sterols.

For the detection of minute amounts, said to be used to misguide the analyst in interpreting the results of the phytosterol acetate test, Polenske's method \* may be applied.

**Microscopical Examination of Oils and Fats.**—In the examination of lard and butter for adulterants, the use of the microscope is often of great value, and will be described more fully under these special fats. In general, the best fat crystals are obtained by slow crystallization at room temperature from an ether solution, or from a mixture of ether and alcohol. The first crystals formed may often with advantage be filtered out, and washed with the alcohol and ether mixture on the filter, dissolved finally in ether, and the latter allowed to evaporate spontaneously. The crystals are then examined in a medium of ether.

If it is desired to separate the liquid oleins from an oil, so that crystals of the solid fats are left for examination, Gladding † recommends dissolving the fat in a mixture of two volumes of absolute alcohol and one volume of ether in a test-tube, which is stoppered with cotton and set for half an hour in ice water, during which time the more solid stearin and palmitin will have crystallized out. This portion is then separated from the mother liquor by filtration through an alcohol-wet filter-paper, and the crystals finally treated as in the preceding section, being examined in a medium of olive or cottonseed oil.

#### CONSTANTS AND VARIABLES OF COMMON EDIBLE OILS AND FATS.

The tables on pages 528 and 529, based on the results of numerous analysts, are designed merely as a guide. The figures given are not the

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\* Arb. kaisl. Gsndhtsam., 22, 1905, p. 576.

† Jour. Amer. Chem. Soc., 18, 1896, p. 189.



RANGE OF CONSTANTS AND VARIABLES OF COMMON EDIBLE OILS AND FATS.

	Specific Gravity.	Melting-point, ° C.	Solidifying-point, ° C.	Refraction, Butyro Scale.	Refractive Index.	Saponification Number.	Iodine Number.	Maumené Number, ° C.	Acetyl Number.
<b>Oils.</b>	<b>15.5°</b>			<b>25°</b>	<b>25°</b>				
Olive.....	0.915-0.918	.....	.....	60-64	1.4650-1.4685	185-196	77-95	42-52	10.6
Peanut.....	0.917-0.926	.....	-6-+10	65-67.5	1.4691-1.4707	186-196	83-105	45-51	9.1
Rape.....	0.913-0.917	.....	0-+3	68-70	1.4708-1.4723	168-179	94-105	55-64	14.7
Black mustard.....	0.915-0.919	.....	-10-+10	68-71	1.4717-1.4729	173-176	99-122	42-43	
White mustard.....	0.914-0.916	.....	-17-15	64-69	1.4688-1.4717	170-178	92-103	44-50	
Charlock.....	0.922-0.923	.....	-16-15	70-72	1.4720-1.4734	170-181	103-121		
Cottonseed.....	0.922-0.926	.....	.....	66-70	1.4698-1.4723	191-195	104-117	66-77	21-25
Maize.....	0.921-0.927	.....	-5-0	71-72	1.4720-1.4734	180-194	116-130	79-86	
Sesame.....	0.921-0.924	.....	-12-10	68-71	1.4710-1.4729	188-193	103-117	63-72	
Soy.....	0.922-0.928	.....	-6-4	71-73	1.4720-1.4742	189-194	121-143	87-88	
Linseed.....	0.931-0.941	.....	-15-8	81-87	1.4780-1.4824	190-196	170-202	90-145	4.0
Poppyseed.....	0.924-0.927	.....	-25	70-75	1.4723-1.4754	180-197	132-143	86-89	
Sunflower.....	0.924-0.926	.....	-18	72	1.4735	188-194	120-135	81-82	
Lard oil.....	0.915-0.918	.....	-19-16	54-60	1.4620-1.4660	190-198	67-88	45-47	
<b>Fats.</b>	<b>100°</b>			<b>40°</b>	<b>40°</b>				
Cocoa butter.....	0.858	28-35	21-27	46-48	1.4566-1.4580	192-202	32-41	.....	2.8
Cocoonut.....	0.863-0.874	20-28	14-23	33-36	1.4474-1.4495	246-268	8-9.5	21	0.9-12.3
Palm kernel.....	0.859-0.873	23-30	20-27	36-39	1.4495-1.4517	242-255	13-18	.....	1.9-8.4
Butter.....	0.865-0.870	28-36	19-24	40.5-46	1.4527-1.4566	220-241	26-38	.....	1.9-8.6
Lard.....	0.859-0.864	36-46	27-30	48.5-52.5	1.4583-1.4609	193-203	54-70	24-42	2.6
Beef tallow.....	0.860-0.863	42-49	27-35	46-49	1.4566-1.4586	193-200	35-45	32	2.7-8.6
Mutton tallow.....	0.858-0.860	44-49	32-41	40	1.4586	192-195	32-50	.....	
Oleo oil.....	0.860-0.863	30-39	.....	47-50	1.4573-1.4593	198-202	40-50	.....	
Oleo stearin.....	0.858-0.862	41-54	40-50	40-48	1.4524-1.4580	192-197	8-27	20-22	
Cottonseed stearin.....	0.864-0.868	26-40	16-33	.....	.....	195	88-104	48	
Cocoonut stearin.....	0.870	20	21-26	.....	.....	251-257	4-6.6	.....	
Palm kernel stearin.....	0.870	31-32	28	.....	.....	242	8	.....	



Insoluble Fatty Acids and Unsaponifiable Matter.									
	Reichert-Meissl Number.	Polenske Number.	Unsaponifiable Matter, Per Cent.	Free Fatty Acids Calculated as Oleic, Per Cent.	Per Cent (Hehner) Number)	Solidifying-point, °C.	Melting-point, °C.	Iodine Number.	Mean Molecular Weight.
<b>Oils:</b>									
Olive.....	0.6	.....	0.4-1.2	0.3-5*	94-96	17-26	19-31	86-90	280-286
Peanut.....	0.5	.....	0.5-1.0	0.5-1†	95-96	22-33	26-36	95-104	282
Rape.....	0-0.8	.....	0.5-1.0	0.5-7	94-97	16-19	16-21	100-106	307-321
Black mustard.....	.....	.....	.....	.....	95-96	6-8	9-17	109-127	300-318
White mustard.....	.....	.....	.....	.....	96-97	9-10	12-16	94-111	302-310
Charlock.....	.....	.....	.....	.....	94-95	.....	.....	.....	312-339
Cottonseed.....	0.7-0.9	.....	0.7-1.7	0.0-3	95-96	28-37	34-40	105-115	275
Maize.....	0.3-5	.....	1.2-2.0	1.4	93-96	13-19	18-23	113-125	.....
Sesame.....	1-2	.....	0.9-1.3	0.2-0.5	95-96	19-24	23-32	109-112	286
Soy.....	0.4-0.7	.....	1.3-1.5	0.3-3.0	95.5	13-21	20-29	122	.....
Linseed.....	.....	.....	0.3-2.0	.....	95-95.5	13-21	17-21	179-210	283
Poppyseed.....	.....	.....	0.4	.....	94.5-95.5	15-17	20-21	139	.....
Sunflower.....	.....	.....	0.3-0.7	.....	95	17-18	17-24	124-134	.....
Lard oil.....	0.2	.....	.....	.....	97	.....	.....	.....	.....
<b>Fats:</b>									
Cocoa butter.....	0.2-0.8	.....	.....	2.8-6.5	94.5	45-51	48-53	32-39	.....
Cocanut.....	6.0-8.5	15.5-20.5	0.17-0.3	3-5†	88.5-90.5	21-25	24-27	8.4-9.3	205-215
Palm kernel.....	4.0-7.6	6.6-12	0.20	2-5‡	91-91.5	20-26	26-29	12	211-223
Butter.....	24-34	1.5-3.5	.....	.....	86.5-90	33-38	38-45	28-31	258-266
Lard.....	0.2-0.8	0.4-0.6	0.2-0.4	0.1-0.6	96	34-42	35-47	64	278
Beef tallow.....	0.2-0.6	0.5-0.6	.....	.....	95-96	42-45	43-47	25-41	270-285
Mutton tallow.....	0.3	.....	.....	.....	95.5	43-46	46-54	35	283
Oleo oil.....	0.3-0.4	0.5-0.6	.....	0.2-0.6	95-96	.....	.....	.....	.....
Oleo stearin.....	.....	.....	.....	.....	.....	48-51	.....	.....	.....
Cottonseed stearin.....	.....	.....	.....	.....	95.5-96.5	35-41	27-30	94	209
Cocanut stearin.....	3.2-6.3	.....	.....	.....	.....	26-27	28	.....	211
Palm kernel stearin.....	2.2	.....	.....	.....	.....	28	28-30	.....	.....

\* Inedible up to 30. † Inedible up to 18. ‡ Inedible up to 25. § Inedible up to 18.



extreme limits reported in the literature, but rather the limits between which the results of analyses of commercial samples will ordinarily fall. Since the figures for each fat are not based on the same set of analyses made by the same analyst, some inconsistencies may be expected.

### OLIVE OIL.

**Source.**—Olive oil is derived from the fruit of the cultivated thornless olive tree, *Olea Europæa sativa*, of which there are a great many varieties, originally grown in Asia Minor, Greece, Palestine, southern Europe, and northern Africa, and now cultivated also in California, Peru, and Mexico, as well as in Australia. Most of the olive oil of commerce is supplied by Italy, Spain, southern France, Tunis and Algeria. The tree is an evergreen of slow growth and great longevity.

The ripe olive fruit is purple or purplish black in color; it is round or oval in shape, and from 2.5 to 4 cm. in diameter. The oil is contained in the parenchyma cells of the fruit suspended in a watery fluid. A thick skin incloses the fruit, and within is a kernel, which itself contains oil. The fruit flesh of European olives may, according to Lewkowitsch, contain as high as 70% of oil, but on an average contains about 50%. California olives, as shown by analyses made by Colby at the California experiment station, have a lower oil content, over 32% in the flesh or 24% in the whole fruit being exceptional.

**Preparation.**—The finest virgin oil is produced from hand-picked, peeled olives, from which the kernels or pits have been removed. A somewhat inferior grade of oil is produced from the whole olive including the pit, while a distinctly low grade oil is obtained from the stones, or kernels, which are ground into a coarse meal and subjected to pressure, or to the action of such solvents as carbon bisulphide.

In the process of manufacture the fruit, picked when nearly ripe, is reduced to a pulp in a stone or iron mill, and the pulpy mass, contained in baskets or bags, is subjected to pressure in an iron press. The very highest grade of virgin oil is that which runs out from the pulp with little or no pressure. After the first pressing, the pomace is ground, treated with water, and again subjected to pressure. Several pressings in this manner may be carried out, each yielding an oil inferior to that preceding, the lowest grades being used for lubricants and in the manufacture of soap.

The oil as it runs from the press being turbid is clarified by washing and



filtering. It is stated that Italian olive oil is sometimes bleached by shaking with a solution of tannic acid.\*

**Nature and Composition.**—The better grades of olive oil, suitable for table and medicinal purposes, possess a pleasant, bland taste, and a distinctive and agreeable odor, unmistakable in character for that of any other oil. The finest virgin oil is pale green in color, due to the presence of chlorophyll, which is closely associated with the oil globules in the cellular tissue of the fruit. Some varieties of olive oil are nearly colorless, while others are a deep golden yellow.

Olive oil is very soluble in chloroform, benzol, and carbon bisulphide, but sparingly in alcohol. Three parts dissolve in 5 of ether.

For range of constants see pages 528 and 529.

For customs purposes the United States Government considers a gallon equivalent to 7.56 pounds, which is slightly below the truth.

Solid fatty acids constitute from 2 to 18% of the total fatty acids (Tolman and Munson). They consist of palmitic with a small amount of arachidic acid. Stearic acid is absent. The liquid acids are oleic (over 90%) and linolic acids. Gill and Tufts † have shown that olive oil contains phytosterol, not cholesterol, and is not therefore, as once held, an exception among vegetable oils.

**Substitutes.**—As a rule the low-grade olive oils are most subject to adulteration, by reason of the fact that it hardly pays to destroy or even modify the fine quality and delicacy possessed by a first-class oil, which would inevitably be the result if even a small amount of foreign oil were added. Furthermore, if olive oil be slightly rancid or high in free fatty acids, the admixture of a bland oil tends to conceal the fact.

The most common substitute and adulterant of olive oil in this country is naturally cottonseed oil, while in Europe peanut, sesame, poppyseed, rape sunflower and other oils are used. Leach has found in samples of alleged olive oil sold in Massachusetts cocoanut oil ‡ and even fish oil.

**Pure Olive Oil of the U. S. Pharmacopœia.**—The requirements of the Pharmacopœia are as follows:

Specific gravity, 0.910 to 0.915 at 25° C.; iodine value not less than

\* Anal. fals., 4, 1911, p. 355.

† Jour. Am. Chem. Soc., 25, 1903, p. 498.

‡ A sample of alleged olive oil purchased in a Massachusetts drug store and found to be adulterated with cocoanut oil, had the following constants:

Specific gravity.....	0.911	Iodine number.....	74.5
Reichert-Meissl number.....	2.90	Butyro-refractometer at 26°.....	56.5



79 nor more than 90; saponification value 190 to 195; very slightly soluble in alcohol, but readily soluble in ether, chloroform, or carbon disulphide.

When cooled to from 10° to 8° C., the oil becomes somewhat cloudy from the separation of crystalline particles, and at 0° C. it usually forms a whitish, granular mass.

Olive oil should not show the cottonseed oil reaction with the Bechi and Halphen test, pages 536 and 537, nor the sesame oil reaction with the Baudouin test, page 538.

**U. S. Standards.**—*Olive oil* is the oil obtained from the sound, mature fruit of the cultivated olive tree (*Olea europaea* L.) and subjected to the usual refining processes; is free from rancidity; has a refractive index (25° C.) not less than 1.4660 and not exceeding 1.4680; and an iodine number not less than 79 and not exceeding 90. *Virgin olive oil* is olive oil obtained from the first pressing of carefully selected, hand-picked olives.

Neither the pharmacopœial nor the federal standards places a limit for free fatty acids. Archbutt considers that they should not exceed 4% (calculated as oleic) in oil designed for food. Analyses made by Colby indicate that genuine olive oil produced in California has a higher iodine number than the European product, reaching 90 and even higher. Samples from other regions have been reported to have an iodine number over 90.

**Hauchecorne or Nitric Acid Test.**—Pure olive oil, when shaken or stirred with an equal volume of concentrated nitric acid, turns from a pale to a dark-green color in a few minutes. If, under this treatment, a reddish to an orange, or brown coloration is produced, the presence of a foreign vegetable oil (usually a seed oil) is to be suspected. The test should be used with caution and under no circumstances regarded as final.

Bach gives the following table showing the action of strong nitric acid on various oils:

Kind of Oil.	After Agitation with Nitric Acid.	After Heating for Five Minutes.	Consistency after Standing Twelve to Eighteen Hours.
Olive.....	Pale green	Orange-yellow	Solid
Peanut.....	Pale rose	Brownish yellow	Solid
Rape.....	Pale rose	Orange-yellow	Solid
Sesame.....	White	Brownish yellow	Liquid
Sunflower.....	Dirty white	Reddish yellow	Buttery
Cottonseed.....	Yellowish brown	Reddish brown	Buttery
Castor.....	Pale rose	Golden yellow	Buttery



The Zeiss Butyro-refractometer furnishes one of the most useful and easily applied preliminary means of judging the purity of the sample. If the reading is beyond the limits of pure olive oil, it at once indicates adulteration and often points to the particular adulterant. On the other hand, it is not always safe to assume the oil to be pure if the reading is correct, since mixtures of higher and lower refracting foreign oils may be so skillfully prepared as to read well within the limits of the pure oil on the refractometer scale. The refractometer reading of pure cottonseed oil is almost five degrees higher than that of pure olive.

READINGS ON ZEISS REFRACTOMETER OF OLIVE AND COTTONSEED OILS.\*

Temperature (Centigrade).	Scale Reading.		Temperature (Centigrade).	Scale Reading.	
	Olive Oil.	Cottonseed Oil.		Olive Oil.	Cottonseed Oil.
35.0	57.0	61.8	25.5	62.4	67.5
34.5	57.2	62.1	25.0	63.0	67.9
34.0	57.4	62.3	24.5	63.3	68.2
33.5	57.7	62.5	24.0	63.6	68.5
33.0	58.0	62.8	23.5	63.9	68.8
32.5	58.3	63.0	23.0	64.2	69.1
32.0	58.5	63.2	22.5	64.5	69.4
31.5	59.0	63.6	22.0	64.8	69.7
31.0	59.2	64.0	21.5	65.1	70.0
30.5	59.4	64.2	21.0	65.4	70.3
30.0	59.9	64.5	20.5	65.7	70.6
29.5	60.1	64.9	20.0	66.0	70.9
29.0	60.3	65.1	19.5	66.3	71.2
28.5	60.6	65.3	19.0	66.6	71.5
28.0	60.9	65.7	18.5	66.9	71.8
27.5	61.1	66.0	18.0	67.2	72.1
27.0	61.5	66.5	17.5	67.5	72.4
26.5	62.0	67.0	17.0	67.8	72.7
26.0	62.2	67.3	16.5	68.1	73.0

The Elaïdin Test, in the case of pure olive oil, is very distinctive, since it yields by far the hardest elaïdin of all the common oils, and solidifies the most quickly.

Archbutt † shows the effect on this test of the mixture with olive oil of various proportions of rape and cottonseed oil, as follows:

Kind of Oil.	Minutes Required for Solidification at 25° C.	Consistency.
Olive oil .....	230	Hard but penetrable
" + 10% rape oil .....	320	Buttery
" + 20% " .....	From 9 to 11½ hours	"
" + 10% cottonseed oil. ....	" 9 " 11½ "	Very soft.
" + 20% " .....	More than 11½ "	" "

\* Ann. Rep. Mass. State Bd. of Health, 1899, p. 647. † Jour. Soc. Chem. Ind., 1897, p. 447.



**Cottonseed Oil** as an adulterant is best detected by means of the Halphen or Bechi tests. Its presence in notable quantities increases the specific gravity, refractometer reading, and iodine number very materially. Its high Maumené figure is also distinctive.

**Peanut Oil**, when present to a considerable extent, betrays its presence by its peculiar bean-like flavor. Most of the constants of peanut oil lie within the limits of olive oil, with the exception of the higher iodine number and refractometer reading. A considerable admixture of peanut oil raises the refractometer reading perceptibly over that of pure olive. Its presence is best shown positively by tests for arachidic acid (p. 543), noting that traces of arachin have been reported in pure olive oil, insufficient, however, to interfere with the detection of added peanut oil.

**Sesame Oil** differs more particularly from olive in its higher specific gravity and iodine and Maumené numbers, and is readily detected by distinctive color tests.

**Rape and Mustard Oils** are characterized by much lower saponification values and higher iodine numbers than olive. They contain erucic acid.

**Corn Oil** differs materially from olive in its exceedingly high iodine number and refractometer reading. Its specific gravity and saponification numbers are also higher.

**Lard Oil**, when present in considerable quantity, is often rendered apparent by its characteristic odor on warming. Its low refractometer reading and iodine number are also distinctive.

**Poppyseed Oil** differs most widely from olive oil in its refractometric reading, its high dispersion, and its Maumené number, which in the case of poppyseed is  $87^{\circ}$  and of olive about  $42^{\circ}$ .

**Cocoanut Oil** perceptibly raises the solidifying-point. If over 12% is present, the sample will become solid when placed in ice water. The low iodine number is distinctive.

**Fish Oils**, when present, are rendered apparent by reason of their strong taste and smell, and by their very high iodine number. Boiling with sodium hydroxide develops a peculiar reddish coloration. The insoluble bromide test is most decisive.

**Routine Examination of Olive Oil for Adulterants.**—First note the smell and taste of the sample, and then take the refractometer reading. An abnormally high refraction indicates adulteration. Next test for cottonseed oil by the Halphen reaction, for sesame oil by the Baudouin reaction or its equivalent, and also apply the Hauchecorne test. Then test for arachidic acid (peanut oil) and for erucic acid (cruciferous oils). Finally



determine the iodine number and other constants if the qualitative tests indicate an admixture; an iodine number as high as 90 is strong indication of adulteration except in the case of California or North African oils.

The edible oils and adulterants are arranged in order of their relative price about as follows: Olive oil, peanut oil, lard oil, sesame oil, poppy-seed oil, rape oil, corn oil, cottonseed oil.

#### COTTONSEED OIL.

**Source and Preparation.**—This oil, largely used as a table oil and as substitute for olive oil, is derived from seeds of the various species of the cotton plant, *Gossipium*, of which the most common are *G. herbaceum*, native to Asia, but cultivated extensively in southern Europe and in the United States, *G. arboreum*, in Asia and Africa, and *G. barbadense*, in the West Indies. *G. religiosum* and *hirsutum* are varieties of *G. herbaceum*.

Cottonseeds contain 5 to 30% of oil according to the variety and are a by-product in cotton production. They are dark brown or black, irregularly oval, measuring from 5 to 8 mm. greatest diameter. Bombay and American upland cottonseed, after ginning, is still woolly, that is, bears a considerable coat of fiber, while Egyptian and Sea Island seed are naked. In the United States the seed is commonly decorticated before pressing although the ground hulls are now commonly added to the ground cake or meal.

The seeds, whether or not decorticated, are cut into small pieces, crushed between rollers, and afterward submitted to hydraulic pressure in bags to express the oil, which is clarified by filtration or refined by washing the crude oil with sodium hydroxide solution. A specially refined grade is sold in the United States under the name Wesson Oil.

**Nature and Composition.**—Refined cottonseed oil is a pale yellow oil of thick consistency, possessing a bland though pleasant taste and odor.

On cooling the oil to a temperature below 12° C. particles of solid fat will separate. At about 0° to -5° C. the oil solidifies. With concentrated sulphuric acid, a dark, red-brown color instantly appears.

For range of constants see pages 528 and 529.

The solid fatty acids constitute from less than 20 to over 30% of the total acids, the lower figure being for winter oils. They consist largely of palmitic acid with a small amount of arachidic acid. Stearic acid, if present at all, occurs in very small amount. Oleic and linolic acids make up the liquid fatty acid, the latter acid constituting, according to Lewkowitsch, upward of 30% of the total fatty acids.



**U. S. Standards.**—*Cottonseed oil* is the oil obtained from the seeds of cotton plants and subjected to the usual refining processes; is free from rancidity, has a refractive index ( $25^{\circ}$  C.) not less than 1.4700 and not exceeding 1.4725; and an iodine number not less than 104 and not exceeding 110.

*"Winter-yellow" cottonseed oil* is expressed cottonseed oil from which a portion of the stearin has been separated by chilling and pressure, and has an iodine number not less than 110 and not exceeding 116.

**Cottonseed Stearin.**—This product, used in lard substitutes, is obtained as a by-product in the manufacture of winter-yellow cottonseed oil. It is a light yellow fat, resembling butter in consistency.

**Hydrogenated Cottonseed Oil** is now often used in place of animal or cottonseed stearin as a stiffener for lard. Under various trade names, unmixed, it is a popular substitute for lard.

**Bechi's Silver Nitrate Test.**—*Hehner's Modification.*—Two grams of silver nitrate are dissolved in 200 cc. of 95% alcohol free from aldehyde, 40 cc. of ether are added, and the reagent made very slightly acid with nitric acid.

In applying the test, a small quantity of the melted fat or oil is mixed in a test-tube with half its volume of the above reagent, and the tube is immersed in boiling water for fifteen minutes. With proper precautions the presence of cottonseed oil is indicated by a more or less strong reduction of the silver, while an oil or fat free from cottonseed oil causes no appreciable reduction.

Certain oils free from cottonseed that have become rancid or decomposed, as well as fats that have been subjected to a high temperature, sometimes show a slight reduction with Bechi's test. In cases of doubt it is well to apply the test on the fatty acids as follows:

*Milliau's Modification of Bechi's Test.\**—Heat 20 grams of the sample with 30 cc. of alcoholic potash solution (20% potassium hydroxide in 70% alcohol), shaking at intervals till saponification is complete. Continue the heating for some minutes afterward until the alcohol is driven off, and dissolve the soap in 250 cc. of hot water. Add a slight excess of 10% sulphuric acid, and wash the separated fatty acids three times by decantation with water. Then proceed with a portion of the fatty acids as in Bechi's test.

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\* *Moniteur Scientifique*, 1888, 366.



**The Halphen Test.\*** is much more delicate and dependable than either of the preceding, as little as 2% of cottonseed oil being rendered apparent in olive oil. A mixture is made of equal volumes of amyl alcohol and carbon bisulphide in which 1% of sulphur has been dissolved. From 3 to 5 cc. of melted fat are mixed with an equal volume of the above reagent in a test-tube, loosely stoppered with cotton, and heated in a bath of boiling saturated brine for fifteen minutes. If cottonseed oil is present, a deep-red or orange color is produced. In its absence little or no color is developed.

Previous heating of the oil diminishes the delicacy of the Halphen test, and Holde and Pelgry † state that if cottonseed oil has been heated at 250° C. for ten minutes, it will fail to respond to the test. Fulmer ‡ finds that it is necessary to heat to 265 to 270° to render it wholly inactive to the test. Hydrogenation also destroys the constituent that produces the color. The influence of feeding hogs cottonseed meal on the reaction with the lard is discussed on p. 579.

Gastaldi § finds that it is the pyridin bases in amyl alcohol that render it useful. The test can be made by heating 5 cc. oil, 4 cc. carbon bisulphide containing 1% of sulphur, and 1 drop of pyridin for from 15 minutes to one hour in a water-bath.

Kapok oil, from two species closely related to cotton, also responds to the Halphen test, but is a rarity on the American market.

### SESAME OIL.

Sesame, benne, teel or gingilli oil is pressed from the seeds of common sesame (*Sesamum Indicum*) and black sesame (*S. radiatum*). These plants are native to southern Asia, but are now cultivated in nearly all tropical countries. The larger portion of commercial sesame oil is manufactured in England, France, Germany, and Austria.

The seeds are flattened pear-shaped, 2 to 3 mm. long; those of *S. Indicum* are yellow to brown, while those of *S. radiatum* are dark brown to nearly black. They contain 35 to 60% of oil. The seeds are commonly subjected to cold pressure once, and afterwards twice pressed when warm, thus yielding three grades of oil.

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\* Jour. pharm. chim., [6] 6, 1899, p. 390.

† Jour. Soc. Chem. Ind., 18, 1899, p. 711.

‡ Jour. Amer. Chem. Soc., 24, 1902, p. 1149.

§ Chem. Ztg., 35, 1911, p. 688.



Sesame oil consists of the glycerides of oleic, stearic, palmitic, and linolic acids together with phytosterol, sesamin, and sesamol, the latter causing the red color in the Baudouin test. It is golden yellow in color, free from odor, and possesses a delicate and characteristic flavor, on account of which the highest grades are by some considered equal to olive oil as a condiment. It is accordingly sold to some extent as an edible oil. It was formerly used as an adulterant of olive oil, but has of late years been largely displaced by cheaper oils for purposes of adulteration. When cooled to  $-3^{\circ}$  C., sesame oil congeals to a yellowish-white mass.

For range of constants see pages 528 and 529.

**U. S. Standards.**—Refractive index ( $25^{\circ}$ ) 1.4704 to 1.4717; iodine number 103 to 112.

**Adulterants** to be looked for in sesame oil are cottonseed, poppyseed, corn, and rape oils.

**Tocher's Test.\***—One gram of pyrogalllic acid is dissolved in 15 cc. of concentrated hydrochloric acid mixed with 15 cc. of the sample in a separatory funnel. After standing for a minute, the aqueous solution is withdrawn and boiled. If sesame oil is present, the solution shows a red coloration by transmitted, and a blue by reflected, light.

**Baudouin's Test.†**—Dissolve 0.1 gram of cane sugar in 10 cc. of hydrochloric acid (specific gravity 1.20) in a test-tube, and shake thoroughly with 20 grams of the oil to be tested for one minute. Then allow the mixture to stand. The aqueous solution quickly separates from the oil, and in the presence of 1% or more of sesame oil will be colored deep red.

Certain pure Tunisian and Algerian olive oils have been found to cause a slight coloration with this test, but of a different shade from sesame. Zega and Todorovic‡ state that 5 cc. of the colored solution made up to 25 cc. and shaken loses its color in five to eight minutes, while the colored solution in the case of olive oil containing 3% of sesame oil required thirty minutes. Moreover, if the test is applied to the fatty acids, no coloration in the case of olive oil is produced, while with sesame the color is the same as with the oil.

**Villavecchia and Fabris Test.§**—This test was suggested on account of the fact that the color reaction in the Baudouin test was attributed to the agency of the levulose produced by the inversion of the sugar by

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\* Chem. Ztg. Rep., 5, 1891, p. 15.

† Zeits. angew. Chem., 1892, p. 500.

‡ Chem. Ztg., 33, 1909, p. 103.

§ Jour. Soc. Chem. Ind., 1894, pp. 13-69.



hydrochloric acid. As furfural is the chief product of the reaction between levulose and hydrochloric acid, it was substituted as follows: Dissolve 2 grams of furfural in 100 cc. of 95% alcohol, and shake 0.1 cc. of this solution in a test-tube with 10 cc. of the oil to be tested and 10 cc. of hydrochloric acid (specific gravity 1.20) for half a minute. The aqueous layer, on settling out, will be colored deep red, if sesame is present.

Or 0.1 cc. of the alcoholic furfural solution is mixed with 10 cc. of oil and 1 cc. of hydrochloric acid in a separatory funnel, shaken well, and the separation aided by the addition of chloroform, which causes the aqueous layer, showing color with sesame oil, to float.

Sesame oil that has become rancid or has been exposed to air for some time gives a blue or green color.

#### RAPE OIL.

Rape or colza oil is expressed from the seeds of the *Brassica* or rape plant, of which there are three principal varieties. *Brassica napus*, *B. campestris*, and *B. rapa*, one or another of which are cultivated in nearly every country of Europe, excepting Greece. Large amounts are also grown in India and China. The seeds are small, round grains, from 2 to 2.5 mm. in diameter, yielding from 35 to 45 per cent of oil.

In the process of preparation the seeds are first crushed, and the oil removed by pressing or extraction. The crude oil is of a brownish yellow color, and when fresh is almost free from taste and smell, so that it serves, when cold pressed, as an edible oil, or an adulterant of such oils. It develops a disagreeable and peculiar taste and odor on long standing, due to the presence of certain albuminous and mucilaginous substances which it contains. These may be removed by refining, usually by treatment with sulphuric acid, but the refined oil has an unpleasant taste and odor.

The characteristic fatty acids of cruciferous oils (rape, mustard, and charlock) are erucic and rapic. Rape oil also contains arachidic acid 0.36-1.61%, Archbutt). Oleic acid occurs in large amount, saturated acids only to the extent of about 1% (Tolman and Munson). The presence of sulphur, formerly thought to be characteristic of cruciferous oil, is accidental. Cruciferous oils properly refined contain none, while other oils extracted by carbon bisulphide may contain an appreciable amount.

**Detection.**—Rape and other cruciferous oils are said to be used as adulterants of edible oils although definite evidence appears to be meagre.



They are detected by the presence of erucic acid \* as well as by their low saponification numbers. The Palas test has been found unreliable. Constants and variables are given on pages 528 and 529.

#### MUSTARD OIL.

The fixed oil of mustard is a by-product expressed from the seeds of black, white, and brown mustard (*Brassica nigra*, *B. alba*, and *B. Besseri-ana*) in the process of preparation of mustard flour as a spice. The seeds contain 25 to 35% of oil.

Black mustard oil is brownish yellow in color, having a mild flavor, and an odor but slightly suggestive of mustard. White mustard oil is golden yellow and has a somewhat sharp taste.

Mustard oil resembles rape oil in composition, containing glycerides of erucic and probably rapic acid and having a low saponification number.

#### CHARLOCK OIL.

Wild mustard, produced in enormous quantities in American grain fields, consists of charlock (*Brassica arvensis*) and brown mustard (*B. Besseri-ana*) in various proportions (page 476). The following results were obtained by Bailey and Burnett † on the oil expressed from the seeds of charlock separated from American screenings and shown by botanical analysis to be 98 and 99% pure:

Specific gravity 15°/15°.....	0.9221
Refractive index, 25°.....	1.4734
Saponification No.....	182.9
Iodine No. (Hanus).....	121.1
Insoluble acids and unsaponifiable.....	95.3
Soluble acids.....	0.0
Mean mol. wt. of ins. acids.....	339.1
Liquid acids, per cent.....	89.3
Liquid acids, iodine No.....	126.0
Solid acids, per cent.....	3.1
Solid acids, iodine No.....	—

\* Tortelli and Fortini, *Gaz. chim. ital.*, 41, I, 1911, p. 173. Biazzo and Vigdorcik *Ann. chim. appl.*, 6, 1916, p. 185.

† *Jour. Ind. Chem.*, 8, 1916, p. 429.



The figures for iodine number given in the foregoing table are higher than reported by Grimme (102.6), due probably to the freedom of the seed from brown mustard and other impurities. Data on the constants of brown mustard are lacking owing to the confusion caused by assigning to the Russian species or true brown mustard (*B. Besseriana*) the specific name *B. juncea* which has been shown to belong only to the Indian Species known as "rai."

#### CORN OR MAIZE OIL.

Corn oil is derived from the kernels of Indian corn or maize (*Zea Mays*) which contain from 3 to 7.5% of fat soluble in ether or calculated to the germ, as separated from the kernels, from 18 to 30%. Whether the corn is designed primarily for the manufacture of mill products, starch, glucose, or alcohol the best approved practice at the present time is to separate the germs mechanically and from them obtain the oil by pressure. E. H. S. Bailey\* states that in the manufacture of starch and glucose the germ is removed from the grain after soaking in sulphurous acid, which gives a high yield, but induces rancidity in the oil even before extraction, while, by the dry process as followed in the corn mills, an oil is obtained which requires little refining. Formerly the oil was separated from the vats in the distilling industry.

**Nature and Composition.**—When obtained from damaged grain or as a by-product from the starch, glucose, or distilling industries the unrefined oil may be high, both in total and volatile fatty acids, but when prepared from sound grain by the dry process it is low in acidity and has an agreeable flavor suggestive of the grain. Such oil is well suited for salads and cooking, also after hydrogenation as an ingredient of butter and lard substitutes. The color is a more or less decided yellow. The oil is semi-drying.

The solid fatty acids, amounting to about 7%, (4.55% Hopkins; 7.44% Tolman and Munson) consist largely or entirely of palmitic acid. Stearic acid appears to be absent. Two liquid acids are present, linolic (29%) and oleic (64%). The amount of unsaponifiable matter is high (1.2–2.9%).

It is claimed by Hopkins,† by Hoppe-Seyler, and others, that corn oil, unlike most vegetable oils, contains cholesterol. Olive oil was long supposed to be unique as a vegetable oil in containing this substance. Hopkins, on the assumption that cholesterol occurs in corn oil, suggested that a test

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\* U. S. Dept. Agric. Yearbook, 1916.

† Jour. Amer. Chem. Soc., 20, 1898, p. 948.



for corn oil as an adulterant of certain vegetable oils lay in the identification of cholesterol.

Gill and Tufts\* claim that, while the alcohol of corn oil is not phytosterol, neither is it cholesterol, but a third substance, known as sitosterol,† occurring in wheat and rye.

There are no color reactions identifying corn oil as such. Its presence in other oils is indicated only by its influence on the various constants, the iodine number and refractometric reading especially being much higher than those of other edible oils. For range of constants and variables see tables pages 528 and 529.

#### PEANUT OIL.

Peanut or arachis oil is obtained from the seeds of the *Arachis hypogæa* (peanut, ground nut, or earth nut) cultivated in most tropical countries, notably in South America, China, India, and Japan. The plant is a creeping herb, developing its blossoms in the axes of the leaves. The flower buds grow down into the earth, where the fruit is ripened, forming the well-known peanuts of commerce. The shelled nut contains nearly 50% of fat.

The oil is extracted by pressure, the first cold-drawn oil being practically colorless, and possessing a pleasant taste suggestive of kidney beans. It is especially adapted for use as a salad or table oil. A second pressure of the moistened residue from the first yields an inferior oil, yellowish in color, also somewhat used for edible purposes, and sometimes commercially called "butterine oil."

**Composition.**—The insoluble fatty acids consist chiefly of arachidic and lignoceric acids which together form about 5%. Stearic and palmitic acids, if present at all, are in small amount. The chief liquid fatty acid is oleic. Linolic (6%) is also present. The presence of hypogæic acid is in dispute.

**U. S. Standards.**—Refractive index (25°) 1.4690 to 1.4707; iodine number 87 to 100.

**Adulterants** of peanut oil are cottonseed, poppyseed, rape, and sesame oils. It was itself formerly used to a considerable extent as an adulterant of French and Italian olive oils but now ranks as a substitute.

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\* Ibid., 25, 1903, p. 251.

† Burian, Monatsh. Chem., 18, 1897, p. 551.



**Detection.**—Peanut oil, when pure or nearly pure, may as a rule be readily distinguished from other common oils by its constants (pages 528 and 529), the presence of arachidic acid, and negative reactions with color tests. When present in olive oil chief dependence for its detection must be placed on the Renard test for arachidic acid, especially in its modified forms or on the Bellier test.

**The Renard Test** \* has long been in use for detecting and estimating peanut oil in mixtures. In its original form this test did not give entirely satisfactory results, and earlier led to some erroneous conclusions. In recent years, however, it has been so modified and improved as to be capable of quite positive results when carefully carried out. While arachin is said to occur in minute traces in olive oil, its presence is not sufficiently marked to interfere with the use of the Renard method in detecting any decided admixture of peanut oil.

**Tolman Modification.**†—Saponify 20 grams of the sample in a 250-cc. Erlenmeyer flask with 200 cc. of a solution of 40 grams of potassium hydroxide in 1 liter of 95% redistilled alcohol. Neutralize with dilute acetic acid, using phenolphthalein as an indicator, and wash into a 500-cc. flask containing a boiling mixture of 100 cc. water and 120 cc. 20% solution of lead acetate.

Boil for a minute and cool the contents of the flask by immersing in cold, or, preferably, ice water, whirling the flask occasionally so that the soap when cold adheres to the sides of the flask. The water and excess of lead acetate can then be poured out, leaving the soap in the flask. Wash by shaking and decantation, first with cold water and then with 90% alcohol. Add 200 cc. of ether, cork the flask, and allow to stand with occasional shaking till the soap is disintegrated after which, boil on a water-bath under a reflux condenser for five minutes. Cool the soap solution down to a temperature between 15° and 17°, and allow it to stand for about twelve hours.

Filter and thoroughly wash the precipitate with ether, after which the soap in the filter is washed back into the original flask with a stream of hot water acidulated with hydrochloric acid.

Add an excess of dilute hydrochloric acid, partially fill the flask with hot water, and heat until fatty acids form a clear oily layer. Fill the flask with hot water, allow the fatty acids to harden and separate from the

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\* Compt. rend., 73, 1871, p. 1330.

† U. S. Dept. of Agric. Bur. of Chem., Bul. 65, 1902, p. 33.



precipitated lead chloride, wash, drain, repeat washing with hot water, and dissolve the fatty acids in 100 cc. of boiling 90% by volume alcohol. Cool to 15° C., shaking thoroughly to aid crystallization.

From 5 to 10% of peanut oil can be detected by this method, as it effects a complete separation of the soluble acids from the insoluble, which interfere with the crystallization of the arachidic acid. Filter, wash the precipitate twice with 10 cc. of 90% alcohol, and then with 70% alcohol. Finally dissolve off the precipitate with boiling absolute alcohol, evaporate to dryness in a tared dish, dry and weigh. To the weight add 0.0025 gram for each 10 cc. of 90% alcohol used in the crystallization and washing, if done at 15° C., and 0.0045 gram for each 10 cc. if done at 20°. The approximate amount of peanut oil is found by multiplying the weight of arachidic acid by 20.

Arachidic acid crystals thus obtained should be examined microscopically. The melting-point should lie between 71° and 72° C.

W. B. Smith\* found that recrystallization is essential for the detection of peanut oil in solid fats.

*The Kerr Modification.*†—Heat to boiling 20 grams of the sample with 200 cc. of 95% ethyl alcohol, add while boiling 10 cc. of potassium hydroxide solution (100 grams in 100 cc. of water), and when saponification is complete neutralize with a solution of glacial acetic acid in 95% alcohol (1 : 3). Add 50 cc. of magnesium acetate solution (10 grams of the salt in 100 cc. of water and 100 cc. of 95% alcohol) and heat again to boiling. Cool to room temperature with occasional shaking and keep in a refrigerator at 10° to 15° C. over night, filter, wash the precipitate twice with 50% alcohol, and three times with water, then return to the saponification flask. Add 100 cc. of hot distilled water and sufficient sulphuric acid (1 : 3) to decompose the magnesium salts. Heat until the separated acids form a clear layer, cool until they solidify, and pour off the acid solution. Add to the fatty acids 100 cc. of hot water and when they have melted, cool and decant off the liquid as before. Drain, dissolve in 100 cc. of 90% (by volume) alcohol, and crystallize as in the Tolman modification.

**Methods of J. Bellier.**‡—*Qualitative Test.*—Saponify 1 gram of the oil with 5 cc. of an alcoholic potash solution containing 85 grams potassium hydroxide per liter of strong alcohol, conducting the saponification in a small Erlenmeyer flask on the water-bath. After saponification,

\* Jour. Amer. Chem. Soc., 29, 1907, p. 1756.

† Jour. Ind. Eng. Chem., 8, 1916, p. 904.

‡ Ann. chim. anal., 1899, 4, p. 49.



boil for two minutes, neutralize with dilute acetic acid, using phenolphthalein as an indicator, and cool by setting the flask in water at a temperature of from  $17^{\circ}$  to  $19^{\circ}$ . After a short time, a precipitate nearly always comes down. Then add to the solution 50 cc. of 70% alcohol, containing 1% by volume of strong hydrochloric acid (specific gravity 1.20). Cork the flask, shake vigorously, and again cool by setting the flask in the above cooling-bath. In the absence of a precipitate, the oil may be pronounced free from peanut. If 10% or more of peanut oil is present, a more or less characteristic precipitate forms, and often with less than 10% a cloudiness in the solution is perceptible after standing between  $17^{\circ}$  and  $19^{\circ}$  for half an hour. Pure olive oil remains perfectly clear as a rule.

A few varieties of olive oil from Tunis especially high in solid fat acids, as well as cottonseed oil and sesame oil, give similar turbidity on the addition of the 70% alcohol. To distinguish between these oils and peanut oil, heat the mixture on the water-bath till complete solution takes place, and again cool to  $17^{\circ}$  to  $19^{\circ}$ . In the case of peanut oil the cloudiness or precipitate again occurs to the same extent as before, while in the other cases the solution should remain clear or nearly so.

*Quantitative Determination.*—Saponify 5 grams of the oil with 25 cc. of the above alcoholic potash solution in a 250-cc. Erlenmeyer flask, neutralize exactly with acetic acid, and cool quickly in water. After standing an hour, pour upon a 9-cc. filter and wash the precipitate with 70% alcohol containing 18% by volume of hydrochloric acid, the temperature of the solution being not less than  $16^{\circ}$  nor more than  $20^{\circ}$ . Continue the washing till the wash water no longer shows turbidity when diluted with water.

Dissolve the precipitate in 25 to 30 cc. of hot 95% alcohol, dilute with water until the alcohol is 70%, let stand in water at  $20^{\circ}$ , filter, wash with 70% alcohol, dry at  $100^{\circ}$ , and weigh.

Bellier states that he has recognized with certainty as small an admixture as 2% of peanut oil by this method.

#### SOY OIL.

This oil, prepared from the seed of *Soja hispida*, is of comparatively recent introduction in the Occident, although long extensively used in China and Japan both for food and technical purposes.

The seed contains about 20% of a semi-drying oil of which somewhat more than half is expressed by the methods employed. Extraction yields



a larger amount but is practiced chiefly with damaged beans the cake of which is not suited for cattle food. The edible oil is commonly filtered through fuller's earth.

**Composition.**—Matthes and Dahl\* have reported in soy oil 15% of palmitic acid and 80% of liquid fatty acids, the latter consisting of 56% of oleic acid, 19% of linolic acid, and 5% of linolenic acid, calculated in percentage of the oil. Because of the presence of linolenic acid, the oil responds to the hexabromide test.

Keimatzu† found 12% of saturated fatty acids and 80% of unsaturated fatty acids, about half of the latter consisting of an isomer of linolic acid, the remaining half containing linolic and oleic acids. The same author isolated phytosterol, but could not find a trace of stigmasterol, which Matthes and Dahle claim is present.

**Detection.**—Quite widely different constants for soy oil have been reported indicating that the product is far from uniform, due to the process employed and other causes. The iodine number is higher than that of most edible oils, approaching that of linseed.

Washburn‡ has determined the values for 202 samples prepared by him by hot pressure (or in a few cases by carbon tetrachloride extraction) from soy beans grown in various states representing 45 varieties, the range being as follows:

	Specific Gravity at 15.5° C.	Refractive Index at 25° C.	Saponification No.	Iodine No. (Hanus).
Maximum.....	0.9310	1.4750	197.4	141.9
Minimum.....	0.9207	1.4710	190.1	115.5

The range of values by all observers appears on pages 528 and 529.

**Settimi Test.**§—The following color test has been proposed: Shake vigorously so as to form a thorough emulsion 5 cc. of the oil with 2 cc. of chloroform and 3 cc. of 2% aqueous uranium nitrate solution. With soy oil the color is intense lemon yellow, with peanut, sesame, and corn oil, white, and with olive oil, slightly green. Certain olive oils give a slight yellowish color but do not give the seed oil reactions which soy does in a marked degree.

\* Arch. Pharm., 240, 1911, p. 424.

† Chem. Ztg., 35, 1911, p. 839.

‡ N. Dak. Agr. Exp. Sta. Bul., 118, 1916.

§ Gior. farm. chim., 61, p. 495.



## LINSEED OIL.

Brief mention should be made of linseed oil. Although not usually classed with edible fats, the oil is used in pharmacy and the cake, either old process (pressed) or new process (extracted) is a valuable cattle food.

Linseed oil is a typical drying oil with a high iodine number, being composed chiefly of the unsaturated acids, linolenic, linolic and oleic, named in the order of the amount present (Fahrion). Constants are given on pages 528 and 529.

## POPPYSEED OIL.

This oil is obtained from the seeds of the opium poppy (*Papaver somniferum*), native in the countries east of the Mediterranean, and cultivated extensively for opium and for oil in all parts of Europe, Asiatic Turkey, Persia, Egypt, India, and China. Most of the oil of commerce comes from France and Germany.

There are two chief varieties of poppy, the black (*P. nigrum*) and the white (*P. album*), the finest oil being produced from the white. The seeds are somewhat flattened in form and kidney-shaped, containing from 40% to 50% of oil.

The oil is obtained by crushing the seeds and applying pressure. The best grade of cold-drawn oil is pale yellow in color, possessing a pleasant taste when fresh, and being practically free from odor. Lower grades shade into deeper yellow and even reddish color, possessing a strong taste and odor.

Poppyseed oil is much used in Europe as a table oil, and does not readily turn rancid. It has been used to some extent as an adulterant of olive oil, and is itself not infrequently adulterated with sesame oil.

**Composition.**—The solid fatty acids (6.67 Tolman and Munson) consist presumably of palmitic and stearic acids. The liquid acids according to Nazura and Grüssner are composed of 65% linolic acid, 30% oleic acid, and 5% linolenic acid.

**Detection.**—The refraction and iodine number are high as they are also in soy bean and sunflower oils (pp. 528 and 529).

## SUNFLOWER OIL.

Sunflower oil is derived from the seed kernels of the plant of the same name (*Helianthus annuus*), originally grown in Mexico, but now



cultivated most extensively on a commercial scale in southern Russia, Hungary, and the Orient.

The whole seed, or rather dry fruit, has, according to S. M. Babcock \* the following composition:

	Air-dry.	Dried.
Water.....	12.68	
Ash.....	3.00	3.43
Albuminoids (N×6.25)...	15.88	18.19
Crude fiber.....	29.21	33.45
Nitrogen-free extract....	18.71	21.43
Fat (ether extract).....	20.52	23.50
	100.00	100.00

The decorticated seed contains as high as 50% or more of oil.

In preparing the oil the decorticated seed is crushed and subjected to either hot or cold pressure.

Sunflower oil is pale yellow in color, has a mild, pleasant taste, and is nearly free from odor. The cold-drawn oil is the variety most used for edible and culinary purposes in Russia, and in Europe as an adulterant of olive oil.

**Composition.**—The liquid fatty acids of sunflower oil consist for the most part of linolic, but little oleic acid being found. The constants as given on pages 528 and 529 show that the oil resembles soy bean and poppyseed oils.

#### ROSIN OIL.

Rosin oil is prepared by the distillation of common rosin, and is an alleged adulterant of olive oil.

**Lieberman-Storch Test.**—Shake 1 to 2 cc. of the sample with acetic anhydride while warming. Cool, remove the anhydride by a pipette, and add a drop of sulphuric acid (specific gravity 1.53). Rosin oil gives a fugitive-violet color.†

Cholesterol also responds to this color reaction.

**Renard's Test for Rosin Oil.**—Prepare a solution of stannic bromide by allowing dry bromine to fall drop by drop upon tin in a dry, cool flask, and dissolving the product in carbon bisulphide.

\* The Sunflower Plant, its Cultivation, Composition, and Uses. U. S. Dept. of Agric., Div. of Chem., Bul. 60, p. 18.

† U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 32.



Add a drop of this reagent to 1 cc. of the oil. In presence of rosin oil a violet color will be produced.

**Polarization Test for Rosin Oil.\***—The oil is dissolved in definite proportion in petroleum ether, and polarized in a 200-mm. tube. Rosin oil polarizes from +30 to +40 on the cane sugar scale, while other oils have a reading between +1 and -1.

### COCOANUT OIL.

Cocoanut oil is the fat expressed from the kernels of the cocoanut or fruit of the cocoa palm (*Cocos nucifera*), indigenous to the South Sea Islands and to the East-Indian archipelago, but grown in many tropical countries.

The oil is prepared from either the fresh or dried kernels (copra). The method employed in India, Ceylon, and Cochin depends on pounding the fresh kernels, boiling with water, and skimming off the fat. The fat is obtained from copra by pressing either in the country of origin or in Europe or America.

While formerly cocoanut oil was used in the Occident chiefly for soap making or in pharmacy, since the beginning of the Great War it has come into very extensive use in the manufacture of vegetable or nut butter.

**Nature and Composition.**—Cold drawn Malabar oil is of a greenish color and is used chiefly by the natives as food. Edible cocoanut oil of commerce is white with a mild taste and characteristic odor. Formerly it was regarded as specially prone to become acid and rancid, but it is now known that if properly made from sound material the fat has good keeping qualities.

Cocoanut and palm kernel oils are characterized by their high content of lauric and myristic acids and consequent high Polenske numbers. They also contain considerable amounts of caproic, caprylic, and capric acids (but no butyric acid), hence the higher Reichert-Meissl numbers than other oils and fats excepting butter fat.

**Detection.**—The constants most valuable in the detection of cocoanut oil in addition to the Reichert-Meissl and Polenske numbers, are the saponification number, and the iodine number as shown in the table on pp. 528 and 529. The iodine number (8-9.5) is strikingly low, although oil from the rind, according to Richardson,† runs as high as 40.

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 32.

† Jour. Ind. Eng. Chem., 3, 1911, p. 574.



According to Andés,\* crystals of cocoanut oil appear under the microscope as a thick network of long needles. Hinks† distinguishes cocoanut oil from butter fat by the needle-shaped crystals which separate out from alcohol.

#### PALM KERNEL OIL.

This oil, also known as palm nut oil, is pressed from the dried endosperm of the seed of *Elæis Guineensis* and is radically different from palm oil which is prepared from the fruit pulp. The tree is indigenous to West Africa where natives gather the fruit in large quantities and separate the endosperm from the shell by hand. The oil was formerly expressed at Marseilles but more recently the industry has been confined chiefly to Hamburg.

Palm kernel oil resembles closely cocoanut oil in physical and chemical properties and serves the same purposes. The better grades are used in butter and lard substitutes while the inferior grades are made into soap.

Palm oil is not classed with the edible fats although being of a deep orange color it has been added to butter substitutes for coloring (p. 565).

#### COCOA (CACAO) BUTTER.

This preparation is a by-product in the manufacture of cocoa, being removed by pressure from the crushed and ground cocoa nibs. The fat in cocoa beans usually is over 50%. The expressed fat is yellowish white, extremely hard, has a pleasant taste and an odor suggestive of chocolate. It keeps a long time without turning rancid. In composition it consists of the glycerides of stearic, palmitic, and oleic acids, with small amounts of the glycerides of arachidic and linolic acids.

It is in demand for pharmaceutical purposes and for adding to the chocolate used in coating candies.

It is subject to adulteration with paraffin, tallow, stearins, hydrogenated oils, and various tropical fats.

#### TALLOW.

The rendered fats of various animals, especially the cow and sheep, constitute what is generally known as tallow. The untreated fatty tissues are more properly known as suet, the tallow being the clear fat separated entirely by heat from the cellular material.

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\* Vegetable Fats and Oils, London, 1897.

† Analyst, 32, 1907, p. 160.



Tallow consists almost entirely of olein, palmitin, and stearin. Mutton tallow is usually, but not always, harder than beef tallow.

Excepting in the manufacture of material for oleomargarine, wherein the heart and caul fats of beef are almost exclusively used, the fats from different parts of the animal are not, as a rule, separated.

Fresh tallow has very little free fatty acid, but when it becomes rancid, the fat contains sometimes as high as 12% of free acid, reckoned as oleic.

Tallow is of chief interest to the food analyst in connection with its use as an adulterant of lard.

### BUTTER.

**Nature and Composition.**—Butter is the product obtained by the churning of cream or milk, whereby the fat particles are caused to adhere together into a compact mass, inclosing a certain portion of the casein, the excess of milk serum being subsequently largely removed by washing and mechanical working.

**Butter Fat** is of extremely complex composition, containing a larger variety of glycerides than any other fat. Besides the glycerides of oleic, palmitic, and stearic acids, the usual glycerides of the insoluble or fixed fatty acids found in most fats, butter contains notable quantities of the glycerides of a number of the volatile fatty acids, chief among which are butyric, caproic, capric, caprylic, lauric and myristic, to which are due in part its distinctive taste, and which by exposure to light and air readily become decomposed into the free acids.

The process of separation of butter fat into its component glycerides is a matter of extreme difficulty, and results obtained by different chemists vary widely. Separation has been attempted by fractional distillation, by methods depending on the difference in chemical affinity of the various acids, and on the difference in solubility of the various lower homologues in water at different temperatures.

According to Browne,\* the composition of butter fat is as shown in table on page 552. Holland, Reed and Buckley† by their improved method find 7% to 22% of stearic acid.

The fatty acids are not all combined as simple triglycerides. Amberger,‡ for example, has isolated palmito-distearin and stearo-dipalmitin.

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\* Jour. Amer. Chem. Soc., 21, 1899, pp. 612, 807 and 975

† Jour. Agric. Res., 6, 1116, p. 101.

‡ Zeits. Unters. Nahr. Genussm., 26, 1913, p. 3802.



Acid.	Fatty Acids.	Equivalent Triglycerides.
Dioxystearic.....	1.00	1.04
Oleic.....	32.50	33.95
Stearic.....	1.83	1.91
Palmitic.....	38.61	40.51
Myristic.....	9.89	10.44
Lauric.....	2.57	2.73
Capric.....	0.32	0.34
Caprylic.....	0.49	0.53
Caproic.....	2.09	2.32
Butyric.....	5.45	6.23
Total.....	94.75	100.00

The constants and variables of butter fat are given on pp. 528 and 529.

**Effects of Feeding Oil Cakes on the Composition of Butter.**—Experiments have shown that the substance which causes cottonseed oil to respond to the Halphen test may pass into the milk fat on feeding cows with cottonseed cake, but the substance that gives the Baudouin reaction is never carried into the milk on feeding with sesame cake. A number of investigators have found that feeding with cocoanut cake raises somewhat the Polenske number of the milk fat. There is good evidence, however, that, while the addition of vegetable oils to butter introduces phytosterol, as detected by Bomer's phytosterol acetate test, this substance can not be introduced into the milk fat by feeding. These facts should be borne in mind in the examination of butter for foreign fats.

#### ANALYSIS OF BUTTER.

**METHODS OF PROXIMATE ANALYSIS.**—**Drawing the Sample.**—Butter is particularly difficult to sample owing to the uneven distribution of water and salt. If in bulk, as in a creamery previous to packing, or in tubs, remove a considerable number of cores with a butter trier to a quart or two-pint jar. If in bricks divide a number into quarters by cutting through the middle in two directions at right angles to the surface and take one quarter of each for the composite sample.

Guthrie and Ross \* have reported results which illustrate the difficulties

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\* Cornell Agr. Exp. Sta. Bul. 336, 1913.



of sampling. Of 51 packages from different sources 9 showed for adjacent samples drawn with a trier differences of moisture over 1% and 19 between 0.5 and 1%. They conclude that the exact composition can only be reached by the analysis of a sample made up of many portions taken from different parts of the package.

**Preparation of the Sample.**—*Wiley Method*.\*—Close the jar and heat at about 40° C. in water or in a hot closet until thoroughly melted, *taking care that no lumps remain*. Cool under the tap with continual shaking until thoroughly congealed. The sample should be kept in a cold place till analyzed.

**Determination of Water.**—*Gravimetric Method*.—Dry 2 grams of the sample to constant weight in a flat-bottomed metal dish heated in a boiling water-oven.

*Patrick's Rapid Method*.†—This method is especially suited for the use of dairymen, inspectors and others not provided with laboratory facilities.

Ten grams of the thoroughly mixed butter are weighed into a 250-cc. aluminium beaker, which, together with a glass rod has been previously tared, and boiled over (but not in) the flame of an alcohol lamp provided with a conical asbestos chimney, holding the beaker by means of a wire clamp in a nearly horizontal position to avoid loss from spattering or foaming, and whirling constantly to prevent overheating. The rod serves to break up lumps of curd which form, thus facilitating the drying. The heating should be so conducted as to avoid any considerable discoloration of the curd. With suitable heating the water may be removed in less than 15 minutes, after which the beaker is cooled in water and weighed. A balance sensitive to 10 milligrams, such as is used in weighing cream for testing by the Babcock method, is sufficiently accurate for weighing the butter.

*Gray's Method*.‡—1. *The Special Apparatus*, for this method, shown in Fig. 100, consists of a flask (A) connected by a close-fitting rubber stopper (B) with a graduated tube (C), and this in turn with a condenser jacket (E) by a rubber stopper (D). The tube C is closed by a glass stopper, the zero mark being the end of the stopper. Each mark of the graduation represents 0.02 cc. or, when 10 grams of butter are used, 0.2%.

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\* Proc. A. O. A. C., 1887.

† Jour. Amer. Chem. Soc., 28, 1906, p. 1611; 29, 1907, p. 1126.

‡ U. S. Dept. of Agric., Bur. of Animal Ind., Circ. 100.



2. *Process*.—Weigh 10 grams of the well mixed butter on a piece of

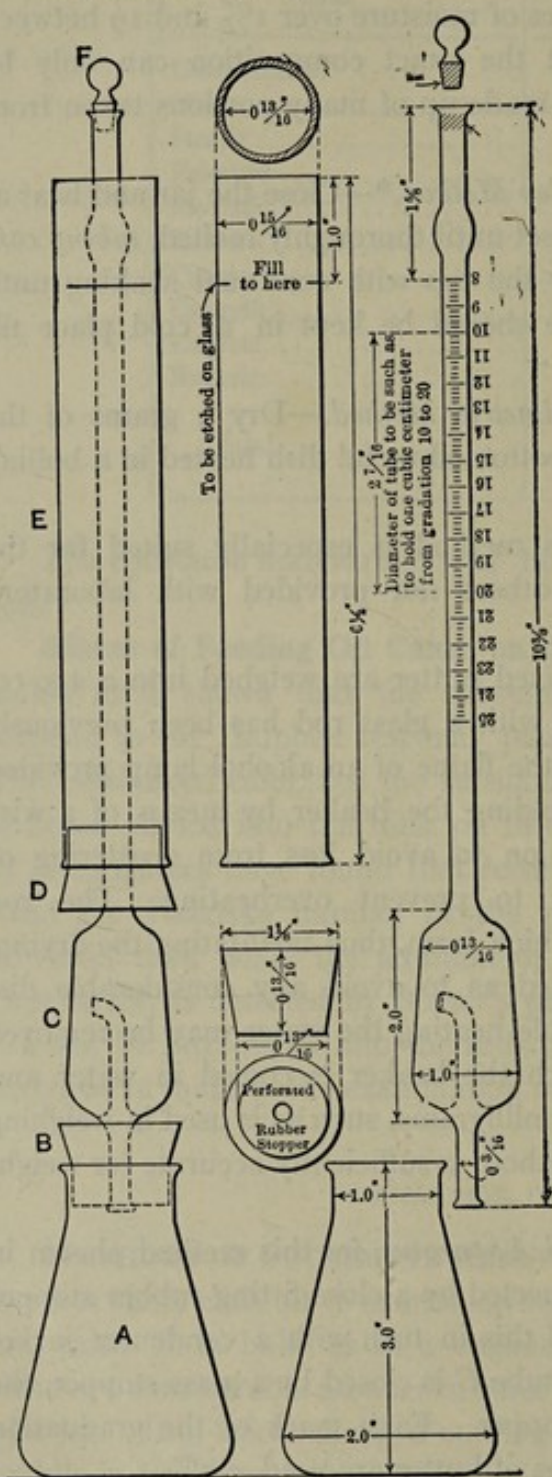


FIG. 100.—Gray's Apparatus for the Rapid Determination of Water in Butter.

parchment paper 13 cm. square, introduce into the flask, and add 6 cc. of a mixture of 5 parts of amyl acetate and 1 part of amyl valerianate, free from water-soluble impurities. Connect the apparatus as shown in Fig. 100, fill the condenser jacket with cool water to within 2.5 cm. of the top, and remove the glass stopper *F*. Heat the flask over a Bunsen burner, thus melting the butter and boiling the water. Watch the condensation of the steam in the graduated part of the tube *C*, and do not allow the steam to get higher than the 15% mark. In case of continued foaming, allow the mixture to cool, add 2 cc. of the amyl reagent, and continue heating. After the water in the sample has boiled out, the temperature rises and the amyl reagent boils, driving the last traces of water and water-vapor from the flask and bottom of the stopper. Some of the amyl reagent is carried into the tube *C* with the steam, and some is boiled over after the water has been driven off. This amyl reagent in the tube is no disadvantage. When the mixture in the flask becomes a brown color and all the crackling noises in boiling cease, which usually requires 5 to 8 minutes, it is safe to conclude

that all water has been driven from the flask.

Disconnect the flask *A* from the stopper *B*, place the glass stopper *F*



in the tube *C*, giving it a turn to insure its being held firmly; invert the tube *C*, first being sure that the mouth of the small tube inside the bulb is held upwards, pour the water from the condensing jacket *E*, and remove the jacket. When the tube *C* is inverted, the water and reagent flow into the graduated part of the tube. To separate these and to get the last traces of water down into the graduated part, the tube *C* is held with the bulb in the palm of the hand, and the stoppered end away from the body, raised to a horizontal position, and swung at arm's length sharply downward to the side. This is repeated a number of times until the dividing line between the water and reagent is very distinct, and no reagent can be seen with the water or *vice versa*. The tube should then be held a short time with the stoppered end downward, and the amyl reagent in the bulb agitated in order to rinse down any adhering water.

The reading should not be taken until the tube and contents have cooled so little warmth is felt. When 10 grams of butter are used, the percentage is read directly at the lower meniscus.

With butter very low in moisture it may be desirable to use 15 grams, and with butter very high, 5 grams.

**Fat.**—This may be determined either directly or indirectly. For the direct determination, a weighed amount of the sample, from 2 to 3 grams, is first dried at  $100^{\circ}$  in sand or asbestos, contained in a thin and fragile round-bottomed evaporating-shell (Hoffmeister's Schälchen). If desired, the moisture may be determined in this connection by loss in weight after drying. The shell is afterwards inclosed in a piece of fat-free filter-paper, and crushed in pieces between the fingers in such a manner as to avoid loss. The pieces are gathered in a mass, and folded together in the filter-paper to form a packet of a size readily transferable to a Soxhlet extractor, in which the fat is removed in the usual manner and weighed, after drying, in a tared flask.

Or, the fat may be indirectly determined by subtracting the sum of the water, casein, and ash from 100.

**Casein.**—*Wiley Method.*\*—The residue from the gravimetric determination of water is stirred with petroleum ether until the fat is dissolved and transferred to a tared Gooch crucible. After thorough washing with petroleum ether, the crucible is dried at  $100^{\circ}$ , cooled, and weighed, thus obtaining the casein and ash. The loss on ignition at a dull red heat represents the casein.

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\* Proc. A. O. A. C., 1887.



If desired, nitrogen may be determined in the residue after removal of the fat with petroleum ether, and casein calculated from the nitrogen, using the factor 6.37.

**Ash.**—The residue left on the Gooch crucible after ignition, obtained as described in the preceding section is the ash. It consists largely of salt, which may be calculated from the percentage of chlorine determined by titration.

**Milk Sugar and Lactic Acid** compose most of the undetermined matter remaining after deducting from the total solids the sum of the fat, casein, and ash. Determine milk sugar, if desired, in an aqueous extract of the butter by Fehling's solution.

**Determination of Salt.**—In a tared dish or beaker weigh out about 5 grams of butter, taking a gram or so at a time from different parts of the sample. Add hot water to the weighed part, and after it has melted, the contents of the dish are poured into a separatory funnel, shaken and allowed to stand till the fat collects at the top, after which the underlying aqueous solution is drawn off into an Erlenmeyer flask, leaving the fat in the funnel bulb. Hot water is again added, and from ten to fifteen extractions are made, using about 20 cc. of water each time, all the water being collected in the Erlenmeyer flask.

A few drops of a solution of potassium chromate are then added for an indicator, and the sodium chloride volumetrically determined by a standard silver nitrate solution.

Salted butter contains from 0.5 to 6% of salt.

**U. S. Standard Butter** is butter containing not less than 82.5% of butter fat. By acts of Congress approved August 2, 1886, and May 9, 1902, butter may also contain added coloring matter.

**DETECTION OF FOREIGN FAT.—Preparation of Fat Sample.**—The butter fat is best obtained by filtering when hot, the sample being melted in a beaker on the water-bath. The water, with the curd and salt, will settle to the bottom. The clear fat is then filtered at a temperature not exceeding 50° C. and subjected to such examination as may be desired to determine its purity.

**Examination of the Fat.**—This is discussed in detail under the head of Distinction of Oleomargarine from Butter, pages 567-571.

**U. S. Standard Butter Fat** has a Reichert-Meissl number not less than 24 and a specific gravity not less than 0.905 at  $\frac{40^{\circ}}{40^{\circ}}$  C.

**DETECTION OF PROCESS BUTTER.**—See pages 571-576.



**ARTIFICIAL COLORING MATTER IN BUTTER.**—Formerly carrot juice and annatto were used almost entirely as butter colors. The carrot furnished to the farmer a ready means of coloring his dairy butter, and its use was long in vogue for this purpose, before the commercial butter colors were available. Other vegetable colors, such as turmeric, marigold, saffron, and safflower, are said to have been used for this purpose, but, with the possible exception of turmeric, the writer is not aware of authentic cases in which they have been found in recent years. While annatto as a butter color is still in use, it is rapidly giving place to various oil-soluble, azo coal-tar colors, which are admirably adapted to the purpose. Butter colors are now put on the market in solution in oil, usually cottonseed in this country and sesame in Europe.

**Detection.**—*Martin* \* devised a general scheme applicable for the detection of various colors in butter. His reagent consists of a mixture of 2 parts of carbon bisulphide with 15 parts of ethyl or methyl alcohol. 25 cc. of this solution are shaken with about 5 grams of the butter to be tested, and, after standing for some minutes, the mixture separates into two layers, of which the lower consists of the fat in solution in the carbon bisulphide, while the upper is the alcohol, which dissolves out and is colored by the artificial dye employed. If *saffron* is present, the alcoholic extract will be colored green by nitric acid and red by hydrochloric acid and sugar.

*Coal-tar dyes*, if present, may be fixed on silk or wool by boiling bits of the fiber in the alcoholic extract, diluted with water and acidulated with hydrochloric acid.

*Turmeric* is to be suspected, if ammonia turns the alcoholic extract brown; *marigold*, if silver nitrate turns it black, and *annatto*, if on evaporating the alcoholic solution to dryness and applying to the residue a drop of concentrated sulphuric acid, a greenish-blue coloration is produced.

*Turmeric* is further tested for in the residue from the alcoholic extract as above obtained, by boiling the residue in a few cubic centimeters of a dilute solution of boric acid (or a solution of borax acidulated with hydrochloric acid), and soaking a strip of filter-paper therein. On drying the paper, if it assumes a cherry-red color, turning dark olive by dilute alkali, the presence of turmeric is assured.

**Test for Carotin.**—This substance, the chief coloring matter of the carrot root and according to Palmer and Eckles † of butter, does not impart

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\* Analyst, 1, p. 70.

† Jour. Biol. Chem., 16, 1914, p. 191.



its color to the alcohol layer in Martin's test. Moore\* has pointed out this exception and shown that with only carotin present the alcohol layer in Martin's test remains colorless. If, however, a drop of very dilute ferric chloride is added and the test-tube shaken, the alcohol will gradually absorb the yellow color from the butter. Care must be taken to avoid an excess of ferric chloride, as very little of this reagent will suffice.

Palmer and Thrum† employ a small crystal of ferric chloride instead of the solution and add it to the hot fat after separation from the alcohol layer. If the crystal is of the right size the yellow color of carotin is replaced by the green color of ferrous chloride which in turn is removed by shaking with 10 cc. of methyl or 95% ethyl alcohol, leaving the fat colorless.

**Detection of Annatto.**—Treat 2 or 3 grams of the melted and filtered fat (freed from salt and water) with warm, dilute sodium hydroxide. After stirring, pour the mixture while warm upon a wet filter, using to advantage a hot funnel. If annatto is present, the filter will absorb the color, so that, when the fat is washed off by a gentle stream of water, the paper will be dyed straw color. It is well to pass the warm alkaline filtrate two or three times through the fat on the filter to insure removal of the color.

If, after drying the filter, the color turns pink on application of a drop of stannous chloride solution, annatto is assured.

**Detection of Coal-tar Colors in Butter.**—*Geisler's Method.*‡—A few drops of the clarified fat are spread out on a porcelain surface and a pinch of fullers' earth added. In the presence of various azo-colors, a pink to violet-red coloration will be produced in a few minutes. Some varieties of fullers' earth react much more readily with the azo-dyes than do others. In fact some do not respond at all. When once a satisfactory sample of this reagent is obtained, a large stock should be secured of the same variety.

*Low's Method.*§—A small amount of material to be tested is melted in a test-tube, an equal volume of a mixture of 1 part of concentrated sulphuric acid and 4 parts of glacial acetic acid are added, and the tube is heated nearly to the boiling-point, the contents being thoroughly mixed by shaking; the tubes are set aside, and after the acid solution has settled out it will have been colored wine-red in the presence of azo-color, while with pure butter fat, comparatively no color will be produced.

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\* Analyst 11, 1886, p. 163.

† Jour. Ind. Eng. Chem., 8, 1916, p. 614.

‡ Jour. Am. Chem. Soc., 20, 1898, p. 110.

§ Ibid., 20, p. 889.



**Doolittle's Method for Azo-colors and Annatto.\***—The melted sample is first filtered. Two test-tubes are taken and into each are poured about 2 grams of the filtered fat, which is dissolved in ether. Into one test-tube are poured 1 or 2 cc. of dilute hydrochloric acid, and into the other about the same volume of dilute potassium hydroxide solution. Both tubes are well shaken and allowed to stand. In the presence of certain azo-dyes, the test-tube to which the acid has been added will show a pink to wine-red coloration, while the potash solution in the other tube will show no color. If annatto has been used, on the other hand, the potash solution will be colored yellow, while no color will be apparent in the acid solution.

**Cornelison's Test for Artificial Colors.†**—Melt 10 grams of the clear, dry fat, and shake well in a separatory funnel with 10 to 20 grams of 99.5% acetic acid. If the materials are too hot, the fat will dissolve, but at about 35° it separates quickly and almost completely. Draw off the clear acid, and after noting its color, test by adding to one portion of 5 cc. a few drops of concentrated nitric acid, and to another portion a few drops of concentrated sulphuric acid.

Natural yellow butter gives by this test a colorless extract, which remains colorless on adding nitric acid and becomes a faint pink color on adding sulphuric acid. The acid extracts containing annatto, curcumin, and carrot are various shades of yellow, both before and after addition of nitric acid, while with sulphuric acid they take on a pink coloration on standing, which in the case of curcumin is very decided. Soudan I and butter yellow give pink extracts, which remain pink on adding the stronger acids, while cerasine orange G, yellow O.B., yellow A.B. and certain other coal-tar dyes give extracts of various shades of yellow, which on treatment with the heavy acids in some cases remain colorless, but in others become pink, while the oil globule which separates remains colorless or takes on a pinkish color according to the dye.

**Mathewson's Method ‡** is quite generally applicable although the color is somewhat contaminated with sterols. Dilute 30 cc. of the melted fat with 120 cc. of low-boiling gasoline and shake out with several portions of a mixture of 90 parts of phenol with 10 of water, using first 45 cc. and then 30 cc. Wash the extract with 2 or 3 portions of gasoline, treat with sufficient cool, strong potassium or sodium hydroxide solution to dissolve the

\* U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 152.

† Jour. Am. Chem. Soc., 30, 1908, p. 1478.

‡ U. S. Dept. of Agric., Bul. 448, 1917, p. 6.



phenol, and shake out with 50 to 100 cc. of ether. Wash the ether with caustic alkali solution to remove all phenol and finally with water, then evaporate or treat further as indicated below and on page 863.

To extract the Soudan dyes shake out the gasoline solution of the fat once or twice with a mixture of 80 parts of 85% phosphoric acid (sp. gr. 1.70) and 20 parts of concentrated sulphuric acid, but the method is not applicable to tolueneazo- $\beta$ -naphthylamin and other colors sensitive to strong acids. The alkali salts of Soudan G and annatto color, being readily soluble in water, are most easily removed by shaking out with dilute sodium or potassium hydroxide solution.

Extraction and separation may be carried out together as follows: Dilute the melted fat with gasoline and shake out first with 2% (N/2) sodium hydroxide solution to remove annatto, Soudan G, etc., and then wash several times with 4 to 6 N hydrochloric acid to remove aminoazo derivatives such as butter yellow and aminoazotoluene. Benzeneazo- $\beta$ -naphthylamin and tolueneazo- $\beta$ -naphthylamin are extracted rather slowly, apparently suffering rearrangement from hydrazo-imin into true azo form before going into solution in the acid. Neutralize the acid extract immediately to avoid decomposition. Separate the Soudans and similar colors not extracted by alkali or acid with phosphoric acid mixture as described above, wash once or twice with gasoline, dilute, partially neutralize, and extract with ether or gasoline, thus obtaining the color.

**PRESERVATIVES AND THEIR DETECTION.**—Fresh or unsalted butter and renovated butter may contain a preservative. Formerly the one most commonly used for this purpose was the so-called "boric mixture" (borax and boric acid) well known as a milk preservative. In England the use of borax or boric acid is permitted. Sodium benzoate may be used in the United States if properly declared, but dairymen seldom take advantage of the ruling in the case of butter designed for domestic use. Some brands of nut butter contain sodium benzoate. Other preservatives less often used in butter are formaldehyde and salicylic and sulphurous acids.

**Boric Acid.**—This, if present, is best detected in the aqueous solution that settles to the bottom when butter is melted at the temperature of the boiling water-bath, the supernatant fat being decanted off. Richmond\* claims to be able to distinguish free boric acid from borax as follows: If on applying turmeric-paper directly to the aqueous liquid the paper turns red, the color being especially evident on drying, free boric acid is indicated.

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\* Dairy Chemistry, p. 254.



As a confirmatory test the reddened turmeric-paper is treated with dilute caustic alkali, whereupon it turns a dark olive-green if boric acid is present.

In the absence of a red color by the above test, or when this color is faint, the aqueous solution is acidified slightly with hydrochloric acid and the turmeric-paper applied as before. If borax be present to an appreciable extent, the red color will now be quite marked, even though not appearing before. In other words, testing with turmeric-paper without acidifying with hydrochloric acid shows, according to Richmond, a slight coloration due to the free acid alone, while the more intense color formed by first acidifying is due to the combined acid or borax.

*Determination of Boric Acid.*—Ten grams of the butter fat are weighed in a beaker and transferred with hot water to a separatory funnel in which the fat is extracted with 10 to 15 portions of hot water as described on page 556. The combined aqueous extract is evaporated to dryness in a platinum dish, the residue made alkaline, and ignited at a dull red heat. Boil the ash with water, filter, and wash with hot water, keeping the volume of the filtrate under 60 cc. Make sure that the solution is perfectly neutral to methyl orange by treatment, if necessary, with sulphuric acid and tenth-normal alkali, add 30 cc. of glycerin, a few drops of the phenolphthalein indicator, make up to 100 cc., and titrate with tenth-normal sodium hydroxide according to Thompson's method (page 886).

Butter being practically free from phosphates, the preliminary treatment for removing phosphoric acid in Thompson's method may be omitted.

**Formaldehyde.**—The aqueous solution, from which the fat of the butter melted at low temperature has been poured off, is added to some milk previously found free from formaldehyde, and the test for the latter with hydrochloric acid and ferric chloride is tried directly in the milk.

**Salicylic Acid.**—*Detection.*—See method No. 2 for detection in milk, page 167.

*Determination of Salicylic Acid.—Method of the Paris Municipal Laboratory.*—Repeatedly exhaust 20 grams of butter in a separatory funnel with a solution of sodium bicarbonate, thus obtaining soluble sodium salicylate, if salicylic acid be present. Acidulate the aqueous extract with dilute sulphuric acid, and extract with ether. Evaporate the ether, and to the residue add a little mercuric nitrate, forming a precipitate nearly insoluble in water. Filter this off, wash the precipitate with water, and decompose into free salicylic acid with dilute sulphuric acid. Redissolve in ether, evaporate the solvent as before, and dry the



residue at a temperature of  $80^{\circ}$  to  $100^{\circ}$ . Extract the residue with petroleum ether, dilute the ethereal liquid with an equal volume of 95% alcohol, and titrate with tenth-normal alkali, using phenolphthalein as an indicator.

1 cc. of tenth-normal alkali = 0.0138 gram salicylic acid.

**Benzoic Acid.**—*Detection.*—*Halphen-Robin Method.*\*—Dissolve 0.4 to 0.5 gram of sodium bicarbonate in 50 cc. of water and 15 cc. of 95% alcohol in a separatory funnel, add 25 grams of the melted sample, agitate with a rotary motion, allow to stand 6 minutes, and draw off the alkaline liquid into a flask. Acidulate the contents of the flask with 7 or 8 drops of concentrated sulphuric or hydrochloric acid, heat nearly to boiling, add a little talc or infusorial earth, shake 1 to 2 minutes, and filter on a folded paper returning the first portions that run through. Cool the filtrate, shake out with 40 cc. of ether, wash the ether extract once with a mixture of 20 cc. of water and 5 cc. of 95% alcohol, then shake with a mixture of 20 cc. of water, 5 cc. of 95% alcohol, and 0.2 to 0.3 gram of sodium bicarbonate. Draw off the alkaline solution into a dish and test by the modified Mohler method (page 892).

**Sulphurous Acid.**—The aqueous liquid, separated from the butter fat, is distilled, and the distillate treated with bromine water and barium chloride. A precipitate on the addition of the latter reagent indicates the presence of sulphurous acid or a sulphite in the butter.

**Glucose in Butter.**—Crampton † states that glucose has been found by him in butter intended for export to tropical countries, added to prevent decomposition. In one sample made for export to Guadeloupe he found over 10% of glucose.

For its detection or estimation 10 grams of the sample are weighed out and transferred to a separatory funnel with hot water, and shaken out with successive portions of hot water. These are combined, and the aqueous extract made up to 250 cc. The reducing sugar may be determined by Fehling's solution or by polarization, using in the latter case alumina cream as a clarifier. While a slight reduction should be disregarded, any considerable reduction may be undoubtedly ascribed to glucose.

**BUTTER "FILLED" WITH WATER.**—Various preparations have been placed on the market to aid in incorporating water with butter. So-called

\* Ann. chim. anal. appl., 13, 1908, p. 431.

† Jour. Am. Chem. Soc., 20, 1898, p. 201.



"black pepsin" has been used for this purpose. By churning the butter with water and a certain amount of the preparation in such a manner as to destroy the grain, it is possible to introduce two or three times the normal amount of water.

#### RENOVATED OR PROCESS BUTTER.

This product is also variously termed "boiled," "aerated," and "sterilized" butter. There are various modifications of the process of manufacture, but the object is to melt up and treat rancid butter in such a manner that for a time at least it is sweet. The following manner of treatment is typical, and shows in the main the necessary steps in carrying out the process, though details of manipulation vary in different localities.

The butter is melted in large tanks surrounded with hot-water jackets at a temperature varying from  $40^{\circ}$  to  $45^{\circ}$  C. By this means the curd and brine settle to the bottom, whence they are drawn off, while the lighter particles rise to the top in the form of a froth or scum and are removed by skimming.

The clear butter fat is then, as a rule, removed to other jacketed tanks, and, while still in a molten condition, air is blown through it, which removes the disagreeable odors. The melted fat is then churned with an admixture of milk (more often skimmed) till a perfect emulsion is formed, after which it is rapidly chilled by running into ice-cold water, with the result that it becomes granular in form. It is then drained and "ripened" for some hours, after which it is worked free from excess of milk and water, salted, and packed.

Under some state laws this product, to be legally sold, must conform to rules of labeling as strict as those prescribed for oleomargarine. In other localities it may be sold with impunity. Not infrequently it is sold as choice creamery butter, and sometimes at the same price.

**U. S. Standard Renovated or Process Butter** should contain not more than 16% of water, and at least 82.5% of butter fat.

#### OLEOMARGARINE.

According to the U. S. revenue laws, artificial butter composed wholly or in part of fat other than butter fat must be branded oleomargarine. The name butterine, although used in advertising matter, does not have the sanction of the government. The product is commonly known in England as margarine. As a rule the oleomargarine of commerce is



composed of refined oleo oil, usually churned up with neutral lard, milk, and a small amount of pure butter, the whole being salted and sometimes colored to resemble butter. Cottonseed oil and other vegetable oils are also used to some extent.

**Oleo Oil** is prepared from the fat of beef cattle somewhat as follows.\* Immediately after the animals are killed the fresh intestinal and caul fats are removed and placed in tanks of water at a temperature of about 80° F. From this water they are transferred to other tanks of cold water and chilled until all animal heat is removed. The fat is then cut or hashed into small pieces and rendered in jacketed kettles, the temperature being kept as low as possible (formerly about 150° F., now less than 110° F.) to avoid a cooked taste, salt being used to aid the separation.

The melted fat (*premier jus*) is drawn off from the connective tissue, clarified, and allowed to crystallize for one or more days in graining or seeding vats at about 85° F.

From these vats the semi-solid emulsion of oil and stearin is dipped into cloths, which are folded and placed in a press between sheets of metal and subjected to powerful pressure. By this means the oil is separated from the stearin, and is drawn into casks for export or for manufacture into oleomargarine. Large quantities are annually exported to Holland, where oleomargarine is manufactured, and either sold for consumption in that country, or re-exported to other countries in Europe.

The oleo oil thus expressed is a mixture of olein and palmitin. When first prepared, it is a clear amber-colored fluid, free from odor or fatty taste. It is packed in tierces, and, when opened at ordinary temperature, is a light-yellow solid.

The further process of manufacture of oleomargarine as formerly conducted consists in the main of mixing the oleo oil as above obtained with varying proportions of neutral lard, milk, and genuine butter, with or without added coloring matter, and churning the mixture at a temperature above the melting-point of the fats, the neutral lard having previously been cured for at least forty-eight hours in salt brine. After the churning, the whole mass is cooled by contact with ice water. The chilled mass is drained, and afterwards salted, worked, and given much the same treatment as butter.

The composition of this type of oleomargarine varies between the

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\* Report on Oleomargarine, Its Manufacture and Sale, 19th Ann. Report, Mass. St. Bd. of Health, 1887.



following limits: Oleo oil, 20 to 25%; neutral lard, 40 to 45%; butter, 10 to 25%; milk, cream, salt, etc., 5 to 30%.

Since the manufacture of cottonseed oil has been carried out on a large scale this oil has been used in considerable quantity in the United States for mixing with animal fats in the production of artificial butter. In certain continental countries the addition of a certain amount of sesame oil (in Germany 10% of the total fat) is required by law for the purpose of distinguishing the product from genuine butter by the Baudouin or some similar test. These mixtures respond respectively to the Halphen and Baudouin tests, although not appreciably different from animal oleomargarine in their content of soluble and insoluble fatty acids (Reichert-Meissl and Polenske numbers) and insoluble fatty acids (Hegner number). Like oleomargarine of purely animal origin they are also readily distinguished from butter by their refraction, saponification number, and several other constants.

Vegetable oleomargarine prepared chiefly from cocoanut and palm kernel oils is a quite different product, as noted in a subsequent section.

**Coloring of Oleomargarine.**—The artificial coloring matters employed are the same as in the case of butter, and are similarly tested for.

In many states oleomargarine cannot be legally sold when colored to resemble butter. Under other state laws coloring matter is allowable. The federal law and most state laws prescribe the most rigid rules for marking packages containing oleomargarine, with a view to affording the utmost protection to the producer of butter against the fraudulent substitution therefor.

**Sulphurized Oil.**—Cottonseed oil treated with sulphur by a secret process has recently been used in oleomargarine. The sulphur is claimed to improve the oil, but the fact that a yellow color is imparted is significant. Sulphurized oil blackens silver foil or a silver coin when heated at about 250° C. Quantitative tests are said to furnish no evidence.

**Crampton and Simon's Test for Palm Oil.\***—So called "butter oils," consisting of cottonseed oil to which has been added 2 to 5% of palm oil are used to color oleomargarine. The following tests serve for the detection of palm oil.

*Preparation of Sample.*—The sample should be kept in a cool, dark place until tested, as exposure to air and light, or the presence of water, alcohol, ether or similar reagents interfere with the tests. Immediately

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\* Jour. Am. Chem. Soc., 27, 1905, p. 270.



before testing, the sample is filtered as quickly as possible at a temperature not exceeding 70° C.

*First Method.*—Dissolve 100 cc. of the fat in 300 cc. of petroleum ether, and shake out with 50 cc. of 0.5% potassium hydroxide. Draw off the watery layer, make distinctly acid with hydrochloric acid, and shake out with 10 cc. of colorless C. P. carbon tetrachloride. Separate the carbon tetrachloride solution, transfer a portion to a porcelain crucible, add 2 cc. of a mixture of one part of colorless, crystallize C. P. phenol and 2 parts of carbon tetrachloride, then 5 drops of hydrobromic acid (sp. gr. 1.19), and mix by gentle agitation.\*

The almost immediate development of a bluish-green color is indicative of palm oil.

*Second Method.*—Shake 10 cc. of the melted and filtered fat with an equal volume of colorless C. P. acetic anhydride, add one drop of sulphuric acid (sp. gr. 1.53), and shake a few seconds longer.†

If palm oil be present, the lower layers on settling out will be found to be colored blue with a tint of green. The color in this as in the preceding test is transient.

Of the edible oils only sesame and mustard oils give a similar color reaction. Sesame oil, after repeated extractions with alcohol, will not give the blue color, but cottonseed oil containing as little as 1% of palm oil still responds to the test.

Gill ‡ believes this test unreliable because it does not give concordant results in the hands of different chemists and being a test for carotin is not characteristic of palm oil.

**Adulterants of Oleomargine.**—This product is liable to adulteration not only by the use of inferior and unwholesome fat, but by the admixture in some cases of paraffin.§ This sophistication is made manifest if an appreciable amount of the adulterant has been used, by the high melting-point and the low saponification number, as well as by the low specific gravity. If a clear saponification is impossible under ordinary conditions, paraffin is to be suspected. It may be separated and quantitatively determined as described on page 527.

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\* Halphen uses a similar reagent to detect rosin oil in mineral oil. Jour. Soc. Chem. Ind., 21, 1902, p. 1474.

† The reagents are the same as used in the Liebermann-Storch test for rosin oil.

‡ Jour. Ind. Eng. Chem., 9, 1917, p. 136.

§ Geissler, Jour. Am. Chem. Soc., 21, 1899, p. 605.



**Healthfulness of Oleomargarine.**—Under the directions of the Massachusetts Board of Health,\* a large number of artificial digestion experiments were made to show the relative nutritive value of butter and oleomargarine, and at the same time the wholesomeness of oleomargarine as a food was carefully investigated. The general conclusions reached were that, when comparing the best grades of both products, there is little if any difference between butter and oleomargarine on grounds of digestibility, while a good oleomargarine is much to be preferred to a poor butter from a nutritive standpoint. As to its wholesomeness, a large number of experts consulted were unanimous in expressing their favorable opinions of oleomargarine as a healthful article of food.

When sold on its own basis in accordance with the law, it forms an excellent cheap substitute for butter. It is only when fraudulently sold as butter or in violation of the various state and federal laws, that it comes within the province of the health authorities to condemn it, and, unfortunately, by reason of its close resemblance to the dairy product, the temptation to sell it for what it is not is always great.

**DISTINCTION OF OLEOMARGARINE FROM BUTTER.**—The two products resemble each other closely in general appearance, consistency, and somewhat in flavor. Their distinction involves preliminary organoleptic test followed by physical and chemical examination.

**Odor and Taste.**—It is easy with a little practice to become so accustomed to the odor and taste of oleomargarine, as to be able to pass judgment with considerable confidence by these senses alone, whether a sample in question is oleomargarine or butter. The distinction is rendered more apparent by melting a portion of the sample on the water-bath. If the product is butter, either fresh or renovated, the butyric odor of the melted fat is very characteristic, while the melted oleomargarine not only is lacking in the butyric odor (a negative property), but possesses often a "meaty" smell peculiar to itself, which, while not unpleasant, is unmistakable. The flavor of oleomargarine although not always the same to one experienced in distinguishing between the two products is very apparent. This flavor, though somewhat variable, may be compared to that of cooked meat.

**Qualitative Tests** for cottonseed and sesame oils are made by the usual color methods and for peanut oil by Renard's or Bellier's method.

**Range of Constants.**—By reference to the tables on pages 528 and 529 it is evident that the constants of oleo oil and lard are very similar but radically

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\* 19th Ann. Report, Mass. State Board of Health, 1887, p. 248.



different from those of butter fat, furthermore that the addition of cottonseed or sesame does not interfere with the ready distinction of oleomargarine from butter. For ordinary purposes it is necessary only to determine the refraction and the Reichert-Meissl number coupling the determination of the latter with that of the Polenske number in case the presence of an oil of the cocoanut oil group is suspected. The presence of cottonseed oil in the American oleomargarine, readily responding to the Halphen test, serves the same purpose as sesame oil compulsorily added to the continental product. Other constants and tests are discussed on p. 571.

The distinction between butter fat, animal fat such as goes to make up oleomargarine, and mixture of the two is brought out in the following table by Villiers and Collin\*:

	Hehner's Number.	Soluble Acids.	Koettstorfer's Equivalent.	Volatile Acids.
Pure butter. ....	88	5	224	26
Butter, 95% foreign fat, 5% .....	88.35	4.8	222.6	24.7
" 90% " " 10% .....	88.70	4.5	221.2	23.4
" 85% " " 15% .....	89.05	4.3	219.8	22.2
" 80% " " 20% .....	89.40	4.1	218.4	20.9
" 75% " " 25% .....	89.75	3.9	217	19.6
" 70% " " 30% .....	90.10	3.6	215.6	18.3
" 65% " " 35% .....	90.45	3.4	214.8	17.1
" 60% " " 40% .....	90.80	3.2	212.8	15.8
" 55% " " 45% .....	91.15	3	211.4	14.5
" 50% " " 50% .....	91.50	2.7	210	13.2
" 45% " " 55% .....	91.85	2.5	208.6	12
" 40% " " 60% .....	92.20	2.3	207.2	10.7
" 35% " " 65% .....	92.55	2.1	205.8	9.4
" 30% " " 70% .....	92.90	1.8	204.4	8.1
" 25% " " 75% .....	93.25	1.6	203	6.9
" 20% " " 80% .....	93.60	1.4	201.6	5.6
" 15% " " 85% .....	93.95	1.2	200.2	4.3
" 10% " " 90% .....	94.30	0.9	198.8	3
" 5% " " 95% .....	94.65	0.7	197.4	1.8
Foreign fat. ....	95	0.5	196	0.5

**Butyro-refractometer Readings.**—The instrument, as its name implies, was primarily intended by Zeiss for the examination of butter, and, while its use has been extended for work with other fats and oils, its construction is such as to show particularly a distinction between butter and oleomargarine by the appearance of the critical line of the fat. This mode of differentiation is due to the peculiar construction of the double prism, which shows differences of dispersive power by different appearances of the critical line. The prisms are so constructed that the critical

\* Les Substances Alimentaires, p. 731.



line of pure butter is colorless, while margarine and artificial butter, which have greater dispersive powers than natural butter, show a blue-colored critical line. But anomalies in the color, both with pure butter and mixtures, are more or less observable, which render it impossible to draw a sharp line between adulterated and genuine butter. The appearance of a blue fringe may, however, be a useful factor in cases of suspected adulteration.

The following figures for index of refraction and butyro-refractometer readings at 25° C. are by Wollny.\* The oleomargarine was undoubtedly free from cocoanut and palm kernel oils:

Natural butter.....	1.4590-1.4620; 49.5-54.0 scale divisions
Oleomargarine.....	1.4650-1.4700; 58.6-66.4 scale divisions
Mixtures (artificial butter).....	1.4620-1.4690; 54.0-64.8 scale divisions

*Limit of Scale Reading for Pure Butter.*—Whenever in the refractometric examination of butter at a temperature of 25° C. higher values than 54.0 are found for the critical line, these samples will, according to Wollny, by chemical analysis always be found to be adulterated; but with all samples in which the value for the position of the critical line does not reach 54.0 chemical analysis may be dispensed with, and the samples may be pronounced to be pure butter. Wollny suggests, as a means of removing all chances of adulterated butter escaping detection, that the above limit be placed still lower, and that all samples exhibiting values exceeding 52.5 (at a temperature of 25° C.) be set aside for chemical analysis.

In calculating the position of the critical line for other temperatures than 25° C. allow per 1° C. variation of temperature a mean value of 0.55 scale division.† The table on page 570, which has been compiled in this manner, shows the values corresponding to various temperatures, each value being the upper limit of scale divisions admissible in pure butter.

If, therefore, at any temperature between 45° and 25° values be found for the critical line which are less than the values corresponding to the same temperature according to the table, the sample of butter may safely

\* Schlussbericht über die Butteruntersuchungsfrage, Milchwirtschaftlicher Verein, Korrespondenzblatt, No. 39, 1891, p. 15.

† With natural butter this number is, as a rule, somewhat less (0.52), with oleomargarine a little greater (0.56).



Temperature.	Scale Division.	Temperature.	Scale Division.	Temperature.	Scale Division.	Temperature.	Scale Division.
45°	41.5	40°	44.2	35°	47.0	30°	49.8
44°	42.0	39°	44.8	34°	47.5	29°	50.3
43°	42.6	38°	45.3	33°	48.1	28°	50.8
42°	43.1	37°	45.9	32°	48.6	27°	51.4
41°	43.7	36°	46.4	31°	49.2	26°	51.9
40°	44.2	35°	47.0	30°	49.8	25°	52.5

be pronounced to be natural, i.e., unadulterated butter. If the reading shows higher numbers for the critical line, the sample should be reserved for chemical analysis.

*Note.*—Dr. Eichel of Metz has suggested that instead of comparing the scale divisions at the same temperature, the position of the critical line may be determined at the moment when the butter begins to set. In this case he gives fifty-four as the highest admissible number for the critical line of pure butter.

No sharp distinction is apparent between pure and renovated butter on the refractometer.

#### Special Thermometer for the Butyro-refractometer.—

Instead of employing the ordinary thermometer, as shown in Fig. 36, a special thermometer (Fig. 101) has been devised for work both with butter and with lard. This instrument has two scales, arranged side by side, one for butter and one for lard, each of which indicates at once the highest allowable reading for the pure fat, corresponding to the temperature at which the observation is made, which, however, need not be noted.

If the scale reading of the instrument, as observed through the telescope, differs materially from the reading of the special thermometer, the fat under examination is undoubtedly adulterated, or, in the case of butter, a higher reading indicates oleomargarine. The special thermometer thus indicates the highest permissible number for pure butter.

#### The Reichert-Meissl Number (see page 497) is by far the

most important single determination in establishing proof of the character of the sample, whether butter or oleomargarine, for evidence in court, and in such cases this determination is indispensable. The result is conclusive, excepting in those instances



FIG. 101.—Special Butyro-refractometer Thermometer for Butter and Lard.



(rare in the United States) where the admixture of animal oleomargarine is small or the foreign fat is cocoanut or palm kernel oil skillfully proportioned to the butter (see table, page 500).

It is difficult to fix a minimum figure below which, in doubtful cases, a sample may be pronounced impure by reason of admixture with foreign fat. In general, however, a Reichert-Meissl number under 20 would be almost sure to show adulteration, though instances are on record where butter of known purity has fallen even lower than this. It is in fact rare that pure butter has a number under 24.

Stebbins \* gives the maximum, minimum, and average of the Reichert number obtained by him on 317 samples of unadulterated butter, some of which were of low grade, as follows: Maximum, 18.2; Minimum, 11.2; Average, 14.7. These figures should be multiplied by 2 for comparison with Reichert-Meissl numbers.

As a rule little difference is apparent between pure and "renovated" samples as regards their Reichert-Meissl numbers.

Vieth has shown that the Reichert number of butter is generally a trifle lower after it becomes rancid.

**The Polenske Number** is valuable in detecting cocoanut or palm kernel oil in butter as is obvious from a study of the figures given in the table on page 500. Other methods which have been proposed for the purpose are the Jensen-Kirschner method (page 501), the Avé-Lallement method,† the Robin method,‡ the Shrewsbury-Knapp method,§ and the Hanus method.||

The Halphen and Baudouin tests serve to detect cottonseed and sesame oils respectively.

**DISTINCTION OF BUTTER, PROCESS BUTTER, AND OLEOMARGARINE.**—With the increased occurrence in the market of the commercial product known as "process" butter, especially in localities where its sale is restricted or regulated by law, it becomes incumbent on the analyst to distinguish it from the other products which it resembles.

As a rule, the tests, chiefly physical, that are applied on the edible product as a *whole* (i.e., without separation of the curd, salt, etc.), such as the foam test, the milk test, the microscopical examination, and the appearance of the melted sample, distinguish broadly between pure fresh butter

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\* Jour. Amer. Chem. Soc., 21, 1899, p. 939.

† Zeits. Unters. Nahr. Genussm., 14, 1907, p. 318.

‡ Compt. rend., 143, 1906, p. 512; 8th Int. Cong. App. Chem., 18, 1912, p. 305.

§ Analyst 35, 1910, p. 385; 37, 1912, p. 3.

|| Zeits. Unter-Nahr. Genussm., 13, 1907, p. 18.



on the one hand, and oleomargarine on the other. In other words, although there are those skilled in making the above tests who claim to be and doubtless are able to note distinguishing features between oleomargarine and process butter, yet these two products respond alike, though perhaps in varying degrees, to these tests, and are classed together as distinguished from pure butter.

On the other hand, such tests as depend upon the refractometer, the Reichert-Meissl number, and, indeed, all the chemical constants which are applied to the separated fat, freed from other substances, will serve to distinguish between oleomargarine and butter, whether "process" butter or otherwise, since the "processing" or "renovating" of butter does not change the character of its fat sufficiently to materially alter these constants.

It is best, therefore, for purposes of routine preliminary separation to submit *all* samples to the "foam" test and to examine them by the butyro-refractometer. These tests alone, which are very quickly and readily applied, will rarely fail to separate into the three classes, butter, process butter, and oleomargarine, the products under examination, after which such confirmatory tests as are desired are made on adulterated samples.

**The Foam Test**, also known as the "boiling" or "spoon" test.\* This, though originally intended as a household test, is in reality one of the very best laboratory methods of separating pure butter samples from renovated butter and oleomargarine. A small lump of the sample (from 3 to 5 grams) is heated in a large spoon over a Bunsen flame, turned very low, stirring constantly during the heating. Genuine butter, under these conditions, will boil quietly, but with the production of considerable froth or foam, which will often swell up over the sides of the spoon, when, just after boiling, the latter is raised from the flame. Renovated butter or oleomargarine, under this treatment, will bump and sputter noisily like hot grease containing water, but will not foam.† Another point of difference is that on removing the spoon from the flame and observing the character of the curdy particles, in the case of genuine butter these particles of curd will be very small and finely divided in the melted fat, being indeed hardly perceptible, while with oleomargarine and renovated butter, the curd will gather in somewhat large masses or lumps.

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\* U. S. Dept. Agric., Farmers' Bul. 131.

† A very slight foam is sometimes observable with occasional renovated samples, but nothing like the abundant amount produced by the genuine product.



The test may be carried out in a test-tube if desired.

**The Waterhouse or Milk Test.\***—This test is based on the assumption that butter fat, which is in itself exclusively the product of milk, will mingle intimately with the milk when added thereto in a melted condition and cooled therein, whereas oleomargarine, being foreign to milk fat, will, under like conditions, refuse to diffuse itself naturally in milk as a medium.

About 50 cc. of well-mixed sweet milk are heated nearly to boiling in a beaker, and from 5 to 10 grams of the fat sample are added. The mixture is then stirred, preferably with a small wooden stick, until the fat is melted. The beaker is then placed in a dish of ice-cold water, and the stirring continued till the fat reaches the solidifying-point, at which period, if the sample is oleomargarine, the fat can readily be collected by the stirrer into one lump or clot, but, if butter, it cannot be so collected, but remains in a granulated condition, distributed through the milk in small particles. It is not necessary to keep up the stirring through the entire term of cooling, but to begin stirring before the fat starts to solidify, which should require from 10 to 15 minutes after the mixture is placed in cold water.

This test, if carefully carried out, shows a marked distinction between butter, whether pure or renovated, and oleomargarine. Under certain conditions, as when the cooling is too rapid, samples of renovated butter fat will sometimes show a slight tendency to clot together as in the case of oleomargarine, but to no such extent as the latter.

The authors' experience with this test has shown it to be very reliable, not only in identifying oleomargarine from butter, but in nearly every case renovated butter can be distinguished from genuine. As a rule, genuine butter fat, even after cooling to the solidifying-point, shows the greatest tendency to emulsify with the milk when stirred, without adhering to the wooden rod, and is slow to come to the surface when the stirring is stopped. Renovated butter fat, when stirred in the cold milk, almost instantly gathers in a film on the surface of the milk when the stirring is stopped, without emulsifying. It does not clot together like oleomargarine, but it tends to adhere to the wooden rod.

Patrick † recommends the use of skimmed or partially skimmed milk, and heats to the boiling-point after the fat has been introduced into the hot milk.

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\* Parsons, Jour. Amer. Chem. Soc., 23, 1901, p. 200.

† Farmer's Bulletin, No. 131.



**Examination of the Curd.**—The curd of genuine butter is made up largely of such of the milk proteins as are insoluble in water, and hence pass into the cream when separated. These proteins form a gelatinous mass in the butter, readily clotting together when the fat is melted. On the other hand, the curd of process butter, which is, as it were, artificially derived from the entire or skim milk used in its manufacture (in order to replace the natural curd which has been removed in the "purifying" process), differs from the proteins of cream in that it is granular and flaky, consisting chiefly of coagulated casein. Hence the distinction noted as to the appearance of the curd in the foam test.

For the same reason, if beakers containing pure and renovated butter are melted on the water-bath, the curd of the pure sample will settle at once, or in a very few minutes, to the bottom after melting, leaving a comparatively clear supernatant fat. The renovated sample will nearly always fail to settle out clear, even after standing on the water-bath for half an hour or more, but will still be cloudy throughout the mass, due to particles of non-cohesive, floating curd.

In the case of oleomargarine, the curd of which is composed partly of pure butter curd (from cream proteins) and partly of the proteins of the milk with which it is churned, the cloudiness of the fat on melting depends on the relative proportion of milk proteins, and in general is not especially characteristic.

**Identification of the Source of the Curd.\*** Half fill a small beaker with the sample and melt on the water-bath. Decant as much as possible of the fat and pour the rest, consisting largely of the water, salt, and curd, upon a wet filter. Acidify the filtrate, which contains the salt and soluble proteins, with acetic acid and boil. If the sample is pure butter, only a slight milkiness is found, indicating absence of albumins, whereas, in the case of process butter, a white, flocculent albuminous precipitate is produced.

Apply to the filtrate also Liebermann's test for albumin; i.e., add strong hydrochloric acid. If a violet coloration is produced, the sample is presumably "process" butter.

**Microscopical Examination of Butter.**—Considerable information may in general be gained by an examination of the sample under ordinary light and with a rather low power, say from 120 to 150 diameters. For examination in this way a bit of the sample on the edge of a knife blade

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\* Hess and Doolittle, Jour. Am. Chem. Soc., 22, 1900, p. 151.



is placed on the glass slide, and simply pressed lightly into a thin film by the cover-glass. A very characteristic difference between genuine and renovated butter is at once seen in the relative opacity of the fields. The fat film, in the case of the fresh, pure butter, is much more transparent than that of the renovated. Again, the curd is so finely divided throughout the mass of genuine butter fat that the field is much more even than that of the renovated, wherein often large and opaque patches of curd are frequently distributed throughout the field.

When a renovated butter sample, mounted as above, is viewed by *reflected light*, for which purpose the microscope mirror is turned so as not to transmit light through the instrument, one sees a very dark and scarcely perceptible field; but the opaque patches of curd above referred to are strikingly apparent as white masses against a dark background.

*With Polarized Light.*—It has already been stated that the microscope is useful in showing whether or not a fat has been melted, the crystalline structure of the fat once melted and afterward cooled being rendered apparent, especially when viewed by polarized light. This fact has long been known and put to practical use in the identification microscopically of butter and oleomargarine.\*

When viewed by polarized light between crossed Nicols under a low magnification, pure butter not previously melted should show no crystalline structure, being uniformly bright throughout, and, if the selenite plate be used, should present an even colored field, entirely devoid of fat crystals. On the other hand, with process butter or oleomargarine, both of which have been melted and subsequently cooled the crystalline structure should be marked, showing with polarized light a more or less mottled appearance, and a play of colors with the selenite.

Various conditions enter in to affect the reliability of the polarized light test. It is nearly always possible in cold weather to observe these distinctions in practice, as above described, in a sharp and striking manner. Figs. 269, 270, and 271, Pl. XXXVIII, show typical fields of the three products with crossed Nicols and selenite plate. The appearance of pure butter is perfectly blank, while oleomargarine presents a much more mottled appearance than renovated butter. Such well-defined points of variation as are shown in Pl. XXXVIII are not always to be seen in practice, even in the hands of an expert. Pure butter sometimes exhibits a somewhat mottled field, due to a slight crystallization at some period of its history. In the summer-time, for instance, when butter melts so

\* Hummell, *ibid.*, 22, p. 327; Crampton, *loc. cit.*, *supra*, p. 703.



easily at ordinary temperatures, these distinctions between pure and adulterated samples as shown by polarized light are by no means as satisfactory as in the winter.

Great care should be taken on this account, on the part of the collector of samples as well as the analyst, to keep the sample from melting under ordinary conditions before it is examined.

**Hess and Doolittle's Method of Examining the Curd.\***—A convenient portion of the sample of suspected butter is melted in a beaker, as much of the fat as possible is decanted off, and the remaining curd, washed free from fat with ether, is poured out on a glass plate, and dried. A sample of pure butter is treated in like manner by way of comparison. When examined under a very low magnification of from 3 to 6 diameters, the curd from the pure sample will be seen to be non-granular and amorphous in appearance, while, in the case of renovated butter, the curd will appear very coarse grained and mottled.

**Zega's Test for Oleomargarine.†**—A portion of the filtered-fat is poured into a test-tube and kept for two minutes in a boiling water-bath. 1 cc. of this fat is then measured with a hot pipette into a 50-cc. tube containing 20 cc. of a mixture of 6 parts ether, 4 parts alcohol, and 1 part glacial acetic acid. The tube is stoppered, shaken well, and cooled in water at 15° to 18° C. In the case of pure butter fat, the solution remains clear for some time, a slight deposit being apparent only after standing an hour or more. With oleomargarine, a deposit is evident in a very short time, and in ten minutes a heavy precipitate comes down. With 10% of oleomargarine in butter, a separation occurs in about fifteen minutes. When a few solid particles have separated out, they are withdrawn and examined under the microscope. With genuine butter, long narrow rods appear, sometimes pointed at the ends, often bent, and grouped as a rule centrally in star-shaped bundles. Oleomargarine presents an appearance of bundles of fine needles, closely packed to form masses frequently resembling sheaves and dumb bells in shape.

#### VEGETABLE OLEOMARGARINE (NUT BUTTER).

Cocoanut oil and palm kernel oil are especially adapted to the preparation of vegetable butter. Following are the constants of the fat of two vegetable butters "Sanella" and "Tomor." ‡

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\* Jour. Am. Chem. Soc., 22, 1900, p. 151.

† Chem. Ztg., 1899, 23, p. 312.

‡ Pharm. Zentrh., 48, p. 16; 49, p. 490.



	Refraction 25° C.	Reichert-Meissl No.	Polenske No.	Saponification No.
Sanella, Summer.....	38.5	7.9	15.6	247.0
Sanella, Winter.....	42.0	6.5	11.2	238.8
Tomor.....	.....	8.0	15.8	250.1

The above figures indicate that the chief constituent of both preparations is cocoanut oil; they also contain sesame oil. Both products were colored, salted, and churned with a liquid preparation of almonds and egg yolk.

Nut butters are coming into favor in the United States. Much of the cocoanut oil brought into the country or made from imported copra since the beginning of the great war is utilized in these products. The products are sold in bricks, packed in attractive cartons. Although notably different from animal oleomargarine they are labeled "oleomargarine" to comply with the federal law, in addition, however, the package usually bears a special name suggesting cocoanut oil.

Some of the brands contain benzoate of soda as a preservative, which is declared on the label; others are free from chemical preservatives. All are placed on the market uncolored, but at the time of sale a capsule containing a liquid color is given the purchaser who from directions on the label can color the product if he sees fit.

#### LARD.

**Nature and Composition.**—Lard is the fat of hogs, separated by heat from the scraps or containing tissues. The choicest or highest grade of lard is known as leaf lard, and is derived from the fat which surrounds the kidneys. A comparatively small part of the lard of commerce is, however, strictly speaking, pure leaf lard. Most of it is derived from the whole fat of the animal by rendering, by the aid of steam under pressure, either in open kettle or in closed tanks, the former being used more often for rendering lard on a small scale, and the latter being the most common commercial method.

Next to the leaf, the fat from the hog's back is considered the best in quality, after which is graded, in the order named, the fat from the head, the region of the heart, and the small intestines, the last two grades constituting what is commonly known as "trimmings."

Good lard is white and granular, having the consistency of salve. It has an agreeable, characteristic odor and taste.



The leaf or kidney fat furnishes also the source of the so-called neutral lard, already mentioned as an ingredient of oleomargarine. The leaf, being first chilled and finely ground, is placed in the kettle and rendered at a temperature of from 40° to 50° C., at which heat only a portion of the lard separates. This portion is, while melted, washed with water containing salt or dilute acid, and forms the neutral lard, a product almost entirely free from odor. The remainder of the leaf is then transferred to the closed tank and subjected for some hours to steam under pressure at a temperature of 230° to 290° F., the resulting lard being graded as pure leaf lard.

The composition of the mixed fatty acids of lard is thus calculated by Twitchell:

Linoleic acid.....	10.06%
Oleic acid.....	49.39%
Solid acids (by difference), stearic and palmitic....	48.55%

The solid triglycerides consist in part of  $\alpha$ -palmito distearin which has a different crystalline form from the  $\beta$ -palmito distearin of tallow, thus explaining the results obtained in the Belfield test.

Dennstedt and Voigtländer \* gives the following constants for American lards made from fat from different parts of the animal:

Fat from	Specific Gravity at 100° C. (Water at 15° C. = 1.)	Iodine Value.	Maumené Number at 40° C.	Melting-point, Bensemann's Method.		Refractive Index. Butyro-refractometer at 40° C.
				Temp. C. of Incipient Fusion.	Melted to a Clear Drop.	
Head.....	0.8637	66.2	33	24	44.8	52.6
	0.8629	66.6	32	24	44.8	52.5
	0.8631	65.0	34	24	45.0	52.0
Back.....	0.8611	61.5	37	28.5	48.5	52.4
	0.8621	65.0	35	28.5	48.5	51.8
	0.8616	65.1	38	31.5	46	51.9
Leaf.....	0.8637	62.2	..	26	45	51.4
	0.8615	59.0	..	29	44	50.2
	0.8700	63.0	30	28.5	44.5	52.0
Foot.....	0.8589	68.8	..	24	40	44.8
Ham.....	0.8641	68.4	38	26	45	51.9
	0.8615	66.6	..	26	44	51.9
	0.8628	68.3	..	26	44.5	53.0
Ham (German) . . .	0.8597	55.0	30	32	46	49.2

\* Lewkowitsch, Chem. Tech. and Anal. of Oils, Fats, and Waxes, 5th Ed., 2, 1914, p. 701.



**Effects of Feeding Hogs on Oil Cakes.**—Fulmer,\* Emmett and Grindley† and other investigators have found that feeding cottonseed meal to hogs causes the lard from these hogs to give a color with the Halphen test, but Tolman‡ Farnsteiner,§ and Polenske || have shown that the lard does not contain phytosterol when examined by Bömer's phytosterol acetate method.

Lard from hogs fed on sesame cake has been shown to respond to the Baudouin test, but not to the phytosterol acetate test.

**Lard from "Oily Hogs"** is quite different from the ordinary products. Richardson and Farey ¶ have analyzed one sample of back fat, three of leaf lard, and two of ham fat with the following average results:

	Melting-point.		Refract- ive Index, 40°.	Saponi- fication No.	Iodine No.	Free Fatty Acids as Oleic.	Insoluble Fatty Acids.		
	Open Capil- lary, Lower Limit.	Closed Capil- lary, Upper Limit.					Titer Test.	Liquid Acids.	Iodine No.
Back fat. . . . .	-1.5°	+12.0°	1.4620	189.0	93.9	0.16	21.2°	89.4	104.5
Leaf lard. . . . .	-0.5	+20.0	1.4620	191.1	93.9	0.19	22.9	87.8	107.9
Ham fat. . . . .	-1.2	+15.5	1.4630	189.7	94.0	0.18	20.6	86.5	109.0

**U. S. Standards.**—*Standard Lard and Standard Leaf Lard* are lard and leaf lard respectively, free from rancidity, containing not more than 1% of substances other than fatty acids, not fat, necessarily incorporated therewith in the process of rendering, and standard leaf lard has an iodine number not greater than 60.

**U. S. P. Standards.**—Melting-point, 36-42° C.; saponification value, 195-203; iodine value, 46-70; 10 grams of sample dissolved in 30 mls of chloroform mixed with 10 mls of neutral alcohol and 1 drop of phenolphthalein solution require not more than 2 mls N/10 alkali for neutralization. Qualitative and microscopic tests are also given.

**Lard Oil.**—This oil is obtained by subjecting lard contained in woolen bags to hydraulic pressure in the cold. The lard oil (chiefly olein) thus expressed constitutes nearly 60% of the whole, and the residue is known as lard stearin.

\* Jour. Amer. Chem. Soc., 26, 1904, p. 837.

† Ibid., 27, 1905, p. 263.

‡ Ibid., 27, 1905, p. 589.

§ Zeits. Unters. Nahr. Genussm., 11, 1906, p. 1.

|| Arb. kaisl. Gesundheitsamt, 22, 105, p. 568.

¶ Jour. Amer. Chem. Soc., 30, 1908, p. 1191.



Lard oil is a thin fluid, pale yellow in color, and with varying specific gravity, due to varying conditions of pressure and temperature. It has a pleasant, though somewhat bland, taste, and is used to some extent as an edible oil. It is used in France as an adulterant of olive oil, and with the Maumené, elaïdin, and nitric acid tests, it behaves much like olive oil.

Adulterants of lard oil are cottonseed and corn oils.

**Compound Lard.**—The article sold under this name is a mixture consisting usually of beef or lard stearin and cottonseed oil. Sometimes no lard whatever is present. Lard stearin is the residue left in the cloths after the lard oil has been removed by pressure (page 579). Beef stearin is, similarly, the residue from which oleo oil has been expressed (page 564). The cottonseed oil used is highly refined, and finally decolorized by mixing with fuller's earth and filtering.

**Lard Substitutes** differ from compound lard in that they contain no hog fat and are sold under distinct names in competition with lard. They consist of various mixtures of cottonseed oil with stearin (usually beef) or of hydrogenated cottonseed oil. Wesson states that cocoanut oil, although used in large amount in vegetable butter in the United States, is not suitable for lard substitutes, as it causes the mixture to froth on heating. When hydrogenated oils are used traces of nickel may be present, although now less often than formerly.

Examples of lard substitutes are Fairbanks' "Cottolene," Southern Cotton Oil Company's "Snowdrift" and "Scoco," Proctor & Gamble's "Crisco," Armour's "Vegetole," Swift's "Jewel" and "Crescent," and Wilson's "Advance."

	Iodine No.	Melting-point.	Titer Test.	Free Fatty Acids.	Color.		Flavor.
					Yellow.	Red.	
I	98.2	42.9	35.7	0.13	32	3.2	Good
II	97.4	50.2	36.2	0.14	35	4.8	Poor
III	95.1	50.8	37.2	0.19	32	3.2	Poor
IV	97.3	47.5	35.9	0.10	26	2.8	Poor
V	95.6	41.6	36.9	0.11	17	1.7	Good
VI	95.1	48.8	37.3	0.14	35	6.2	Poor
VII	98.0	46.8	35.9	0.13	27	2.7	Good
VIII	89.0	50.4	.....	0.14	35	4.9	Fair
IX	96.1	42.2	36.4	0.11	35	3.6	Good
X	93.7	43.0	36.7	0.12	20	2.0	Fair
XI	85.2	53.1	35.6	0.18	35	4.3	Poor
XII	78.1	33.7	34.4	0.09	22	2.2	Good



The table on p. 580, kindly furnished by Dr. David Wesson, gives the results of examination of the leading brands of compound lard and lard substitutes on the American market. The color values are of the melted fat in terms of yellow and red as read in  $5\frac{1}{4}$ -inch cells of the Lovibond tintometer.

**Adulteration of Lard.**—"Compound lard," although properly branded by the manufacturer, may be sold for pure lard by the retailer. This substitution and the addition of beef tallow to lard are the common forms of adulteration practiced in the United States. Other oils that may be looked for are cocoanut, palm kernel, corn peanut, and sesame. Formerly water was incorporated with the fat to such an extent as to materially cheapen it, but this sophistication is now rare.

**Analyses of Pure and Compound Lard.**—Leach gives analyses together with conclusions drawn therefrom as follows:

ANALYSES OF SAMPLES ILLUSTRATING TYPES OF LARD, LARD SUBSTITUTES, AND MIXTURES.

	Nitric Acid Test.	Crystallization.	Bechi Reaction.	Butyro-refractometer.			Iodine Number.	Conclusion.
				Temperature, Degrees.	Reading.	Variation from Standard.		
A	Slight color.....	Lard stearin	None	42.5	49.7	+0.1	58.1	Lard
B	Red .....	" "	"	42	50	+0.2	59.9	"
C	Slight color .....	" "	"	41.5	50.1	+0.0	58.7	"
D	" " .....	" "	"	43	50	+0.6	63.7	"
E	" " .....	" "	"	41.3	51	+0.8	64.6	"
F	" " .....	" "	"	42	50.5	+0.7	64.8	"
G	" " .....	" "	"	42	49.7	-0.1	56.4	Leaf lard
H	Very slight color	Beef stearin	"	50	41.2	-3.8	37.3	Beef tallow
I	Deep-brown red	Few small bunches	Deep color	42	58.7	+8.9	108	Cottonseed oil
J	Red .....	Lard stearin	" "	43	50.5	+1.3	69.5	Lard and cottonseed oil
K	Very slight color	Lard and beef stearin	None	43	48.5	-0.7	55.2	Lard and beef tallow
L	Deep brown....	Lard stearin	Deep color	43.5	51	+1.1	71.4	Lard and cottonseed oil
M	Red.....	"	" "	43.7	50.1	+1.3	66.7	Ditto
N	" .....	Lard and beef stearin	" "	43.5	49.1	+0.3	54.7	Lard, beef tallow, and cottonseed oil

*Notes on the Above Table.*—It will in general be noted that adulteration of lard with cottonseed oil alone is indicated by an abnormally high refractometer number, while the presence of tallow will result in an



abnormally low refraction. But both adulterants may be present and a normal refraction result. In such a case the positive detection of one of them, such as the cottonseed oil by the Bechi or Halphen test, will indirectly show the presence of the other (tallow), and this indirect proof will be confirmed by crystallization.

Samples A, B and C give reactions corresponding to normal, pure lard. D, E, and F show somewhat high refractometer and iodine numbers, but give no direct reaction for cottonseed oil by the Bechi test. G, although showing low iodine and refractometer numbers, gives no evidence of the presence of tallow by crystallization. In fact, the crystals from this sample proved under all circumstances to be most clearly typical of pure lard, broad and flat plates with obliquely cut ends.

This sample was, in fact, pure leaf lard. It is generally true that a stiff, strictly pure leaf lard, which both by its consistency and by its low iodine and refractometer numbers might suggest the presence of beef fat, shows on crystallization much more definitely characteristic lard stearin than does a whole-hog lard, whose iodine and refractometer numbers are more nearly the normal standard.

In distinction from such leaf lard, a sample which may have a similar consistency and iodine and refractometer numbers, but which is composed of a whole-hog lard of a comparatively high iodine number, together with beef fat, gives unmistakable proof of its adulteration by its crystallization.

#### METHODS OF ANALYSIS.

**Detection of Beef Stearin or Tallow.**—*Belfield-Gladding Microscopic Method.*\*—Dissolve 2 to 2.5 grams of the melted sample with the aid of heat in 7.5 cc. of ether-absolute alcohol (1 : 2), cool in ice water until a copious precipitate forms, filter, and wash once or twice with the ether-alcohol mixture. Dry at room temperature, transfer to a test-tube, and dissolve in 15 cc. of ether. Loosely stopper the test-tube with cotton, place in a vessel containing sufficient water to insure a uniform temperature, and allow to stand for some hours until abundant crystals are formed. Examine under the microscope in ether as a medium or if desired in alcohol or oil.

If the crystals are, however, in a pulverulent condition, a drop of alcohol can be used as a mountant, or oil, as preferred. Mounted under a cover-glass they are examined under various powers of the microscope.

\* Jour. Amer. Chem. Soc., 18, 1896, p. 189.



Figs. 272 and 273, Pl. XXXIX, show the typical appearance of pure lard stearin from a leaf lard of known purity, and Figs. 276, 277, and 278, Pl. XL, illustrate beef stearin. These figures show distinctive crystallization of each form under the best conditions. The lard stearin crystals when thus obtained are flat rhomboidal plates cut off obliquely at one end, and are grouped irregularly, as if thrown carelessly together. The beef stearin crystals, on the other hand, are cylindrical rods or needles, often curved, with sharp ends, and are arranged as shown in fan-shaped clusters. Conditions of crystallization are frequently such as not to show the sharp distinctions noted above. Both forms of crystals are at times apt to gather in clusters that at first sight appear somewhat similar, and are often misleading as to their true character. It is found almost invariably that the beef stearin crystals gather in clusters, radiating from a common center or point, often with a peculiar twisted appearance, breaking up into little fans. Lard crystals, it is true, do not always lie flat in irregular groups as shown in Fig. 272, but, as in Fig. 274, form clusters that, unless studied carefully, might at first sight be considered as identical with the fan shapes of the beef stearin already described. It will be seen, however, that if the best possible conditions are attained, the crystals of lard, instead of radiating from a point, are arranged more like feathers or alternate leaves on a branch, each crystal being given forth from another close at hand. Moreover, the lard crystals are themselves straight and not curved, the apparent curve in the appearance of the clusters being, on careful examination, especially under high power, seen to be chiefly due to several of these straight crystals arranged at angles to each other.

Even when the highest powers of the microscope are applied to the beef stearin crystals, they will always appear as cylindrical, sharp-pointed rods, some straight, others curved; while with the lard crystals they should be capable of showing the thin, flat, oblique-ended structure when examined with higher powers, even when they are arranged in the feathery clusters, the apparently pointed ends of some of the crystals being due to the fact that the plates are viewed edgewise. This is apparent in Fig. 275, in which the crystals are magnified to 480 diameters.

*Leys-Emery Melting-point Method.\**—Place 5 grams of the warm filtered fat in a glass-stoppered 25-cc. cylinder, 150 to 175 mm. high and 18

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\* Leys, *jour. pharm. chim.*, [6], 26, 1907, p. 289; Emery, U. S. Dept. Agric. Bur. Anim. Ind. Circ., 132.



mm. inside diameter, add 25 cc. of warm ether, stopper, shake vigorously until the fat is dissolved, and allow to stand 18 hours at 20 to 25° C. Decant off the liquid, wash with two portions of 5 cc. of ether by decantation, and use a third portion for transferring to the filter. Wash with cold ether, using a total of 10 to 15 cc. with the aid of slight suction at the end. Spread out the crystals on filter-paper and when dry mix well and determine the melting-point in a capillary tube 1 mm. in diameter, filling so as to form a column 9 mm. high and compacting firmly by tapping. The point when the substance appears perfectly clear and transparent is taken as the true melting-point.

Thirty samples of leaf lard gave melting-points from 63.6 to 64.1. Samples below 63.4 are regarded as suspicious and below 63 as adulterated.

**Detection of Cottonseed Oil.**—This is best accomplished by the Halphen test and the determination of the refraction, specific gravity, and the iodine number. See tables pages 528 and 529.

**Detection of Coconut and Palm Kernel Oils.**—The Reichert-Meissl and Polenske numbers furnish the best evidence. The iodine and saponification numbers also are useful in diagnosis. See table page 528, also figures given after descriptions of the methods.

**Detection of Corn Oil.**—In the absence of cottonseed oil and sesame oils, as shown by qualitative tests, corn oil is indicated by an abnormally high refraction and iodine number, the maximum for cottonseed oil being usually lower than the minimum for corn oil. In case these constants are not decisive apply the Bömer phytosterol acetate test. According to McPherson and Ruth,\* the crystals thus obtained from lard melt at 113°, those from corn oil at 127 to 128°.

**Detection of Sesame Oil.**—The Baudouin and Villavecchia-Fabris tests are decisive for appreciable amounts. The iodine number is also useful.

**Detection of Peanut Oil.**—The test for arachidic and lignoceric acids should be applied.

**Detection of Nickel in Hydrogenated Lard Substitutes.**—*Prall-Kerr Method.*†—Warm 10 grams of the fat with 10 cc. of hydrochloric acid (sp. gr. 1.12) in a test-tube on a water-bath for 2 or 3 hours with repeated shaking and filter on a wet paper into a porcelain dish. Evaporate off

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\* Ohio Dairy and Food Com. Ann. Rep. 1906.

† Zeits. Unters. Nahr. Genussm., 24, 1912, p. 109; Jour. Ind. Eng. Chem., 6, 1914, p. 207.



most of the acid on a water-bath, add 2 to 3 cc. of concentrated nitric acid and continue the evaporation to dryness to destroy organic matter. Dissolve the residue in a few cc. of water and add a few drops of each 1% solution of dimethylglyoxime in alcohol, and dilute ammonia water. If nickel is present a red color appears which for quantitative estimation may be compared with the color developed in a standard solution of a nickel salt.

Iron and copper are said to interfere although the color of ferric hydroxide is not the same as that formed in the presence of nickel.

**Detection of Paraffin.**—See page 527.



## CHAPTER XIV.

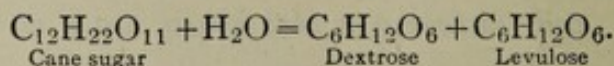
### SUGAR AND SACCHARINE PRODUCTS.

**Nature and Classification of Sugars.**—Of all classes of food materials the sugars from their great solubility are the most readily available, and on this account are very valuable as nutrients. As in the natural processes of digestion the starches and more difficultly digestible of the carbohydrates are converted into sugar and thus rendered assimilable, so by processes quite analogous to those that take place in the alimentary tract, the chemist converts these same carbohydrates into sugar as an end-point for purposes of definite determination.

The sugars are characterized by their sweet taste, their ready solubility in water, their power to rotate the plane of polarized light, and their insolubility in ether and absolute alcohol.

The sugars occurring commonly in food naturally divide themselves into two groups: First, the *Hexoses* ( $C_6H_{12}O_6$ ) including dextrose, levulose, and galactose; second, the *Disaccharides* ( $C_{12}H_{22}O_{11}$ ) especially sucrose, maltose, and lactose. Other sugars occurring less frequently or in smaller amount as well as a classification of all the food carbohydrates are given on pages 35-38.

The members of both groups are intimately related. Thus by the ordinary process of so-called inversion sucrose, or cane sugar, belonging to the disaccharides, is converted by the action of heat and dilute acid into two sugars, dextrose and levulose, belonging to the hexoses, in accordance with the following reaction:



The same equation expresses also the result that takes place when lactose, or milk sugar, is heated with dilute acids, breaking up into dextrose and galactose.



**Occurrence.**—Sugars occur in roots, grasses, stems of plants, trunks of trees, leaves, and fruits, usually in the form of cane sugar, or sucrose, and of invert sugar (dextrose and levulose) mixed in varying proportions.

The following table from Buignet \* shows the kind and amount of sugars occurring in some of the common fruits:

	Cane Sugar.	Reducing Sugar.	Acid.
Apricots .....	6.04	2.74	1.864
Pineapples .....	11.33	1.98	.547
English cherries.....	.00	10.00	.661
Lemons.....	.41	1.06	4.706
Figs .....	.00	11.55	.057
Strawberries. ....	6.33	4.98	.550
Raspberries .....	2.01	5.22	1.380
Gooseberries .....	.00	6.40	1.574
Oranges .....	4.22	4.36	.448
Peaches (green).....	.92	1.07	3.900
Pears (Madeleine).....	.36	8.42	.115
Apples.....	5.28	8.72	1.148
" .....	2.19	5.45	.633
Prunes.....	5.24	2.43	1.288
Grapes (hothouse) .....	.00	17.26	.345
" green .....	.00	1.60	2.485

#### CANE SUGAR, OR SUCROSE.

**Nature and Occurrence.**—This, the most common of all the sugars, is nearly always understood by the unqualified term of sugar. It crystallizes in monoclinic prisms. Its specific gravity is 1.595. Its melting-point is about 160° C. Its specific rotary power  $[\alpha]_D$  in solutions having a concentration of from 10 to 20 grams in 100 cc. is, according to Tollens, 66.48°. Sucrose is extremely soluble in water, which, when cold, will hold in solution twice its weight of the sugar.

Cane sugar is ordinarily derived from four sources—the sugar beet, the sugar cane, the maple tree, and the sorghum plant. The first two sources supply the principal output of commercial cane sugar, about half the sugar on the world's market being furnished, by the sugar beet and the other half by the sugar cane. It should be understood that the product sucrose, or cane sugar, is chemically the same whether derived from either of the above sources and thoroughly refined.

**U. S. Standard Sugar** is white sugar containing at least 99.5% of sucrose.

\* Ann. Chim. Phys., 59, 233.



**The Sugar Cane** (*Saccharum officinarum*) is cultivated principally in Louisiana and other southern states, in Cuba and the West Indies, and in the Hawaiian Islands. Its growth and cultivation form an industry in nearly all tropical countries.

Allen\* has compiled the following table showing the composition of the juice of the sugar cane from different localities:

Locality and Kind of Cane.	Water.	Sugar.	Woody Fiber.	Salts.	Authority.
Martinique. ....	72.1	18.0	9.9	.....	Peligot
Guadaloupe. ....	72.0	17.8	9.8	0.4	Dupuy
Havana. ....	77.0	12.0	11.0	....	Casaseca
Cuba. ....	65.9	17.7	16.4	....	Casaseca
Mauritius. ....	69.0	20.0	10.0	1.0	Icery
Ribbon cane. ....	76.73	13.39	9.07	.39	Avequin
Tahiti. ....	76.08	14.28	8.87	.35	Avequin

The composition of raw cane sugar ash according to Monier is as follows:

#### RAW CANE SUGAR ASH.

Carbonate of calcium. ....	49.00
“ “ potassium. ....	16.50
Sodium and potassium sulphate. ....	16.00
Sodium chloride. ....	9.00
Silica and alumina. ....	9.50
	<hr/>
	100.00

**Manufacture of Cane Sugar.**—The process of manufacturing raw sugar from sugar cane is briefly as follows: The juice is first extracted from the canes by crushing in roll mills and is freed from nitrogenous bodies, organic acids, etc., by the process of *defecation*, which consists in heating to coagulate the albumin, and nearly neutralizing with milk of lime, the impurities being removed as a scum. The juice is then subjected to evaporation and crystallization, the raw, or muscovado sugar, which contains from 87 to 91 per cent of sucrose, being separated from the molasses, which is the mother liquor, by draining or by centrifugal.

Some of the best grade of muscovado, or raw sugar, is used as “brown sugar” without further refining, and much of the molasses is used as a table syrup and for cooking, while the lower grades of molasses are used in the manufacture of rum.

\* Com. Org. Anal., 4 Ed., Vol. I, p. 359.



The following table from Thorpe\* shows the average composition of raw and refined sugar:

	Cane Sugar.	Glucose <sup>1</sup> .	Water.	Organic Matter.	Ash.
RAW SUGAR.					
Good centrifugal .....	96.5	0.75	1.50	0.85	0.40
Poor centrifugal .....	92.0	2.50	3.00	1.75	0.75
Good muscovado .....	91.0	2.25	5.00	1.10	0.65
Poor muscovado .....	82.0	7.00	6.00	3.50	1.50
Molasses sugar .....	85.0	3.00	5.00	5.00	2.00
Jaggery sugar .....	75.0	11.00	8.00	4.00	2.00
Manilla sugar .....	87.0	5.50	4.00	2.25	1.25
Beet sugar, 1st .....	95.0	0.00	2.00	1.75	1.25
Beet sugar, 2d .....	91.0	0.25	3.00	3.25	2.50
REFINED SUGAR.					
Granulated sugar .....	99.8	0.20	0.00	0.00	0.00
White coffee sugar .....	91.0	2.40	5.50	0.80	0.30
Yellow X C sugar .....	87.0	4.50	6.00	1.50	1.00
Yellow sugar .....	82.0	7.50	6.00	2.50	2.00
Barrel sugar .....	40.0	25.00	20.00	10.00	5.00

<sup>1</sup> The term "glucose" includes sugars which reduce Fehling's solution, but are not necessarily optically active.

The following minimum and maximum figures are taken from analyses made by Babington† of twenty-two samples of brown sugar and thirty-one samples of molasses.

## BROWN SUGAR.

Direct polarization. ....	84	to	87
Invert " .....	-27	"	-29
Sucrose by Clerget. ....	83.5	"	91.5
Reducing sugar .....	3	"	6
Moisture .....	3.5	"	6
Ash. ....	0.8	"	3.0

## MOLASSES.

Direct polarization. ....	30	to	50
Invert " .....	-10	"	-21
Sucrose by Clerget. ....	32	"	52
Reducing sugar .....	13	"	24
Moisture .....	29	"	32
Ash. ....	0.5	"	4

\* Outlines of Industrial Chem., p. 383.

† Can. Int. Rev. Dept. Bul. 25.



**The Sugar Beet** (*Beta vulgaris*) is grown chiefly in France and Germany, and to a lesser extent in Holland and England. The successful growth of the sugar beet in the United States is confined mainly to California, Colorado, Utah, and Nebraska, and the entire output of beet sugar in this country is comparatively small.

According to R. Hoffmann, sugar beets have about the following composition, three types being selected—first, those poor in sugar; second, those having a medium sugar content, and third, those rich in sugar:

COMPOSITION OF THE SUGAR BEET.

	First Type.	Second Type.	Third Type.
Water .....	89.20	83.20	75.20
Sugar.....	4.00	9.42	15.00
Nitrogenous compounds.....	1.00	1.64	2.20
Non-nitrogenous compounds:			
Soluble .....	4.13	3.34	4.23
Insoluble (cellulose) .....	1.01	1.50	2.07
Ash .....	0.66	0.90	1.30
	100.00	100.00	100.00

The following is the mean composition of ten samples of California sugar beet: \*

Per cent juice extracted.....	61.38
Specific gravity.....	1.062 to 1.075
Per cent of reducing sugar.....	0.91
Per cent of sucrose.....	14.38
Total solids calculated.....	16.58
Total solids weighed.....	17.20
Per cent of ash.....	0.994

The composition of beet sugar ash according to Monier is as follows:

## RAW BEET SUGAR ASH.

Carbonates of potassium and sodium.....	82.20
Carbonate of calcium.....	6.70
Potassium and sodium sulphate and sodium chloride. ....	11.10
	100.00

**Manufacture of Beet Sugar.**—In making raw sugar from sugar beets the latter are first washed and sliced by machinery and the juice extracted

\* U. S. Dept. of Agric., Div. of Chem., Bul. 27, p. 202.



by *diffusion* or digestion with warm water. The juice is then clarified or defecated in much the same manner as that from the sugar cane, after which it is usually bleached with sulphur dioxide.

The subsequent evaporation and crystallization are carried out usually in vacuum pans, and the sugar separated out by centrifugals.

Beet sugar molasses is unfit for food, due to the presence of nitrogenous bodies, which give it a very unpleasant taste and smell.

**Process of Refining.**—In refining raw sugar, a syrup is made, which is subjected to centrifuging and further defecation, using lime, clay, liquid blood, calcium acid phosphate, and other substances as clarifiers. The syrup is then filtered, first through cloth bags and then through bone char, after which it is evaporated and allowed to crystallize, the resulting granulated sugar being separated, as in the case of raw sugar, by centrifugal machines.

*Granulated Sugar* of commerce is without doubt the purest food product on the market, being generally 99.8% sucrose. It is usually treated with an extremely weak solution of ultramarine to counteract the natural yellow color.

The syrup from which the granulated sugar is separated forms the "golden," or "drip," syrup used on the table. Its typical composition is as follows: Sucrose, 40%; reducing sugars, 25%; water, 20%; organic matter, 10%; ash, 5%.

The dry sugars, whether white or brown, are rarely subjected to adulteration.

**Maple Sap.**—The sap of the maple tree, *Acer saccharinum*, or *Acer barbatum*, furnishes a sugar considerably prized for its peculiar flavor. The maple sugar industry is largely confined to the northeastern states and to Canada, and the maple sugar season is generally limited to six weeks or two months in the spring.

The following are minimum and maximum figures from the analyses of five samples of maple sap made in Massachusetts:

Specific gravity.....	1.007 to 1.015
Sucrose.....	0.769 " 2.777
Reducing sugar.....	" 0.012

The ash of maple sap varies from 0.04 to 0.1 per cent. Albuminoids are present in amount varying from 0.008 to 0.03 per cent.

**Maple Sugar and Syrup** are made by simply boiling down the sap to the proper consistency, usually in open pans, and removing the scum



with great care, since this contains nitrogenous matters that would cause fermentation in the finished product. Pure cane sugar is never commercially produced from the maple sap, since the refining process would deprive it of the flavor which gives to maple sugar the chief value.

McGill gives the following as the average analyses of six samples of maple syrup of known purity:

Saccharimeter Direct.	Saccharimeter Invert.	Cane Sugar by Clerget.	By Copper.		Ash.	Water.	Solids.
			Reducing Sugar.	Cane Sugar.			
+ 62.2	- 21.2	62.4	.42	63.36	.53	35.70	64.30

The variation in the composition of pure maple products is shown by the following table compiled by A. H. Bryan \* from analyses published by Hortvet,† Jones,‡ and Winton§, and some sixty analyses made at the sugar laboratory of the Bureau of Chemistry, U. S. Department of Agriculture.

	Maple Sugar.			Maple Syrup.		
	Minimum.	Maximum.	Average.	Minimum.	Maximum.	Average.
Water . . . . . per cent	3.05	11.0	.....	Not more than	32.00	.....
Direct polarization . . . . . "	72.6	87.4	.....	51.0	62.2	.....
Invert sugar . . . . . "	1.16	8.37	.....	0.34	9.17	.....
Lead number . . . . . "	1.83	2.48	2.23	1.19	2.03	1.49
Total ash . . . . . per cent	0.64	1.32	0.91	0.46	1.01	0.60
Soluble ash . . . . . "	0.33	0.67	0.46	0.21	0.63	0.38
Insoluble ash . . . . . "	0.20	0.87	0.46	0.14	0.56	0.23
Alkalinity of soluble ash . . . . . "	0.40	0.95	0.63	0.26	0.68	0.50
Alkalinity of insoluble ash . . . . . "	0.55	1.72	0.94	0.31	0.94	0.54
Ratio of insoluble to soluble ash . . . . . "	0.50	2.20	1.00	0.60	3.20	1.70
Iodine reaction . . . . . "	.....	.....	none	.....	.....	none
Polarization at 87° after inversion . . . . . °V.	- 2.0	+ 2.0	.....	- 2.0	+ 2.0	.....
Malic acid value . . . . . "	0.65	1.67	1.01	0.41	1.76	0.78

In the table which follows appear the maximum, minimum, and average results obtained in the four most extensive investigations of genuine maple

\* U. S. Dept. Agric., Bur. of Chem., Circular No. 40, p. 10.

† Jour. Am. Chem. Soc., 26, 1904, p. 1523.

‡ Vt. Agric. Exp. Sta. Rep., 1904, p. 446; 1905, p. 315.

§ Jour. Am. Chem. Soc., 28, 1906, p. 1204.



syrup which have yet been undertaken \* as compiled by Snell and Scott. The electrical conductivity and volumetric lead values are not included. (See pages 659 and 661.)

SUMMARY OF ANALYSES OF MAPLE SYRUP BY DIFFERENT ANALYSTS  
CALCULATED TO THE DRY SUBSTANCE.

	No. of Sam- ples.	Ash.			Alkalinity of Ash.		Lead No.			Malic Acid Value.
		Total.	Sol- uble.	Insol- uble.	Sol- uble.	Insol- uble.	Can- adian.	Win- ton (25 g. Syrup)	Win- ton (25 g. Dry Mat- ter).	
Maximum:										
Bryan.....	481	1.68	1.23	1.01	122	208	.....	4.41	.....	1.60
Jones.....	48	1.32	0.72	0.78	102	145	.....	.....	.....	1.11
McGill.....	456	1.38*	0.79*	0.75*	.....	.....	6.56	2.38†	.....	1.16‡
Snell and Scott.	126	1.58	0.77	0.92	103	201	7.50	.....	4.09	1.46
Minimum:										
Bryan.....	481	0.68	0.35	0.23	41	41	.....	1.76	.....	0.29
Jones.....	48	0.77	0.45	0.25	46	55	.....	.....	.....	0.65
McGill.....	456	0.69*	0.33*	0.12*	.....	.....	1.37	1.05†	.....	0.30‡
Snell and Scott.	126	0.61	0.30	0.16	51	48	1.74	.....	1.41	0.38
Average:										
Bryan.....	481	1.00	0.63	0.37	75	97	.....	2.70	.....	0.84
Jones.....	48	0.92	0.58	0.34	79	83	.....	.....	.....	0.82
McGill.....	456	0.89*	0.56*	0.33*	.....	.....	2.83	1.75†	.....	0.77‡
Snell and Scott.	126	0.88	0.48	0.40	68	116	3.48	.....	2.30	0.75

\* 115 samples. † 47 samples. ‡ 452 samples.

A summary of 363 analyses of authenticated samples of maple sugar by A. H. Bryan† with the collaboration of Straughn, Church, Given, and Sherwood appears in the table which follows. The analyses were made on syrup prepared as described on page 656, but the results are calculated to the dry basis.

\* Bryan, U. S. Dept. of Agric. Bur. of Chem. Bul. 134, 1910; Jones, Vt. Agr. Exp. Sta. Rep., 1904-5, p. 315; McGill, Lab. Int. Rev. Dept. Ottawa Bul. 228, 1911; Snell and Scott, Jour. Ind. Eng. Chem., 6, 1914, p. 216.

† U. S. Dept. of Agric. Bul. 466, 1917.



	Number of Analyses.	Sucrose.	Invert Sugar.	Total Ash.	Soluble Ash.	Insoluble Ash.	Undetermined	Winton Lead No.*	Malic Acid Value.
United States:	283								
Maximum..		98.62	37.30	1.66	1.14	0.81	5.84	4.95	1.72
Minimum..		57.04	0.09	0.76	0.37	0.21	0.00	1.85	0.51
Average....		91.89	5.46	0.95	0.62	0.33	1.70	2.68	0.91
Canada:	80								
Maximum..		96.59	35.26	1.70	0.89	1.00	8.18	4.14	1.51
Minimum..		58.92	0.88	0.76	0.31	0.24	0.02	1.86	0.62
Average....		86.46	8.76	1.06	0.61	0.45	3.70	3.04	1.03

\* Determinations on 308 samples by Ross modification: Max. 5.90, min. 2.20, av. 3.50.

Partial ash analyses of maple products and brown sugar have been made by Jones \* with the following maxima and minima results:

		Number of Analysis.	100 Parts of Ash Contain			Ratio of		
			CaO.	K <sub>2</sub> O.	SO <sub>3</sub> .	CaO to K <sub>2</sub> O × 100.	CaO to SO <sub>3</sub> × 100.	K <sub>2</sub> O to SO <sub>3</sub> × 100.
Maple syrup:	Min. . . .	6	18.03	30.00	0.68	150	3.4	1.9
	Max. . .		23.98	38.98	2.30	181	12.7	7.2
Maple sugar:	Min. . . .	4	21.03	18.26	1.51	57	5.2	5.1
	Max. . . .		31.74	32.95	2.42	153	10.4	9.4
Brown sugar:	Min. . . .	4*	4.17	30.72	4.58	257	27	11
	Max. . . .		21.62	55.40	17.78	949	157	58

\* Including one analysis by Hortvet.

**U. S. Standards for Maple Products.**—*Maple Sugar* is the solid product resulting from the evaporation of maple sap, and contains in the water-free substance not less than 0.65% of maple sugar ash.

*Maple syrup* is syrup made by the evaporation of maple sap or by the solution of maple concrete, and contains not more than 32% of water and not less than 0.45% of maple syrup ash.

**Adulteration of Maple Sugar and Syrup.**—The chief adulterants of maple sugar are brown, or molasses sugar, and white, or refined sugar, the latter being often used in mixture with burnt or inferior maple stock, which\* itself would be abnormally dark in color and of a rank taste. Maple syrup is commonly adulterated with a syrup made from refined cane sugar, less often with golden or drip syrup, or molasses. Glucose, which formerly was a common adulterant, is now seldom employed.

\* Loc. cit., 1905, p. 331.



*Refined Sugar* or refined sugar syrup added to maple products, while not greatly affecting the polarization, diminishes the percentage of total ash and the lead number, as well as the malic acid value and ash constants.

According to analyses by Jones and Hortvet, brown sugar of various grades contains from 0.59 to 4.33% of total ash, some of the grades with low ash content, or syrups made from them, not being distinguishable from maple sugar or maple syrup respectively by this determination alone; the ratio of insoluble to soluble ash, however, is commonly higher in brown sugar than in maple products. It is frequently possible to identify brown, or molasses sugar, especially when it forms the larger portion of the alleged maple sugar or syrup, by the physical sense of taste. When the perfectly characteristic taste of brown, or molasses sugar, or of "drip syrup," so far predominates over the maple flavor as to be unmistakable, especially in cases where the maple flavor is entirely lacking, one need have little hesitation in condemning the product.

*Glucose* in maple products is detected by polarization both before and after inversion. A reading of the inverted solution much in excess of 3° Ventzke at 87° C. furnishes evidence of the presence of this adulterant.

**Sorghum** (*Andropogon sorghum*, variety *saccharatus*) has for many years been grown quite extensively in the southern and western states, and used as a source of syrup which is highly prized because of its distinctive flavor.

Much experimental work was carried out by Collier\* in the early eighties and prior thereto with the belief that the sorghum plant would become an important source of commercial crystallized sugar, but experiments were at length abandoned.

The composition of the juice of the sorghum plant is shown by the following results of analyses of eleven varieties made by Hardin.†

Total solids.....	15.97	to 18.71
Specific gravity.....	1.0656	to 1.0775
Solids not sugar.....	5.02	to 10.63
Cane sugar.....	2.81	to 8.01
Reducing sugars.....	3.87	to 7.55

Some varieties of sorghum juice have been known to contain 15 or even 17% of sucrose.

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\* See numerous government reports.

† U. S. Dept. Agric. Div. Chem. Bul. 37, p. 75.



In making syrup from sorghum, the ripe canes are crushed, the juice is heated with milk of lime, and the scum removed. The juice is then concentrated usually in open pans to the required consistency.

The following analysis of sorghum syrup is by Jordan and Chesley.\*

Total solids.....	74.63
Sucrose.....	40.00
Reducing sugars.....	28.42
Gums and extractives.....	4.03
Ash.....	2.82
Acidity as tartaric.....	0.79

#### GRAPE SUGAR, OR DEXTROSE.

Dextrose ( $C_6H_{12}O_6 + H_2O$ ), designated *d*-glucose by Fisher and known in its commercial form as starch sugar, occurs in honey with levulose, and in fruits with both levulose and cane sugar. It is produced by the action of dilute acids or of certain ferments on starch, dextrin, or cane sugar. Grapes contain about 15% of dextrose. Anhydrous dextrose is soluble in 1.2 parts of cold water. It is soluble in alcohol, but less so than cane sugar. It is much less sweet than cane sugar.

The specific rotary power of dextrose is

$$[\alpha]_D = 52.3, \quad [\alpha]_f = 58.$$

A normal solution of dextrose on the Soleil-Ventzke scale polarizes at  $78.6^\circ$ . For the commercial preparation of dextrose see p. 598.

**U. S. Standards for Various Sugars.**—*Standard 70 sugar*, or *brewers' sugar*, is hydrous starch sugar containing not less than 70% of dextrose, and not more than 0.8% of ash.

*Standard 80 sugar, climax*, or *acme sugar*, is hydrous starch sugar containing not less than 80% of dextrose, and not more than 1.5% of ash.

*Standard anhydrous starch sugar* is anhydrous starch sugar containing not less than 95% of dextrose without water of crystallization, and not more than 0.8% of ash.

The ash of these standard products consists almost entirely of chlorides and sulphates of lime and soda.

#### LEVULOSE.

Levulose, also known as *d*-fructose and *l*- $\delta$ -fructose, occurs in foods as the product of inversion of cane sugar. It is prepared by the action

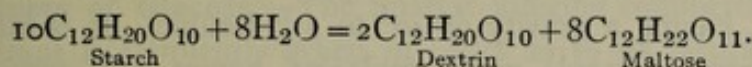
\* Jour. Ind. Eng. Chem., 9, 1917, p. 256.



of dilute acids on inulin. Normally it is in the form of a syrup, but with extreme care pure anhydrous levulose can be obtained. Diabetene is a commercial form of dry levulose. Levulose is formed with dextrose in the inversion of cane sugar (page 586), and with dextrose occurs in honey and in many fruits. The specific rotary power of levulose varies with the temperature. At  $15^{\circ}$  C.  $[\alpha]_D = -98.8^{\circ}$ , decreasing by  $0.6385^{\circ}$  for each degree increase in temperature. Its left-handed reading on the Ventzke sugar scale at  $15^{\circ}$  C. is equivalent to  $148.6^{\circ}$ . Levulose is sweeter than dextrose. Its reducing power on Fehling's solution is assumed to be the same as that of dextrose.

#### MALT SUGAR, OR MALTOSE.

Maltose ( $C_{12}H_{22}O_{11} + H_2O$ ) is of little importance from the standpoint of the food analyst, excepting as an ingredient of commercial glucose, and as being the sugar produced by the action of ptyaline, the ferment of the saliva on the starch of food in the ordinary process of digestion. When gelatinized starch is subjected to treatment with malt extract at  $55^{\circ}$  to  $60^{\circ}$  C., it is converted into dextrin and maltose as follows:



In its commercial preparation maltose is separated from dextrin by crystallization in alcohol. By the action of weak acids and heat both dextrin and maltose are further converted into dextrose.

Maltose usually crystallizes in minute needles, and its molecule of water is expelled at  $110^{\circ}$  C. It is somewhat less soluble in water than dextrose. It is slightly soluble in alcohol, though less than sucrose. Solutions of maltose possess the property of birotation; i.e., when freshly prepared they do not at once assume their true optical activity. The rotation of a freshly prepared solution of maltose increases on standing, requiring several hours to reach its maximum. The specific rotary power, according to O'Sullivan, of anhydrous maltose is  $[\alpha]_D = 139.2$ ,  $[\alpha]_J = 154.5$ . For hydrated maltose  $[\alpha]_D$  would thus be  $132.2$ .

A normal solution of maltose hydrate on the Soleil-Ventzke scale should polarize at  $198.8^{\circ}$ .

#### DEXTRIN. COMMERCIAL GLUCOSE.

**DEXTRIN**,  $(C_6H_{10}O_5)_n$ , possesses more the nature of a gum than of a sugar, and is sometimes called British gum. It is said to occur naturally in the sap of various plants, but this is not definitely assured.



It undoubtedly occurs in beer and in bread crust, and is one of the constituents of commercial glucose. Like starch, it is convertible by hydrolysis with acid into dextrose. By treatment of starch with malt extract or diastase, starch is converted into dextrin and maltose, these two bodies being separated, in the commercial preparations of dextrin by repeated treatment with alcohol.

Dextrin is an uncrystallized, colorless, tasteless body, capable of being pulverized. It is readily soluble in water, slightly soluble in dilute alcohol, but insoluble in alcohol of 60% or stronger. It is not colored by iodine, and exercises no reducing action on alkaline copper solution. Its specific rotary power is  $[\alpha]_D = 200$ ,  $[\alpha]_j = 222$ .

Amylodextrin, erythrodextrin and achroodextrin are intermediate products formed in the transformation of starch into dextrose. Amylodextrin is colored purple and erythrodextrin red by iodine solution, while achroodextrin produces no coloration. It is probable that some of these dextrins are not simple substances.

**Commercial Glucose**, otherwise known as *mixing syrup*, *crystal syrup*, and *starch*, or *corn syrup*, is a heavy, mildly sweet, colorless, semi-fluid substance, having a gravity of 40° to 45° Baumé. It is largely used as an adulterant of maple syrup, molasses, honey, drip syrup, and jellies and jams, and as an ingredient of confectionery.

In France and Germany it is made from potato starch, but in the United States mainly from corn starch. The conversion is effected by boiling with dilute sulphuric or hydrochloric acid, after which the acid is neutralized with marble dust, or sodium carbonate respectively, the juice is filtered through bone black, and finally concentrated by evaporation, the degree of conversion and of concentration depending on whether the liquid glucose or the solid dextrose is wanted for the final product. The end product obtained by complete conversion is the dry commercial grape sugar, or dextrose, which is purified by repeated crystallization.

Commercial glucose is a mixture of dextrin, maltose, and dextrose of the following varying composition:

Dextrin.....	29.8%	to 45.3%
Maltose.....	4.6%	" 19.3%
Dextrose.....	34.3%	" 36.5%
Ash.....	0.32%	" 0.52%
Water.....	14.2%	" 17.2%



Calcium sulphate is usually found in the ash if sulphuric acid was used for conversion.

Solid commercial grape sugar, or dextrose, has the following composition:

Dextrin.....	0%	9.1%
Maltose.....	0%	1.8%
Dextrose.....	72%	99.4%
Ash.....	0.3%	0.75%
Water.....	0.6%	17.5%

*U. S. Standard glucose, mixing glucose, or confectioners' glucose*, is colorless glucose, varying in density between 41° and 45° Baumé, at a temperature of 100° F. (37.7° C.). It conforms in density, within these limits, to the degree Baumé it is claimed to show, and for a density of 41° Baumé contains not more than 21% of water, and for a density of 45° not more than 14%. It contains on a basis of 41° Baumé not more than 1% of ash, consisting chiefly of chlorides and sulphates of lime and soda.

**Healthfulness of Glucose.**—The analyst alleging commercial glucose as an adulterant is frequently asked in court as to its healthfulness, so that the following conclusions of a committee appointed some years ago by the National Academy of Sciences to ascertain among other things whether there is any danger attending the use of this product in food are in point: "First, that the manufacture of sugar from starch is a long-established industry, scientifically valuable and commercially important; second, that the processes which it employs at the present time are unobjectionable in their character and leave the product uncontaminated; third, that the starch sugar thus made and sent into commerce is of exceptional purity and uniformity of composition and contains no injurious substances; and fourth, that though having at best only about two-thirds the sweetening power of cane sugar, yet starch sugar is in no way inferior in healthfulness, there being no evidence before the committee that maize starch sugar, either in its normal condition or fermented, has any deleterious effect upon the system, even when taken in large quantities."

#### MILK SUGAR, OR LACTOSE.

Lactose ( $C_{12}H_{22}O_{11} + H_2O$ ) is prepared commercially from skim-milk by coagulating with rennet and digesting the whey with chalk and aluminum hydroxide. The insoluble matter is filtered out, and the filtrate is concentrated *in vacuo* to a syrup, which, on standing, yields



crystals of lactose. The product is purified by repeated crystallization.

Lactose ordinarily crystallizes in rhombic, hemihedral crystals. Its specific gravity is 1.525. Its water of crystallization is lost by drying at  $130^{\circ}\text{C}$ . It is soluble in 6 parts of cold water, and in  $2\frac{1}{2}$  or less of boiling water. It is insoluble in absolute alcohol and ether. It has a very slightly sweet taste.

The specific rotary power of milk sugar, after remaining in solution long enough to overcome its birotation, is

$$[\alpha]_D = 52.5.$$

In the ordinary souring of milk the lactose becomes converted into lactic acid.

On heating lactose with dilute acids it undergoes inversion, forming dextrose and galactose in accordance with the formula given on p. 565 illustrating the inversion of cane sugar.

Milk sugar is of considerable importance by reason of the large amount used of late in the preparation of modified milk for infant feeding.

Grape sugar and cane sugar are to be looked for as adulterants of milk sugar.

The purity of milk sugar is best established by titrating against Fehling's solution, 10 cc. of which are equivalent to 0.067 gram of lactose.

#### RAFFINOSE.

Raffinose,  $\text{C}_{18}\text{H}_{32}\text{O}_{16.5}\text{H}_2\text{O}$ , is a sugar belonging neither to the saccharose nor the glucose group, but to the so-called *saccharoid* group, the other members of which do not occur in foods.

Raffinose occurs in beet root molasses to the extent of from 3 to 4 per cent. It is a crystalline, slightly sweet substance, soluble in water and slightly soluble in alcohol. It does not reduce Fehling's solution, but readily undergoes fermentation with bottom yeast. On inversion it splits up into levulose and melibiose ( $\text{C}_{12}\text{H}_{22}\text{O}_{11}$ ).

The melting-point of raffinose is  $118^{\circ}$  to  $119^{\circ}\text{C}$ . Its specific rotary power  $[\alpha]_D = +104.5$  at a temperature of  $20^{\circ}\text{C}$ .

#### THE POLARISCOPE AND SACCHARIMETRY.

A full discussion of the principles of polarized light and even a detailed description of their application to the polariscope will not be given here, but the reader who wishes full information along this line is referred to



the various treatises such as those of Browne,\* Landolt,† Rolfe,‡ Spencer,§ Tucker,|| and Wiechmann,¶ in which various forms of polariscopes are described and their underlying principles discussed.

The **Soleil-Ventzke Saccharimeter** is the one most commonly used in this country, being adopted as the standard for all United States government work. Fig. 102 shows this instrument, known as the half-shadow apparatus, in its simplest form with a single movable wedge in its compensating system.

An excellent light for work with this instrument is that furnished by the Welsbach burner, a convenient form of lamp being shown in Fig. 111, in which the burner is inclosed in a sheet-metal chimney of suitable con-

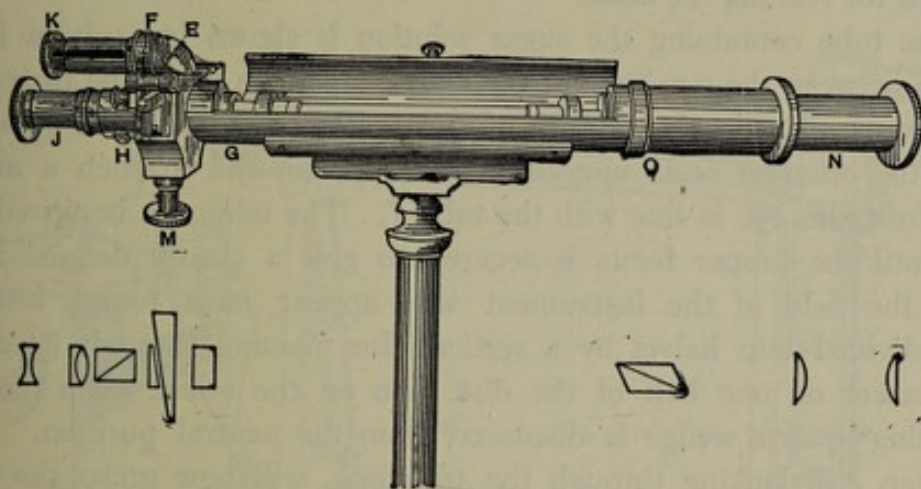


FIG. 102.—Single-wedge Saccharimeter.

struction. An argand, gas, or kerosene burner may, however, be used, and in a late form of Schmidt and Haensch instrument, Fig. 103, a specially constructed incandescent electric lamp is supplied.

The International Commission for Uniform Methods of Sugar Analysis at its seventh session held at New York, 1912, passed the following resolution based on studies by A. H. Bryan: "Wherever white light is used in polarimetric determinations, the same must be filtered through a solution of potassium bichromate of such a concentration that the percentage con-

\* Handbook of Sugar Analysis, New York, 1912.

† Optical Rotation of Organic Substances, trans. by Long, Easton, 1902.

‡ The Polariscopes in the Chemical Laboratory, New York, 1905.

§ Handbook for Sugar Manufacturers and their Chemists, New York, 1905.

|| Manual of Sugar Chemistry, New York, 1905.

¶ Sugar Analysis, New York, 1914.



tent of the solution multiplied by the length of the column of solution in centimeters is equal to nine."

*The Single-wedge Saccharimeter.*—The following description of the saccharimeter and directions for its use are from the revised regulations of the U. S. Internal Revenue Department. The tube *N*, Fig. 102, contains the illuminating system of lenses and is placed next to the lamp; the polarizing prism is at *O* and the analyzing prism at *H*. The quartz wedge compensating system is contained in the portions of the tube marked *FEG* and is controlled by the milled head *M*. The tube *J* carries a small telescope, through which the field of the instrument is viewed, and just above is the reading-tube *K*, which is provided with a mirror and magnifying lens for reading the scale.

The tube containing the sugar solution is shown in position in the trough between the two ends of the instrument. In using the instrument the lamp is placed at a distance of at least 200 mm. from the polarizing end; the observer seats himself at the opposite end in such a manner as to bring his eye in line with the tube *J*. The telescope is moved in or out until the proper focus is secured to give a clearly defined image, when the field of the instrument will appear as a round, luminous disk, divided into halves by a vertical line passing through its center, and darker on one half of the disk than on the other, when the compensating quartz wedge is displaced from the neutral position. If the observer, still looking through the telescope, will now grasp the milled head *M* and rotate it first one way and then the other, he will find that the appearance of the field changes, and at a certain point the dark half becomes light and the light half dark. By rotating the milled head delicately backward and forward over this point he will be able to find the exact position of the quartz wedge operated by it, in which the field is neutral, or of the same intensity of light on both halves. The three different appearances presented by the field are shown in Fig. 106, opposite page 605.

One of the compensating quartz wedges is fixed and the other is movable, sliding one way or the other according as the milled head is turned, so that for different relative positions of the two wedges a different thickness of quartz is interposed in the path of the polarized ray. By this means the amount of the rotation which the sugar solution or other optically active substance examined exerts upon the light polarized by the prism at *O* may be, as it were, counteracted by varying the relative position of the wedges.



With the milled head set at the point which gives the appearance of the middle disk shown in Fig. 106, the eye of the observer is raised to the reading tube *K*, which is adjusted to secure a plain reading of the divisions, and the position of the scale is noted. It will be seen that the scale proper is attached to the quartz wedge, which is moved by the milled head; and attached to the other quartz wedge is a small scale called a *vernier*, which is fixed, and which serves for the exact determination of the position of the movable scale with reference to it. On each side of the zero line of the vernier a space corresponding to *nine* divisions of the movable scale is divided into *ten* equal parts. By this device the fractional part of a degree indicated by the position of the zero line is ascertained in

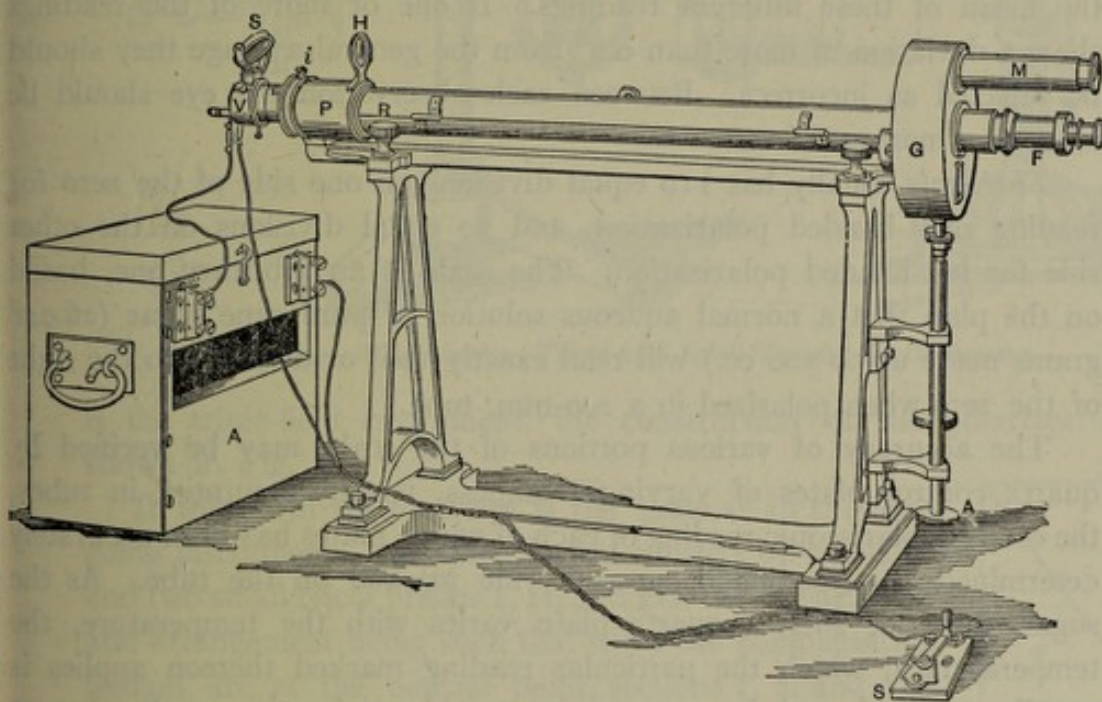


FIG. 103.—Double-wedge Soleil-Ventzke Saccharimeter, mounted on Bock Stand and provided with Incandescent Electric Lamp.

tenths; it is only necessary to count from zero until a line is found which makes a continuous line with one on the movable scale.

With the neutral field, as indicated above, the zero of the movable scale should correspond closely with the zero of the vernier, unless the zero point is out of adjustment.

*Adjusting the Instrument.*—If the observer desires to secure an *exact* adjustment of the zero of the scale, or in any case if the latter deviates more than two-tenths of a degree, the zero lines are made to coincide by moving the milled head and securing a neutral field at this point by



means of the small key which comes with the instrument, and which fits a small nipple on the *left*-hand side of *F*, the fixed quartz wedge of the compensating system. This nipple must not be confounded with a similar nipple on the *right*-hand side of the analyzing prism *H*, which it fits as well, but which *must never be touched*, as the adjustment of the instrument would be seriously disturbed by moving it. With the key on the proper nipple it is turned one way or the other until the field is neutral. Unless the deviation of the zero be greater than  $0.2^\circ$  it will not be necessary to use the key, but only to note the amount of the deviation, and for this purpose the observer must not be content with a single setting, but must perform the operation five or six times and take the mean of these different readings. If one or more of the readings show a deviation of more than  $0.2^\circ$  from the general average they should be rejected as incorrect. Between each observation the eye should be allowed a moment of rest.

*The Scale* usually has 110 equal divisions on one side of the zero for reading right-handed polarization, and 20 equal divisions on the other side for left-handed polarization. The scale is an arbitrary one, based on the plan that a normal aqueous solution of pure cane sugar (26.048 grams made up to 100 cc.) will read exactly  $100^\circ$  or divisions to the right of the zero when polarized in a 200-mm. tube.

The accuracy of various portions of the scale may be verified by quartz control plates of varying thickness, usually mounted in tubes, the correct polariscopic reading of each of which plates has been accurately determined, this reading being as a rule marked on the tube. As the sugar value of such a quartz plate varies with the temperature, the temperature at which the particular reading marked thereon applies is usually specified, and in many cases a table giving its exact value at different temperatures from  $10^\circ$  to  $35^\circ$  accompanies the plate.

*The Double-wedge Saccharimeter* is shown in Fig. 104, the arrangement of the optical parts being also shown.

In this instrument the two sets of wedges employed are of opposite optical properties, so that extreme accuracy may be arrived at by making the readings with both, the inaccuracies of one being compensated for by the other. Ordinarily in using this form, one movable wedge, say the one controlled by the right-hand milled screw head, is set at zero, while the reading of the sugar solution or other substance to be polarized is made with the other movable wedge.

*The Triple-field Saccharimeter.*—The latest form of saccharimeter



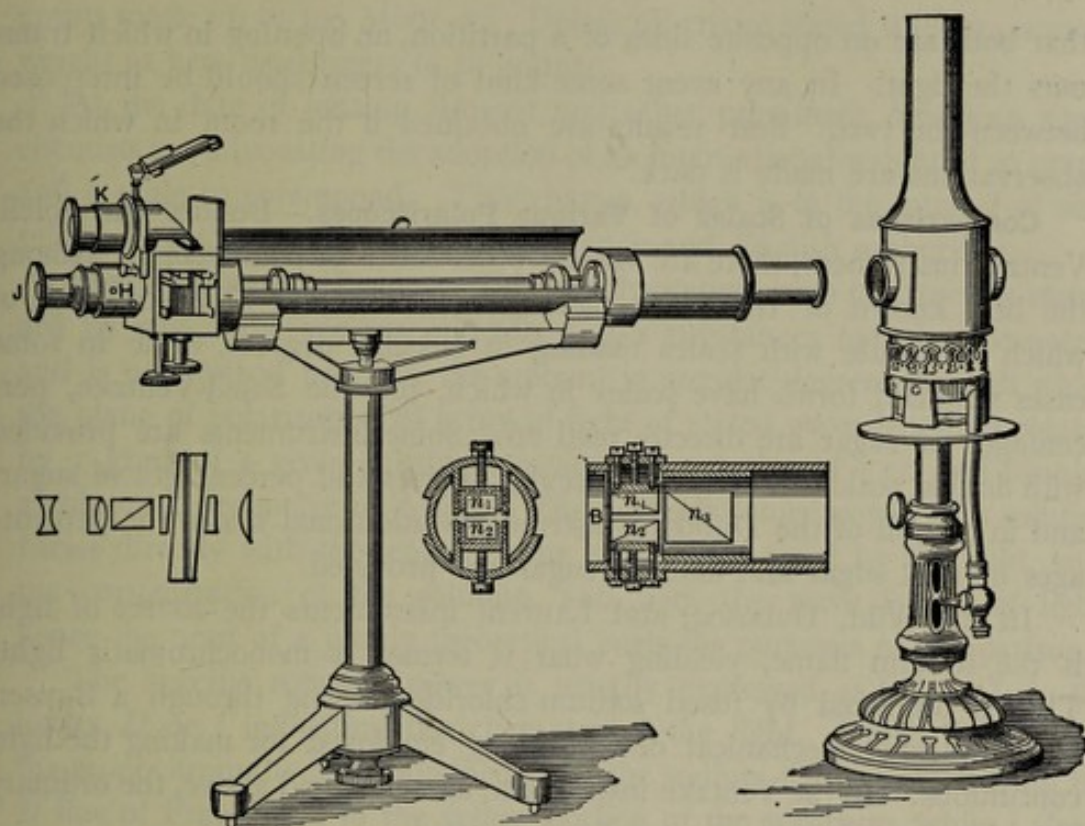


FIG. 104.—Triple-wedge, Triple-field Soleil-Ventzke Saccharimeter.

is the triple-field instrument, the construction of the polarizer being shown in Fig. 105.

In this form the analyzer is the same as in the foregoing instruments, but the polarizer consists of one large and two small Nicol prisms I, II, and III, the construction and arrangement being such that when the compensating wedges are at the neutral point, sections 1, 2, and 3 of the circular field (corresponding respectively to the prisms I, II, and III) are evenly lighted, forming a circular uniformly colored field, while in any other position of the wedges section 1 is dark while 2 and 3 are light or vice versa. The accompanying diagram, Fig. 106, shows the appearance of the field of this instrument in the three positions of the quartz wedge, viz., at the neutral point and at both sides thereof.

The lamp used for illumination should be separated from the polariscope on account of the influence of its heat on the readings. This is best accomplished by having the lamp in a separate compartment from the polariscope, so

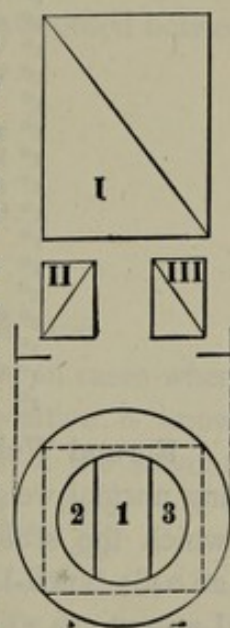


FIG. 105.



that both are on opposite sides of a partition, an opening in which transmits the light. In any event some kind of screen should be interposed between the two. Best results are obtained if the room in which the observations are made is dark.

**Comparisons of Scales of Various Polariscopes.**—Besides the Soleil-Ventzke instrument, there are various other forms of polariscope. Among the best known of these are Laurent's, Wild's, and Duboscq's, all of which are made with scales reading in circular degrees, while in some cases modified forms have scales in which, like the Soleil-Ventzke, percentages of sugar are directly read off. Some instruments are provided with double scales reading both circular degrees and percentages of sugar, and in certain of the Duboscq instruments additional scales for percentages of milk sugar and diabetic sugar are provided.

In the Wild, Duboscq, and Laurent instruments the source of light is the sodium flame, yielding what is termed a monochromatic light. This is produced by fused sodium chloride passing through a Bunsen flame, various mechanical devices being employed for making the light continuous. In the Ventzke instrument, as was stated above, the ordinary light from a bright gas or oil flame is used.

For convenience in conversion of readings on one instrument to their equivalents on other scales, the following factors can be used:

1° Ventzke	=0.3468° angular rotation <i>D</i> .
1° angular rotation <i>D</i>	=2.8835° Ventzke.
1° Ventzke	=2.6048° Wild (sugar scale).
1° Wild (sugar scale)	=0.3840° Ventzke.
1° " " "	=0.1331° angular rotation <i>D</i> .
1° angular rotation <i>D</i>	=7.5110° Wild (sugar scale)
1° Laurent (sugar scale)	=0.2167° angular rotation <i>D</i> .
1° angular rotation <i>D</i>	=4.6154° Laurent (sugar scale).
1° Soleil-Duboscq	=0.2167° angular rotation <i>D</i> .
1° " " "	=0.2450° " " <i>j</i> .
1° " " "	=0.620° Soleil-Ventzke.
1° " " "	=1.619° Wild.
1° Soleil-Ventzke	=1.608° Soleil-Duboscq (old scale).
1° " " "	=1.593° " " (new scale).
1° Wild	=0.611° " " (Wild normal weight 10).
1° " "	=1.223° " " ( " " " 20).

**Normal Weights of Sugar for Different Instruments.**—The following normal weights (number of grams in 100 cc. at 17.5° C.) are those on which the scales of the various instruments are based: Soleil-Ventzke, 26.048; Soleil-Duboscq 16.29 (formerly 16.19); Wild, usually, 10 or 20; Laurent, 16.29.

The International Commission for Uniform Methods in Sugar Analysis has decided to use for the Ventzke scale 26 grams and make up at 20° C. to 100 metric cc., which figures are approximately equivalent to 26.048



grams made up to 100 Mohr cc. Unless otherwise stated the term normal weight as here used refers to 26 grams.

At the date of writing Browne and other prominent American sugar chemists are advocating the adoption of an international weight of 20 grams and a scale to correspond. This change, which is in the interest of simplicity, is endorsed also by leading French and English authorities.

**Specific Rotatory Power.**—This is a theoretical term to express a standard by which the various optically active substances may be compared, and is understood to mean the amount in angular degrees through which the plane of polarization of a ray of light of stated wave length is rotated by 1 gram of a given substance in aqueous solution of 1 cc. and forming a column 1 decimeter in length. The actual rotatory power of a solution varies directly with the length of the column traversed by the light, with the concentration of the solution, and with the wave length of light, hence the need of a purely theoretical basis for purposes of comparison.

The specific rotatory power is usually expressed as  $[\alpha]_D$  or  $[\alpha]_j$ , the letters *D* or *j* indicating the character of the light. Thus, *D* indicates the monochromatic light obtained from the sodium flame, named from the *D* line of Fraunhofer in the yellow portion of the spectrum, while *j* (from the French *jaune*) indicates what is known as the *transition tint*, the rose-purple color produced when ordinary white light passes through the polarizer and analyzer, placed with their principal sections parallel to each other and with a plate of quartz 3.75 mm. thick interposed between them.\*

The specific rotatory power is determined as follows:

$$[\alpha]_D \text{ or } [\alpha]_j = \frac{100a}{cl},$$

where *a* = observed angular rotation,

*c* = grams of the substance in 100 cc. of the solution, and

*l* = length of the observation-tube in decimeters; or in cases where, instead of the grams per 100 cc., the percentage composition is known (expressed by *p* = grams of the substance in 100 grams of the solvent),

and the specific gravity (expressed by *d*), then  $[\alpha]_D$  or  $[\alpha]_j = \frac{100a}{pdl}$ .

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\* Some confusion is caused by the adoption of the characters *D* and *j*, since both would naturally seem to indicate yellow light. The so-called *transition tint* above defined is, however, complementary to the mean yellow, or *jaune moyen*, and it is the complementary color and not the yellow itself that is indicated by the character *j*.



**Birotation.**—In polarizing solutions of all the common sugars other than sucrose the phenomenon of birotation should be taken into account, whereby a change in optical activity is shown by standing. Thus, solutions of dextrose, levulose, and lactose polarize much higher when freshly prepared than after long standing, requiring in some instances several hours before the lowest or normal figure is reached. Maltose, on the other hand, increases in polarization after standing in solution. By boiling the solution it may at once be brought to its correct reading. The desired result may also be accomplished by adding a few drops of ammonia, either treatment being resorted to before the solution is made up to the required volume.

#### ANALYSIS OF CANE SUGAR AND ITS PRODUCTS.

**Qualitative Tests for Sucrose.**—(a) *Polariscope Test.*—The substance to be tested, if not already in solution, is dissolved in water, and if the solution is not perfectly clear, is clarified by the addition of alumina cream or by subacetate of lead (page 610) and filtered. An observation tube is filled with the clear solution and the polariscope reading noted. A measured portion of the same solution is then treated with one-tenth its volume of concentrated hydrochloric acid and is subjected to inversion (page 611), after which the same tube as before is filled with the inverted solution and a second reading obtained, one-tenth of the observed reading being added for the true invert polariscopic reading. If the two readings are virtually the same, sucrose is absent, but, in the presence of sucrose, the second reading will be considerably lower than the first or may even be to the left of the zero.

(b) *Test with Nitrate of Cobalt.\**—Prepare a 5% solution of cobaltous nitrate, and a 50% solution of potassium hydroxide. If the sugar solution to be tested contains dextrin or gums, these should be first removed by treatment with alcohol. 15 cc. of the sugar solution to be tested are mixed with 5 cc. of the cobaltous nitrate reagent, and 2 cc. of the potassium hydroxide solution are added. Sucrose produces under these conditions a permanent amethyst-blue color, while dextrose gives at first a turquoise-blue passing over into light green. In a mixture of the two sugars the color due to sucrose will predominate.

According to Wiley, 1 part of sucrose in 9 parts of dextrose may be

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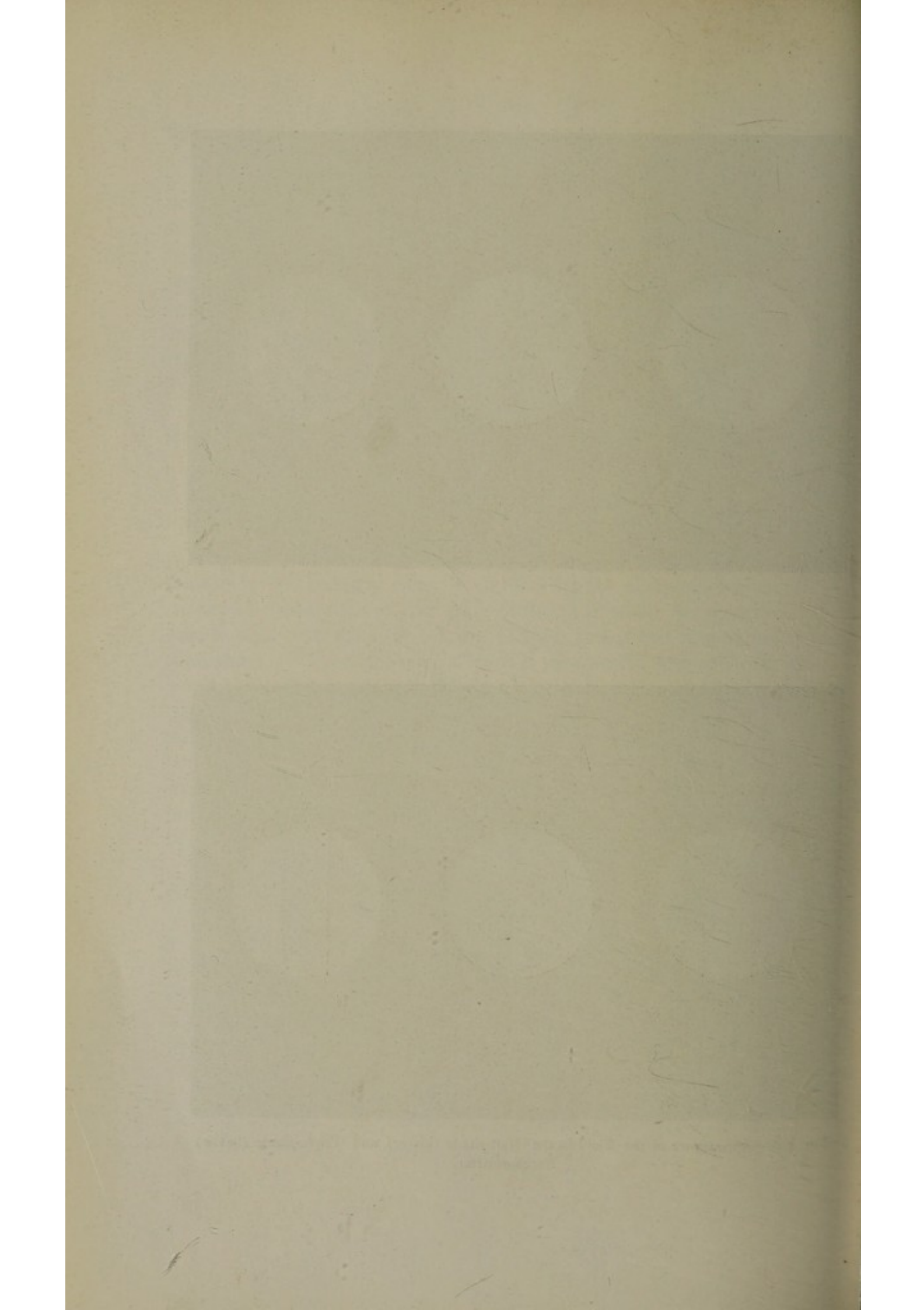
\* Wiley, Ag. Anal., p. 189.





FIG. 106.—Appearance of the Field in the Half-shade (above) and Triple-shade (below) Saccharimeter.







detected by this test. Browne\* notes that other sugars give a similar coloration, hence the test is not infallible although a useful guide.

**ANALYSIS OF CANE SUGAR.**—In the case of commercial granulated or loaf sugar the sucrose determination is usually all that is necessary to determine its purity, and the same is true, as a rule, of the powdered white sugars. A fairly complete analysis of raw or brown sugar consists in the determinations of moisture, sucrose, invert sugar, ash, organic non-sugars, and quotient of purity. Care should be taken that the portion subjected to analysis is a fair representation of the whole, and is perfectly homogeneous.

**Determination of Moisture.**—Two to five grams of the sample are dried in a flat, tared metal dish, to constant weight *in vacuo*, or in a McGill oven † in a current of air, at about 70° C., at which temperature levulose is not decomposed. For ordinary purposes drying to constant weight in a boiling-water oven is sufficiently accurate.

**Determination of Ash.**—The residue from the moisture determination is burned slowly and cautiously over a low flame until frothing has ceased. Afterwards increase the flame and ignite to a white ash at a low red heat, preferably in a muffle furnace.

In igniting saccharine substances which contain an appreciable amount of cane sugar, the contents of the dish will swell up and froth, unless great care be taken, to such an extent as to flow over the sides of the dish, occasioning loss and inconvenience. Such frothing may be largely held in check by directing the flame at first down from above upon the pasty mass, instead of from under the dish as ordinarily, till all is reduced to a dry char, afterwards continuing the ignition from below in the usual manner.

**Organic Non-sugars.**—These consist mainly of compounds of organic acids, together with gum, coloring matter, albuminous bodies, etc. They

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\* Sugar Anal., p. 681.

† A. McGill, Laboratory of Inland Revenue, Ottawa, Canada, has devised a forced-draft water-oven for drying at temperatures between 60° and 90° C. The oven is heated by means of ordinary gas-burners, and the temperature is controlled by introducing at the bottom of the oven a blast of air from a blower run by a small water-motor. Before discharging into the oven, the air-tube enters the water-chamber and is coiled a number of times in order to sufficiently warm the air before it enters the oven. The exit end of the air-tube is covered with a concavo-convex disk in order to distribute the blast and to prevent harmful currents. By regulating the burners and the flow of air, a fairly constant temperature can be obtained. The bottom of the oven is curved instead of flat, to prevent bumping when the water is boiling; a perforated plate serves as a false bottom.



are determined by difference between 100% and the sum of the sucrose, invert sugar, moisture, and ash.

**Quotient of Purity.**—By this term is meant the percentage of pure sugar in the dry substance. It is calculated by dividing the per cent of sucrose by the percentage of total solids and multiplying the result by 100.

**Determination of Sucrose by the Polariscope.**—*Reagents.*—(a) *Lead-Subacetate Solution.*\*—Boil for half an hour 430 grams of normal lead acetate, 130 grams of litharge, and 1000 cc. of water, allow to cool and settle. Dilute the supernatant liquid to 1.25 specific gravity with recently boiled water.

Anhydrous lead subacetate, first proposed by Horne,† may be substituted for the solution.

(b) *Alumina Cream.*—Divide a cold, saturated solution of alum into two unequal portions, add to the larger a slight excess of ammonia, then by degrees the remaining portion to faint acid reaction.

*Process.*—If the Soleil-Ventze polariscope is to be used, weigh out 26 grams of the sugar, which may conveniently be done in the German-silver, tared tray especially designed for this purpose (Fig. 107). If any

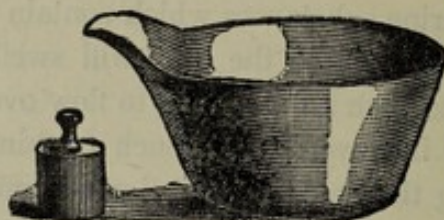


FIG. 107. German-silver Sugar-tray with Tare.

other instrument is employed, weigh out the standard or normal weight for that instrument (see page 606). Transfer the sugar by washing to a 100-cc. graduated sugar-flask, and if the solution is perfectly clear, as would be the case with a refined sugar, make up to the mark and shake to insure a uniform solution. If the solution is slightly turbid, or more or less opaque or dark-colored, a clarifier must be added before making up to the mark to obtain a clear solution for polarization. The kind and amount of clarifier to be used depends on the nature of the sugar solution and must be learned by experience. If the turbidity is only slight, from 5 to 10 cc. of alumina cream alone will often prove sufficient; if

\* U. S. P. lead subacetate, sometimes sold as Goulard's extract, may also be used.

† Jour. Am. Chem. Soc., 26, 1904, p. 186.



more opaque, 10 cc. of lead subacetate solution or a small amount of the dry salt may be used.

For additional details as to clarification see page 644, under Molasses.

After adding the clarifier, the flask is filled to the mark with water and shaken, the solution being poured upon a dry filter and the first few cubic centimeters of the filtrate rejected. A 200-mm. observation-tube is filled with the clear sugar solution and the polarization noted. If sucrose is the only optically active substance present, the direct reading on the polariscope will indicate its percentage.

*Inversion by the Clerget-Herzfeld Method.*—In the presence of invert or other sugars the normal solution is subjected to inversion as follows: Free a portion of the solution from lead by treating with anhydrous

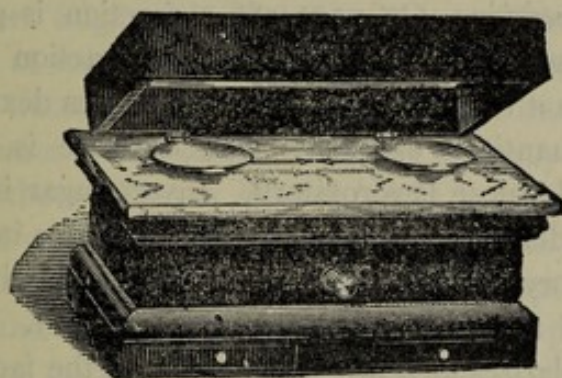


FIG. 108.—A Convenient Sugar-scale.

sodium carbonate, sodium sulphate or potassium oxalate, filter, place 50 cc. in a 100-cc. flask, add 25 cc. of water and little by little, while rotating the flask, 5 cc. of 38.8% hydrochloric acid. Heat in a water bath at 70° C., so that the solution in the flask reaches 67° to 69° C. in 2½ to 3 minutes. Maintain at 69° C. during 7 to 7½ minutes, making a total time of heating of 10 minutes. Remove the flask, cool the contents rapidly to 20° C., and dilute to 100 cc. Polarize this solution in a 200-mm. tube provided with a lateral branch and a water jacket, passing a current of water around the tube to maintain a temperature of 20° C.

The inversion may also be accomplished by allowing a mixture of 50 cc. of the clarified solution, freed from lead, and 5 cc. of the acid to stand for 24 hours at no less than 20° C. or for 10 hours at not less than 25°.

The sucrose is obtained by the Clerget-Herzfeld formula based on the rotation of cane sugar before and after inversion,

$$S = \frac{100(a-b)}{142.66 - t/2},$$



where  $S$  = per cent of sucrose,  $a$  = direct polarization,  $b$  = invert polarization, and  $t$  = temperature. Note that if the direct polarization is to the right or positive, and the invert to the left or negative, then  $a - b$  would be the sum of the two polarizations.

In many cases where it is almost impossible to obtain a colorless solution for polarization in the 200-mm. tube, a 100-mm. tube may be employed, and the readings multiplied by 2, or half the normal weight,\* viz., 13 grams of the sample may be taken and made up to 100 cc., the 200-mm. tube employed, and the readings multiplied by 2.

The determination of sucrose by the Clerget-Herzfeld formula is applicable to all mixtures of the common sugars excepting those in which lactose, or milk sugar, is present.

*Theory of Inversion.*—On page 586 a reaction is given showing that when sucrose is subjected to inversion by the action of dilute acids or of invertase or yeast it splits up into the two sugars dextrose and levulose, forming equal quantities of each. The dextrose is, however, dextro-rotatory and the levulose lævorotatory. Invert sugar is the term applied to the mixture of dextrose and levulose formed by the inversion of sucrose. The specific rotatory power of sucrose varies so little with the temperature as to be regarded for practical purposes as constant. At  $87^{\circ}$  a solution of invert sugar polarizes at zero. This is due to the fact that the rotatory power of levulose, unlike that of sucrose and dextrose, varies with the temperature. At from  $87^{\circ}$  to  $88^{\circ}$  the left-handed rotation of the levulose balances the right-handed rotation of the dextrose in the invert sugar, hence the zero reading. As the temperature decreases from  $87^{\circ}$ , the rotatory power of the levulose proportionally increases, till at  $0^{\circ}$  the normal invert sugar solution would polarize  $-42.66$ . On these facts the Clerget-Herzfeld formula is based, assuming that a normal solution of pure cane sugar polarizes  $+100$ , while after inversion the reading for  $0^{\circ}$  temperature would be  $-42.66$  and would decrease  $0.5$  for each degree in temperature above  $0^{\circ}$ . Thus at  $20^{\circ}$  the invert reading would be  $-32.66$ .

*Neutralization* of the free acid after inversion is sometimes practiced to avoid the disturbing influence of mineral acid on the polarization of *d*-fructose as well as of certain impurities present in molasses, juices, etc.

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\* Wherever the term "normal weight" occurs hereafter will be meant, unless otherwise noted, the normal weight of sugar for the Soleil-Ventzke polariscope, viz., 26 grams, and by a "normal solution" will be meant 26 grams in 100 cc. of water at  $20^{\circ}$  C. Clerget's formula, as originally worked out by him, was not based on this normal weight, but on 16.35 grams. It is, however, applicable to 26 grams.



When neutralization is practiced the factor in the Clerget-Herzfeld formula should be 141.7 instead of 142.66.\* Neutralization, however, introduces another disturbing factor, namely sodium chloride, to counterbalance which Saillard † adds an equivalent amount of this salt to the solution used for direct polarization. The most rational system of defecating and inverting is that proposed by Deerr.‡ He employs for defecation barium hydroxide in conjunction with a acid reagent containing aluminum sulphate and sulphuric acid in such proportions that the two solutions neutralize each other forming aluminum hydroxide and barium sulphate. After direct polarization of the filtered solution inversion is carried on with another portion of the acid reagent, then an equivalent amount of barium hydroxide is added thus again precipitating all added substances.

**Detection of Invert Sugar.**—*Methyl-blue Test.*§—This test depends on the decolorization of methyl blue by invert sugar. Twenty grams of sugar are dissolved in water and made up to 100 cc. If the solution is not clear, sufficient subacetate of lead solution is added to clarify before making up to the mark, and the solution is filtered. Add to the filtrate enough 10% sodium carbonate solution to make alkaline, and filter a second time. Take about 50 cc. of the filtrate in a casserole, add 2 drops of a 1% solution of methyl blue, and boil over a free flame, noticing particularly the time the solution begins to boil.

If the color disappears in one minute after boiling, there is present at least 0.01% of invert sugar. If it is not completely decolorized by 3 minutes' boiling, no invert sugar is present.

**Determination of Invert Sugar in Cane Sugar Products by the Polariscopes.**—While invert sugar is best determined by Fehling's solution as described elsewhere, it may be approximately estimated by the polariscopes, though less satisfactorily. On page 671 a method is given for the determination of levulose by polariscopic readings at two different temperatures. Since invert sugar is composed of equal parts by weight of dextrose and levulose, the percentage of levulose multiplied by 2 would give that of invert sugar.

**Test for Ultramarine in Sugar.**||—A large amount of the sugar is dissolved in water and the coloring matter is allowed to settle out, wash-

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\* Browne, Handbook of Sugar Analysis, New York, 1912, p. 271.

† 8th Int. Cong. App. Chem., 27, 1912, p. 63.

‡ Int. Sugar Jour., 17, 1915, p. 179.

§ Wiechmann, Sugar Analysis, New York, 1914, p. 110.

|| Leffmann and Beam, Select Methods of Food Analysis, p. 126.



ing the residue several times by decantation. On treatment with hydrochloric acid, the blue color is discharged if due to ultramarine.

#### SUGAR DETERMINATION BY COPPER REDUCTION.

Various convenient methods of determining sugars depend on the readiness with which certain of them, known as reducing sugars, act on copper salts, especially on the tartrate of copper, reducing it to cuprous oxide.

This reducing power is exercised in a definite degree under fixed conditions, so that the amount of reducing sugar present may be accurately determined. Of the common sugars, sucrose is the only one that has practically no direct reducing action, but on undergoing inversion it is converted into reducing sugars, which are readily determined.

**Use of Fehling's Solution.**—There are various well-known mixtures of copper sulphate, tartaric acid salts (usually Rochelle salts or cream of tartar), and alkalies, called after chemists who have employed them in the determination of the reducing sugars, each one possessing certain advantages, but none have become so widely adopted as Fehling's solution, the use of which in one form or another is now well-nigh universal.

There are a number of methods by which Fehling's solution is employed for this purpose, both volumetric and gravimetric. The former are simpler and quicker of manipulation, and thus are preferable for commercial work where extreme accuracy is not required. The gravimetric methods are usually considered more delicate and accurate, calling for less skill, but more time in arriving at results, and with less of the "personal element" than the volumetric.

Some modifications of the Fehling method, especially as carried out gravimetrically, differ for the various reducing sugars to be determined, and others are carried out alike, so far as manipulation is concerned, whether the particular sugar to be determined be dextrose, maltose, or lactose.

While, strictly speaking, the reducing power of dextrose, levulose, and invert sugar are not identical, it is customary in commercial work to regard them as such, and no appreciable error arises in consequence except in extreme cases. Thus the term "reducing sugars" is commonly applied indiscriminately to dextrose, levulose, and invert sugar, the same factor being used in calculating either, in mixtures wherein other reducing sugars, as lactose, maltose, etc., having widely different reducing powers are absent.



Fehling's solution is made up in two separate parts as follows:

*A. Fehling's Copper Solution.*—34.639 grams of carefully selected crystals of pure copper sulphate dissolved in water and diluted to exactly 500 cc.

*B. Fehling's Alkaline Tartrate Solution.*—173 grams Rochelle salts and 50 grams sodium hydroxide are dissolved in water and diluted to exactly 500 cc.

The Fehling solution should be standardized by dissolving 0.5 gram of pure anhydrous dextrose in water, and diluting to exactly 100 cc. Ten cubic centimeters of this dextrose solution should exactly reduce the copper in 10 cc. of the Fehling (5 cc. each of solutions *A* and *B*) when conducted according to the volumetric process described below.

**VOLUMETRIC FEHLING PROCESS.**—For determining dextrose, levulose, or invert sugar, prepare a clarified, delead, and neutralized solution of the sugar of such a strength that an accurately weighed amount dissolved in water and made up to 100 cc. shall not contain more than 1% of the reducing sugar, as nearly as can be estimated with or without a rough preliminary titration. For the determination of lactose or maltose a 1½% solution may be used.

Measure accurately into a flask of about 250 cc. capacity 5 cc. Fehling's copper sulphate solution, *A*, and 5 cc. of the alkaline solution, *B*. Add about 40 cc. of water, mix and boil over a free flame, with copper gauze beneath the flask. While still boiling, add from a pipette or burette a measured quantity of the sugar solution, prepared as above, until the copper after three minutes' boiling is all reduced to cuprous oxide. The end-point is determined in a variety of ways. Practice will soon enable the eye to judge the near approach of the end-point by the changes in color that take place in the solution, which turns from a deep blue, first to green, then to a dull-red tint, and finally to a bright brick-red. The sugar-containing solution may be added from the burette quite rapidly until the solution reaches the dull-red tint, after which care is taken to add a little at a time, keeping account of the total amount added. If the flask be removed from the flame, and the bright, diffused light from a window viewed through the solution with the eye on a level with the surface, a thin film scarcely wider than a line will be observed just below the surface (see Fig. 109), which is blue so long as some of the copper in the solution remains unreduced. When, however, all the copper has been reduced, this film ceases to be blue and becomes colorless or yellow.

If the film is not at once apparent, it may often be made quite notice-



able by simply diluting the solution in the flask with water. At the approach of the end-point the sugar-containing solution should be added a very little at a time. The exact end-point is best arrived at by filtering off a few drops

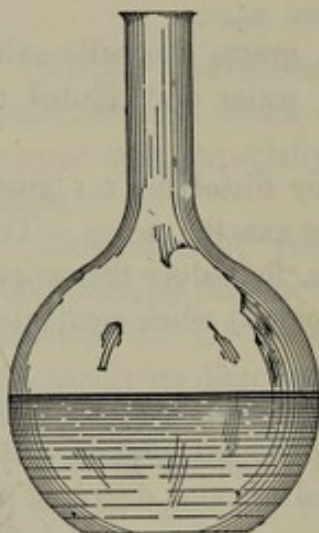


FIG. 109.—Flask and Contents used in Volumetric Fehling Determinations. Showing layer just beneath the surface, the color of which indicates the end-point in adding the sugar-containing liquid.

of the liquid, acidifying the filtrate with acetic acid, and adding a drop of a solution of ferrocyanide of potassium. As long as there is un-reduced copper present, a precipitate or brown-red coloration will appear when the ferrocyanide is added. The testing is greatly facilitated by lowering a small filter into the liquid by means of forceps and removing a small portion of the clear solution thus obtained with a medicine dropper (B. B. Ross). The sugar solution toward the end should be added to the contents in the flask in small installments (say half a cubic centimeter each time), boiling the liquor for at least three minutes after each addition, until no brown-red coloration is produced by adding the ferrocyanide to a little of the filtered acidified liquid. When the number of cubic centimeters of sugar solution necessary to reduce the copper has thus been determined, a second titration should be made to verify the first, running the entire amount of sugar-containing

liquid found necessary in the first case into the second flask.

The equivalents of 10 cc. of the mixed alkaline copper solution in the above method are, in terms of the common reducing sugars, as follows:

- 0.05 gram of invert sugar, dextrose, or levulose;
- 0.0475 gram of cane sugar after inversion;
- 0.0807 gram of maltose;
- 0.067 gram of lactose.

Suppose, for example, a sample of brown sugar is to be examined for invert sugar. This class of sugar has usually from 2 to 6 per cent of invert sugar. Hence, if 10 grams of the sample are dissolved in 100 cc., the resulting solution will contain not more than 1% of invert sugar.

Suppose 12.9 cc. of this 10% sugar solution were found by the above process to reduce 10 cc. of Fehling's solution.

10 cc. Fehling's solution are equivalent to 0.05 gram invert sugar.



Therefore 12.9 cc. of the sugar solution contain 0.05 gram invert-sugar.

100 cc. sugar solution contain 10 grams sample, and 12.9 cc. contain 1.29 grams sample, the equivalent of 0.05 gram invert sugar.

$$\text{Hence per cent invert sugar} = \frac{0.05 \times 100}{1.29} = 3.9.$$

**GRAVIMETRIC FEHLING PROCESSES.**—In determining reducing sugars by gravimetric processes, a measured volume of the sugar solution is allowed to act upon a measured volume of hot Fehling's solution for a fixed time, thus forming cuprous oxide. This may be dried and weighed direct, but is more commonly converted either into cupric oxide by ignition, or into metallic copper by reduction with hydrogen or by electrolysis. In any case the sugar is calculated from the weight of the cuprous oxide, the cupric oxide, or the metallic copper (whichever method be used) by the employment of the proper factor, or by the use of tables compiled for the purpose.

*Note.*—Much difference of opinion exists as to the best and most accurate Fehling gravimetric method to employ. For the determination of dextrose, the Association of Official Agricultural Chemists has given its approval to the Allihn method, wherein the cuprous oxide deposited is further reduced to metallic copper and the dextrose calculated from the copper by Allihn's table.

The author for two reasons prefers the method of O'Sullivan as employed by Defren, with the use of the Defren tables, in accordance with which the reducing sugar is expressed in terms of its equivalence to cupric oxide, first because of its comparative simplicity, involving as it does less processes than the Allihn method (each additional process introducing a possible source of error), and, second, because the same method as carried out is applicable for the determination not only of dextrose, but also of maltose and lactose, Defren having worked out tables adopted for them all. Munsen and Walker\* have also devised a simple method with accompanying tables, adapted, with a uniform system of procedure, to the determination of the various reducing sugars. In using the tables for dextrose, maltose, and lactose compiled by Allihn, Wein, and Soxhlet, the method employed must in each case be carried out in strict accordance to the minutest details adopted by each of the above authorities, and they are by no means uniform.

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 107 (rev.), p. 241.



**The Defren-O'Sullivan Method.\***—Mix 15 cc. of Fehling's copper solution *A* (page 615), with 15 cc. of the tartrate solution, *B*, in a quarter-liter Erlenmeyer flask, and add 50 cc. of distilled water. Place the flask and its contents in a boiling water bath and allow them to remain five minutes. Then run rapidly from a burette into the hot liquor in the flask 25 cc. of the sugar solution to be tested (which should contain not more than one-half per cent of reducing sugar). Allow the flask to remain in the boiling water bath just fifteen minutes after the addition of the sugar solution, remove, and with the aid of a vacuum filter the contents rapidly in a platinum or porcelain Gooch crucible containing a layer of prepared asbestos fiber about 1 cm. thick, the Gooch with the asbestos having been previously ignited, cooled, and weighed. The cuprous oxide precipitate is thoroughly washed with boiling distilled water till the water ceases to be alkaline.

The asbestos used should be of the long-fibered variety, and should be specially prepared as follows: Boil first with nitric acid (specific gravity 1.05 to 1.10), washing out the acid with hot water, then boil with a 25% solution of sodium hydroxide, and finally wash out the alkali with hot water. Keep the asbestos in a wide-mouthed flask or bottle, and transfer it to the Gooch by shaking it up in the water and pouring it quickly into the crucible while under suction.

Dry the Gooch with its contents in the oven, and finally heat to dull redness for fifteen minutes, during which the red cuprous oxide is converted into the black cupric oxide. If a platinum Gooch is used (and this variety is preferred by the writer), it may be heated directly over the low flame of a burner. If the Gooch is of porcelain, considerable care must be taken to avoid cracking the crucible, the heat being increased cautiously and the operation preferably conducted in a radiator or muffle. After oxidation as above, the crucible is transferred to a desiccator, cooled, and quickly weighed. From the milligrams of cupric oxide, calculate the milligrams of dextrose from the following table:

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\* Jour. Am. Chem. Soc., 18, 1896, p. 749, and Tech. Quart., 10, 1897, p. 167.



DEFREN'S TABLE FOR THE DETERMINATION OF DEXTROSE, MALTOSE, AND LACTOSE.

Milligrams of Cupric Oxide.	Milligrams of Dextrose.	Milligrams of Maltose.	Milligrams of Lactose.	Milligrams of Cupric Oxide.	Milligrams of Dextrose.	Milligrams of Maltose.	Milligrams of Lactose
30	13.2	21.7	18.8	80	35.4	58.1	50.5
31	13.7	22.4	19.5	81	35.9	58.9	51.1
32	14.1	23.1	20.1	82	36.3	59.6	51.7
33	14.6	23.9	20.7	83	36.8	60.3	52.4
34	15.0	24.6	21.4	84	37.2	61.1	53.0
35	15.4	25.3	22.0	85	37.7	61.8	53.6
36	15.9	26.1	22.6	86	38.1	62.5	54.3
37	16.3	26.8	23.3	87	38.5	63.3	54.9
38	16.8	27.5	23.9	88	39.0	64.0	55.5
39	17.2	28.3	24.5	89	39.4	64.7	56.2
40	17.6	29.0	25.2	90	39.9	65.5	56.8
41	18.1	29.7	25.8	91	40.3	66.2	57.4
42	18.5	30.5	26.4	92	40.8	66.9	58.1
43	19.0	31.2	27.1	93	41.2	67.7	58.7
44	19.4	31.9	27.7	94	41.7	68.4	59.3
45	19.9	32.7	28.3	95	42.1	69.1	60.0
46	20.3	33.4	29.0	96	42.5	69.9	60.6
47	20.7	34.1	29.6	97	43.0	70.6	61.2
48	21.2	34.8	30.2	98	43.4	71.3	61.9
49	21.6	35.5	30.8	99	43.9	72.1	62.5
50	22.1	36.2	31.5	100	44.4	72.8	63.2
51	22.5	37.0	32.1	101	44.8	73.5	63.8
52	23.0	37.7	32.7	102	45.3	74.3	64.4
53	23.4	38.4	33.3	103	45.7	75.0	65.1
54	23.8	39.2	34.0	104	46.2	75.7	65.7
55	24.2	39.9	34.6	105	46.6	76.5	66.3
56	24.7	40.5	35.2	106	47.0	77.2	67.0
57	25.1	41.3	35.9	107	47.5	77.9	67.6
58	25.5	42.1	36.5	108	48.0	78.7	68.2
59	26.0	42.8	37.1	109	48.4	79.4	68.9
60	26.4	43.5	37.8	110	48.9	80.1	69.5
61	26.9	44.3	38.4	111	49.3	80.9	70.1
62	27.3	45.0	39.0	112	49.8	81.6	70.8
63	27.8	45.7	39.7	113	50.2	82.3	71.4
64	28.2	46.5	40.3	114	50.7	83.1	72.0
65	28.7	47.2	40.9	115	51.1	83.8	72.7
66	29.1	47.9	41.6	116	51.6	84.5	73.3
67	29.5	48.6	42.2	117	52.0	85.2	74.0
68	30.0	49.4	42.8	118	52.4	85.9	74.6
69	30.4	50.1	43.5	119	52.9	86.6	75.2
70	30.9	50.8	44.1	120	53.3	87.4	75.9
71	31.3	51.6	44.7	121	53.8	88.1	76.6
72	31.8	52.3	45.4	122	54.2	88.9	77.2
73	32.2	53.0	46.0	123	54.7	89.6	77.9
74	32.6	53.8	46.6	124	55.1	90.3	78.5
75	33.1	54.5	47.3	125	55.6	91.1	79.1
76	33.5	55.2	47.9	126	56.0	91.8	79.8
77	34.0	56.0	48.5	127	56.5	92.5	80.4
78	34.4	56.7	49.2	128	56.9	93.3	81.1
79	34.9	57.4	49.8	129	57.3	94.0	81.7



DEFREN'S TABLE FOR THE DETERMINATION OF DEXTROSE, MALTOSE, AND LACTOSE—(Continued).

Milligrams of Cupric Oxide.	Milligrams of Dextrose.	Milligrams of Maltose.	Milligrams of Lactose.	Milligrams of Cupric Oxide.	Milligrams of Dextrose.	Milligrams of Maltose.	Milligrams of Lactose.
130	57.8	94.8	82.4	180	80.4	131.8	114.6
131	58.2	95.5	83.0	181	80.8	132.5	115.2
132	58.7	96.2	83.6	182	81.3	133.2	115.8
133	59.1	97.0	84.2	183	81.8	134.0	116.5
134	59.6	97.7	84.9	184	82.2	134.7	117.1
135	60.0	98.4	85.5	185	82.7	135.5	117.8
136	60.5	99.2	86.1	186	83.1	136.2	118.4
137	60.9	99.9	86.8	187	83.5	136.9	119.1
138	61.3	100.7	87.4	188	84.0	137.7	119.7
139	61.8	101.4	88.1	189	84.4	138.4	120.4
140	62.2	102.1	88.7	190	84.9	139.1	121.0
141	62.7	102.8	89.3	191	85.4	139.9	121.7
142	63.1	103.5	90.0	192	85.9	140.6	122.3
143	63.6	104.3	90.6	193	86.3	141.4	123.0
144	64.0	105.0	91.3	194	86.8	142.1	123.6
145	64.5	105.8	91.9	195	87.2	142.8	124.3
146	64.9	106.5	92.6	196	87.7	143.6	124.9
147	65.4	107.2	93.2	197	88.1	144.3	125.6
148	65.8	108.0	93.9	198	88.6	145.1	126.2
149	66.3	108.7	94.5	199	89.0	145.8	126.9
150	66.8	109.5	95.2	200	89.5	146.6	127.5
151	67.3	110.2	95.8	201	89.9	147.3	128.2
152	67.7	111.0	96.5	202	90.4	148.1	128.8
153	68.3	111.7	97.1	203	90.8	148.8	129.5
154	68.7	112.4	97.8	204	91.3	149.6	130.1
155	69.2	113.2	98.4	205	91.7	150.3	130.8
156	69.6	113.9	99.1	206	92.2	151.1	131.5
157	70.0	114.7	99.7	207	92.6	151.8	132.1
158	70.5	115.4	100.4	208	93.1	152.5	132.8
159	70.9	116.1	101.0	209	93.5	153.3	133.4
160	71.3	116.9	101.7	210	94.0	154.1	134.1
161	71.8	117.6	102.3	211	94.4	154.8	134.7
162	72.3	118.4	103.0	212	94.9	155.6	135.4
163	72.7	119.1	103.6	213	95.3	156.3	136.0
164	73.2	119.9	104.3	214	95.8	157.1	136.7
165	73.6	120.6	104.9	215	96.3	157.8	137.3
166	74.1	121.4	105.6	216	96.7	158.6	138.0
167	74.5	122.1	106.2	217	97.2	159.3	138.6
168	74.9	122.9	106.9	218	97.6	160.0	139.3
169	75.4	123.6	107.5	219	98.1	160.8	139.9
170	75.8	124.4	108.2	220	98.6	161.5	140.6
171	76.3	125.1	108.8	221	99.0	162.3	141.2
172	76.8	125.8	109.5	222	99.5	163.0	141.9
173	77.3	126.6	110.1	223	99.9	163.7	142.5
174	77.7	127.3	110.8	224	100.4	164.5	143.2
175	78.2	128.1	111.4	225	100.9	165.3	143.8
176	78.6	128.8	112.0	226	101.3	166.0	144.5
177	79.1	129.5	112.6	227	101.8	166.8	145.1
178	79.5	130.3	113.3	228	102.2	167.5	145.8
179	80.0	131.0	113.9	229	102.7	168.3	146.4



EFREN'S TABLE FOR THE DETERMINATION OF DEXTROSE, MALTOSE,  
AND LACTOSE—(Concluded).

Milligrams of Cupric Oxide.	Milligrams of Dextrose.	Milligrams of Maltose.	Milligrams of Lactose.	Milligrams of Cupric Oxide.	Milligrams of Dextrose.	Milligrams of Maltose.	Milligrams of Lactose.
230	103.1	169.1	147.0	280	126.1	206.8	179.6
231	103.6	169.8	147.7	281	126.5	207.5	180.2
232	104.0	170.6	148.3	282	127.0	208.3	180.9
233	104.5	171.3	149.0	283	127.4	209.0	181.5
234	105.0	172.1	149.6	284	127.9	209.8	182.2
235	105.4	172.8	150.3	285	128.3	210.5	182.9
236	105.9	173.6	150.9	286	128.8	211.3	183.6
237	106.3	174.3	151.6	287	129.3	212.1	184.2
238	106.8	175.1	152.2	288	129.7	212.8	184.9
239	107.2	175.8	152.9	289	130.2	213.6	185.6
240	107.7	176.6	153.5	290	130.6	214.3	186.2
241	108.1	177.3	154.2	291	131.1	215.1	186.9
242	108.6	178.1	154.8	292	131.5	215.9	187.6
243	109.0	178.8	155.5	293	132.0	216.6	188.2
244	109.5	179.6	156.1	294	132.5	217.4	188.9
245	109.9	180.3	156.8	295	133.0	218.2	189.5
246	110.4	181.1	157.4	296	133.4	218.9	190.2
247	110.9	181.8	158.1	297	133.9	219.7	190.8
248	111.3	182.6	158.7	298	134.3	220.4	191.5
249	111.8	183.3	159.4	299	134.8	221.2	192.1
250	112.3	184.1	160.0	300	135.3	221.9	192.8
251	112.7	184.8	160.7	301	135.7	222.7	193.4
252	113.2	185.5	161.3	302	136.2	223.5	194.1
253	113.7	186.3	162.0	303	136.6	224.2	194.7
254	114.1	187.1	162.6	304	137.1	225.0	195.3
255	114.6	187.8	163.3	305	137.6	225.8	196.0
256	115.0	188.6	163.9	306	138.0	226.5	196.6
257	115.5	189.3	164.6	307	138.5	227.3	197.3
258	116.0	190.1	165.2	308	138.9	228.1	197.9
259	116.4	190.8	165.9	309	139.4	228.8	198.6
260	116.9	191.6	166.5	310	139.9	229.6	199.3
261	117.3	192.4	167.2	311	140.3	230.4	199.9
262	117.8	193.1	167.8	312	140.8	231.1	200.6
263	118.3	193.9	168.1	313	141.2	231.9	201.3
264	118.7	194.6	169.5	314	141.7	232.7	202.0
265	119.2	195.4	169.8	315	142.2	233.4	202.6
266	119.6	196.1	170.4	316	142.6	234.2	203.3
267	120.1	196.9	171.1	317	143.1	234.9	203.9
268	120.6	197.7	171.7	318	143.6	235.7	204.6
269	121.0	198.4	172.4	319	144.0	236.5	205.3
270	121.4	199.2	173.0	320	144.5	237.2	205.9
271	121.9	199.9	173.7				
272	122.4	200.7	174.4				
273	122.8	201.5	175.0				
274	123.3	202.2	175.7				
275	123.7	203.0	176.3				
276	124.2	203.7	177.0				
277	124.6	204.5	177.6				
278	125.1	205.2	178.3				
279	125.6	206.0	178.0				



**Munson and Walker Method.\***—1. *Preparation of Solutions and Asbestos.*—Use the copper sulphate solution and alkaline tartrate solution as given on page 615. Prepare the asbestos, which should be the amphibole variety, by first digesting with 1:3 hydrochloric acid for two or three days. Wash free from acid, and digest for a similar period with soda solution, after which treat for a few hours with hot alkaline copper tartrate solution of the strength employed in sugar determinations. Then wash the asbestos free from alkali, finally digest with nitric acid for several hours, and after washing free from acid, shake with water for use. In preparing the Gooch crucible, load it with a film of asbestos one-fourth inch thick, wash this thoroughly with water to remove fine particles of asbestos; finally wash with alcohol and ether, dry for thirty minutes at 100° C., cool in a desiccator and weigh. It is best to dissolve the cuprous oxide with nitric acid each time after weighing, and use the same felts over and over again, as they improve with use.

2. *Process.*—Transfer 25 cc. each of the copper and alkaline tartrate solutions to a 400-cc. Jena or Non-sol beaker, and add 50 cc. of reducing sugar solution, or, if a smaller volume of sugar solution be used, add water to make the final volume 100 cc. Heat the beaker upon an asbestos gauze over a Bunsen burner, so regulate the flame that boiling begins in four minutes, and continue the boiling for exactly two minutes. Keep the beaker covered with a watch-glass throughout the entire time of heating. Without diluting, filter the cuprous oxide at once on an asbestos felt in a porcelain Gooch crucible, using suction. Wash the cuprous oxide thoroughly with water at a temperature of about 60° C., then with 10 cc. of alcohol, and finally with 10 cc. of ether. Dry for thirty minutes in a water oven at 100° C., cool in a desiccator and weigh as cuprous oxide.

The number of milligrams of copper reduced by a given amount of reducing sugar differs when sucrose is present and when it is absent. In the tables on pages 623 to 631 the absence of sucrose is assumed, except in the two columns under invert sugar, where one for mixtures of invert sugar and sucrose (0.4 gram of total sugar in 50 cc. of solution), and one for invert sugar and sucrose when the 50 cc. of solution contains 2 grams of total sugar are given, in addition to the column for invert sugar alone.

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\* Jour. Am. Chem. Soc., 28, 1906, p. 163; 29, 1907, p. 541; U. S. Dept. Agric., Bur. of Chem., Bul. 107 (rev.), p. 241; Circ. 82.



## MUNSON AND WALKER'S TABLE FOR CALCULATING DEXTROSE, INVERT SUGAR, LACTOSE, AND MALTOSE.

[Weights in milligrams.]

Cuprous Oxide (Cu <sub>2</sub> O).	Copper (Cu).	Dextrose.	Invert Sugar.	Invert Sugar and Sucrose.		Lactose.			Maltose.		Cuprous Oxide (Cu <sub>2</sub> O).
				0.4 Gram Total Sugar.	2 Grams Total Sugar.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> .	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + $\frac{1}{2}$ H <sub>2</sub> O.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + H <sub>2</sub> O.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> .	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + H <sub>2</sub> O.	
10	8.9	4.0	4.5	1.6	.....	3.8	3.9	4.0	5.9	6.2	10
11	9.8	4.5	5.0	2.1	.....	4.5	4.6	4.7	6.7	7.0	11
12	10.7	4.9	5.4	2.5	.....	5.1	5.3	5.4	7.5	7.9	12
13	11.5	5.3	5.8	3.0	.....	5.8	5.9	6.1	8.3	8.7	13
14	12.4	5.7	6.3	3.4	.....	6.4	6.6	6.8	9.1	9.5	14
15	13.3	6.2	6.7	3.9	.....	7.1	7.3	7.5	9.9	10.4	15
16	14.2	6.6	7.2	4.3	.....	7.8	8.0	8.2	10.6	11.2	16
17	15.1	7.0	7.6	4.8	.....	8.4	8.6	8.9	11.4	12.0	17
18	16.0	7.5	8.1	5.2	.....	9.1	9.3	9.5	12.2	12.9	18
19	16.9	7.9	8.5	5.7	.....	9.7	10.0	10.2	13.0	13.7	19
20	17.8	8.3	8.9	6.1	.....	10.4	10.7	10.9	13.8	14.6	20
21	18.7	8.7	9.4	6.6	.....	11.0	11.3	11.6	14.6	15.4	21
22	19.5	9.2	9.8	7.0	.....	11.7	12.0	12.3	15.4	16.2	22
23	20.4	9.6	10.3	7.5	.....	12.3	12.7	13.0	16.2	17.1	23
24	21.3	10.0	10.7	7.9	.....	13.0	13.4	13.7	17.0	17.9	24
25	22.2	10.5	11.2	8.4	.....	13.7	14.0	14.4	17.8	18.7	25
26	23.1	10.9	11.6	8.8	.....	14.3	14.7	15.1	18.6	19.6	26
27	24.0	11.3	12.0	9.3	.....	15.0	15.4	15.8	19.4	20.4	27
28	24.9	11.8	12.5	9.7	.....	15.6	16.1	16.5	20.2	21.2	28
29	25.8	12.2	12.9	10.2	.....	16.3	16.7	17.1	21.0	22.1	29
30	26.6	12.6	13.4	10.7	4.3	16.9	17.4	17.8	21.8	22.9	30
31	27.5	13.1	13.8	11.1	4.7	17.6	18.1	18.5	22.6	23.7	31
32	28.4	13.5	14.3	11.6	5.2	18.3	18.7	19.2	23.3	24.6	32
33	29.3	13.9	14.7	12.0	5.6	18.9	19.4	19.9	24.1	25.4	33
34	30.2	14.3	15.2	12.5	6.1	19.6	20.1	20.6	24.9	26.2	34
35	31.1	14.8	15.6	12.9	6.5	20.2	20.8	21.3	25.7	27.1	35
36	32.0	15.2	16.1	13.4	7.0	20.9	21.4	22.0	26.5	27.9	36
37	32.9	15.6	16.5	13.8	7.4	21.5	22.1	22.7	27.3	28.7	37
38	33.8	16.1	16.9	14.3	7.9	22.2	22.8	23.4	28.1	29.6	38
39	34.6	16.5	17.4	14.7	8.4	22.8	23.5	24.1	28.9	30.4	39
40	35.5	16.9	17.8	15.2	8.8	23.5	24.1	24.8	29.7	31.3	40
41	36.4	17.4	18.3	15.6	9.3	24.2	24.8	25.4	30.5	32.1	41
42	37.3	17.8	18.7	16.1	9.7	24.8	25.5	26.1	31.3	32.9	42
43	38.2	18.2	19.2	16.6	10.2	25.5	26.2	26.8	32.1	33.8	43
44	39.1	18.7	19.6	17.0	10.7	26.1	26.8	27.5	32.9	34.6	44
45	40.0	19.1	20.1	17.5	11.1	26.8	27.5	28.2	33.7	35.4	45
46	40.9	19.6	20.5	17.9	11.6	27.4	28.2	28.9	34.4	36.3	46
47	41.7	20.0	21.0	18.4	12.0	28.1	28.9	29.6	35.2	37.1	47
48	42.6	20.4	21.4	18.8	12.5	28.7	29.5	30.3	36.0	37.9	48
49	43.5	20.9	21.9	19.3	12.9	29.4	30.2	31.0	36.8	38.8	49
50	44.4	21.3	22.3	19.7	13.4	30.1	30.9	31.7	37.6	39.6	50
51	45.3	21.7	22.8	20.2	13.9	30.7	31.5	32.4	38.4	40.4	51
52	46.2	22.2	23.2	20.7	14.3	31.4	32.2	33.0	39.2	41.3	52
53	47.1	22.6	23.7	21.1	14.8	32.1	32.9	33.7	40.0	42.1	53
54	48.0	23.0	24.1	21.6	15.2	32.7	33.6	34.4	40.8	42.9	54
55	48.9	23.5	24.6	22.0	15.7	33.4	34.3	35.1	41.6	43.8	55
56	49.7	23.9	25.0	22.5	16.2	34.0	34.9	35.8	42.4	44.6	56
57	50.6	24.3	25.5	22.9	16.6	34.7	35.6	36.5	43.2	45.4	57
58	51.5	24.8	25.9	23.4	17.1	35.4	36.3	37.2	44.0	46.3	58
59	52.4	25.2	26.4	23.9	17.5	36.0	37.0	37.9	44.8	47.1	59
60	53.3	25.6	26.8	24.3	18.0	36.7	37.6	38.6	45.6	48.0	60
61	54.2	26.1	27.3	24.8	18.5	37.3	38.3	39.3	46.3	48.8	61
62	55.1	26.5	27.7	25.2	18.9	38.0	39.0	40.0	47.1	49.6	62
63	56.0	27.0	28.2	25.7	19.4	38.6	39.7	40.7	47.9	50.5	63
64	56.8	27.4	28.6	26.2	19.8	39.3	40.3	41.4	48.7	51.3	64



## MUNSON AND WALKER'S TABLE FOR CALCULATING DEXTROSE, INVERT SUGAR, LACTOSE, AND MALTOSE—(Continued).

[Weights in milligrams.]

Cuprous Oxide (Cu <sub>2</sub> O).	Copper (Cu).	Dextrose.	Invert Sugar.	Invert Sugar and Sucrose.		Lactose.			Maltose.		Cuprous Oxide (Cu <sub>2</sub> O).
				0.4 Gram Total Sugar.	2 Grams Total Sugar.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> .	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + $\frac{1}{2}$ H <sub>2</sub> O.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + H <sub>2</sub> O.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> .	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + H <sub>2</sub> O.	
65	57.7	27.8	29.1	26.6	20.3	40.0	41.0	42.1	49.5	52.1	65
66	58.6	28.3	29.5	27.1	20.8	40.6	41.7	42.8	50.3	53.0	66
67	59.5	28.7	30.0	27.5	21.2	41.3	42.4	43.5	51.1	53.8	67
68	60.4	29.2	30.4	28.0	21.7	41.9	43.1	44.2	51.9	54.6	68
69	61.3	29.6	30.9	28.5	22.2	42.6	43.7	44.8	52.7	55.5	69
70	62.2	30.0	31.3	28.9	22.6	43.3	44.4	45.5	53.5	56.3	70
71	63.1	30.5	31.8	29.4	23.1	43.9	45.1	46.2	54.3	57.1	71
72	64.0	30.9	32.3	29.8	23.5	44.6	45.8	46.9	55.1	58.0	72
73	64.8	31.4	32.7	30.3	24.0	45.2	46.4	47.6	55.9	58.8	73
74	65.7	31.8	33.2	30.8	24.5	45.9	47.1	48.3	56.7	59.6	74
75	66.6	32.2	33.6	31.2	24.9	46.6	47.8	49.0	57.5	60.5	75
76	67.5	32.7	34.1	31.7	25.4	47.2	48.5	49.7	58.2	61.3	76
77	68.4	33.1	34.5	32.1	25.9	47.9	49.1	50.4	59.0	62.1	77
78	69.3	33.6	35.0	32.6	26.3	48.5	49.8	51.1	59.8	63.0	78
79	70.2	34.0	35.4	33.1	26.8	49.2	50.5	51.8	60.6	63.8	79
80	71.1	34.4	35.9	33.5	27.3	49.9	51.2	52.5	61.4	64.6	80
81	71.9	34.9	36.3	34.0	27.7	50.5	51.9	53.2	62.2	65.5	81
82	72.8	35.3	36.8	34.5	28.2	51.2	52.5	53.9	63.0	66.3	82
83	73.7	35.8	37.3	34.9	28.6	51.8	53.2	54.6	63.8	67.1	83
84	74.6	36.2	37.7	35.4	29.1	52.5	53.9	55.3	64.6	68.0	84
85	75.5	36.7	38.2	35.8	29.6	53.1	54.6	56.0	65.4	68.8	85
86	76.4	37.1	38.6	36.3	30.0	53.8	55.2	56.6	66.2	69.7	86
87	77.3	37.5	39.1	36.8	30.5	54.5	55.9	57.3	67.0	70.5	87
88	78.2	38.0	39.5	37.2	31.0	55.1	56.6	58.0	67.8	71.3	88
89	79.1	38.4	40.0	37.7	31.4	55.8	57.3	58.7	68.5	72.2	89
90	79.9	38.9	40.4	38.2	31.9	56.4	58.0	59.4	69.3	73.0	90
91	80.8	39.3	40.9	38.6	32.4	57.1	58.6	60.1	70.1	73.8	91
92	81.7	39.8	41.4	39.1	32.8	57.8	59.3	60.8	70.9	74.7	92
93	82.6	40.2	41.8	39.6	33.3	58.4	60.0	61.5	71.7	75.5	93
94	83.5	40.6	42.3	40.0	33.8	59.1	60.7	62.2	72.5	76.3	94
95	84.4	41.1	42.7	40.5	34.2	59.7	61.3	62.9	73.3	77.2	95
96	85.3	41.5	43.2	41.0	34.7	60.4	62.0	63.6	74.1	78.0	96
97	86.2	42.0	43.7	41.4	35.2	61.1	62.7	64.3	74.9	78.8	97
98	87.1	42.4	44.1	41.9	35.6	61.7	63.4	65.0	75.7	79.7	98
99	87.9	42.9	44.6	42.3	36.1	62.4	64.1	65.7	76.5	80.5	99
100	88.8	43.3	45.0	42.8	36.6	63.0	64.7	66.4	77.3	81.3	100
101	89.7	43.8	45.5	43.3	37.0	63.7	65.4	67.1	78.1	82.2	101
102	90.6	44.2	46.0	43.8	37.5	64.4	66.1	67.8	78.8	83.0	102
103	91.5	44.7	46.4	44.2	38.0	65.0	66.7	68.5	79.6	83.8	103
104	92.4	45.1	46.9	44.7	38.5	65.7	67.4	69.1	80.4	84.7	104
105	93.3	45.5	47.3	45.2	38.9	66.4	68.1	69.8	81.2	85.5	105
106	94.2	46.0	47.8	45.6	39.4	67.0	68.8	70.5	82.0	86.3	106
107	95.0	46.4	48.3	46.1	39.9	67.7	69.5	71.2	82.8	87.2	107
108	95.9	46.9	48.7	46.6	40.3	68.3	70.1	71.9	83.6	88.0	108
109	96.8	47.3	49.2	47.0	40.8	69.0	70.8	72.6	84.4	88.8	109
110	97.7	47.8	49.6	47.5	41.3	69.7	71.5	73.3	85.2	89.7	110
111	98.6	48.2	50.1	48.0	41.7	70.3	72.2	74.0	86.0	90.5	111
112	99.5	48.7	50.6	48.4	42.2	71.0	72.8	74.7	86.8	91.3	112
113	100.4	49.1	51.0	48.9	42.7	71.6	73.5	75.4	87.6	92.2	113
114	101.3	49.6	51.5	49.4	43.2	72.3	74.2	76.1	88.4	93.0	114
115	102.2	50.0	51.9	49.8	43.6	73.0	74.9	76.8	89.2	93.9	115
116	103.0	50.5	52.4	50.3	44.1	73.6	75.6	77.5	90.0	94.7	116
117	103.9	50.9	52.9	50.8	44.6	74.3	76.2	78.2	90.7	95.5	117
118	104.8	51.4	53.3	51.2	45.0	75.0	76.9	78.9	91.5	96.4	118
119	105.7	51.8	53.8	51.7	45.5	75.6	77.6	79.6	92.3	97.2	119



## MUNSON AND WALKER'S TABLE FOR CALCULATING DEXTROSE, INVERT SUGAR, LACTOSE, AND MALTOSE—(Continued).

[Weights in milligrams.]

Cuprous Oxide (Cu <sub>2</sub> O).	Copper (Cu).	Dextrose.	Invert Sugar.	Invert Sugar and Sucrose.		Lactose.			Maltose.		Cuprous Oxide (Cu <sub>2</sub> O).
				0.4 Gram Total Sugar.	2 Grams Total Sugar.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> .	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + $\frac{1}{2}$ H <sub>2</sub> O.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + H <sub>2</sub> O.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> .	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + H <sub>2</sub> O.	
120	106.6	52.3	54.3	52.2	46.0	76.3	78.3	80.3	93.1	98.0	120
121	107.5	52.7	54.7	52.7	46.5	76.9	79.0	81.0	93.9	98.9	121
122	108.4	53.2	55.2	53.1	46.9	77.6	79.6	81.7	94.7	99.7	122
123	109.3	53.6	55.7	53.6	47.4	78.3	80.3	82.4	95.5	100.5	123
124	110.1	54.1	56.1	54.1	47.9	78.9	81.0	83.1	96.3	101.4	124
125	111.0	54.5	56.6	54.5	48.3	79.6	81.7	83.8	97.1	102.2	125
126	111.9	55.0	57.0	55.0	48.8	80.3	82.4	84.5	97.9	103.0	126
127	112.8	55.4	57.5	55.5	49.3	80.9	83.0	85.2	98.7	103.9	127
128	113.7	55.9	58.0	55.9	49.8	81.6	83.7	85.9	99.4	104.7	128
129	114.6	56.3	58.4	56.4	50.2	82.2	84.4	86.6	100.2	105.5	129
130	115.5	56.8	58.9	56.9	50.7	82.9	85.1	87.3	101.0	106.4	130
131	116.4	57.2	59.4	57.4	51.2	83.6	85.7	88.0	101.8	107.2	131
132	117.3	57.7	59.8	57.8	51.7	84.2	86.4	88.7	102.6	108.0	132
133	118.1	58.1	60.3	58.3	52.1	84.9	87.1	89.4	103.4	108.9	133
134	119.0	58.6	60.8	58.8	52.6	85.5	87.8	90.1	104.2	109.7	134
135	119.9	59.0	61.2	59.3	53.1	86.2	88.5	90.8	105.0	110.5	135
136	120.8	59.5	61.7	59.7	53.6	86.9	89.1	91.5	105.8	111.4	136
137	121.7	60.0	62.2	60.2	54.0	87.5	89.8	92.1	106.6	112.2	137
138	122.6	60.4	62.6	60.7	54.5	88.2	90.5	92.8	107.4	113.0	138
139	123.5	60.9	63.1	61.2	55.0	88.9	91.2	93.5	108.2	113.9	139
140	124.4	61.3	63.6	61.6	55.5	89.5	91.9	94.2	109.0	114.7	140
141	125.2	61.8	64.0	62.1	55.9	90.2	92.5	94.9	109.8	115.5	141
142	126.1	62.2	64.5	62.6	56.4	90.8	93.2	95.6	110.5	116.4	142
143	127.0	62.7	65.0	63.1	56.9	91.5	93.9	96.3	111.3	117.2	143
144	127.9	63.1	65.4	63.5	57.4	92.2	94.6	97.0	112.1	118.0	144
145	128.8	63.6	65.9	64.0	57.8	92.8	95.3	97.7	112.9	118.9	145
146	129.7	64.0	66.4	64.5	58.3	93.5	95.9	98.4	113.7	119.7	146
147	130.6	64.5	66.9	65.0	58.8	94.2	96.6	99.1	114.5	120.5	147
148	131.5	65.0	67.3	65.4	59.3	94.8	97.3	99.8	115.3	121.4	148
149	132.4	65.4	67.8	65.9	59.7	95.5	98.0	100.5	116.1	122.2	149
150	133.2	65.9	68.3	66.4	60.2	96.1	98.7	101.2	116.9	123.0	150
151	134.1	66.3	68.7	66.9	60.7	96.8	99.3	101.9	117.7	123.9	151
152	135.0	66.8	69.2	67.3	61.2	97.5	100.0	102.6	118.5	124.7	152
153	135.9	67.2	69.7	67.8	61.7	98.1	100.7	103.3	119.3	125.5	153
154	136.8	67.7	70.1	68.3	62.1	98.8	101.4	104.0	120.0	126.4	154
155	137.7	68.2	70.6	68.8	62.6	99.5	102.1	104.7	120.8	127.2	155
156	138.6	68.6	71.1	69.2	63.1	100.1	102.8	105.4	121.6	128.0	156
157	139.5	69.1	71.6	69.7	63.6	100.8	103.4	106.1	122.4	128.9	157
158	140.3	69.5	72.0	70.2	64.1	101.5	104.1	106.8	123.2	129.7	158
159	141.2	70.0	72.5	70.7	64.5	102.1	104.8	107.5	124.0	130.5	159
160	142.1	70.4	73.0	71.2	65.0	102.8	105.5	108.2	124.8	131.4	160
161	143.0	70.9	73.4	71.6	65.5	103.4	106.2	108.9	125.6	132.2	161
162	143.9	71.4	73.9	72.1	66.0	104.1	106.8	109.6	126.4	133.0	162
163	144.8	71.8	74.4	72.6	66.5	104.8	107.5	110.3	127.2	133.9	163
164	145.7	72.3	74.9	73.1	66.9	105.4	108.2	111.0	128.0	134.7	164
165	146.6	72.8	75.3	73.6	67.4	106.1	108.9	111.7	128.8	135.5	165
166	147.5	73.2	75.8	74.0	67.9	106.8	109.6	112.4	129.6	136.4	166
167	148.3	73.7	76.3	74.5	68.4	107.4	110.3	113.1	130.3	137.2	167
168	149.2	74.1	76.8	75.0	68.9	108.1	110.9	113.8	131.1	138.0	168
169	150.1	74.6	77.2	75.5	69.3	108.8	111.6	114.5	131.9	138.9	169
170	151.0	75.1	77.7	76.0	69.8	109.4	112.3	115.2	132.7	139.7	170
171	151.9	75.5	78.2	76.4	70.3	110.1	113.0	115.9	133.5	140.5	171
172	152.8	76.0	78.7	76.9	70.8	110.8	113.7	116.6	134.3	141.4	172
173	153.7	76.4	79.1	77.4	71.3	111.4	114.3	117.3	135.1	142.2	173
174	154.6	76.9	79.6	77.9	71.7	112.1	115.0	118.0	135.9	143.0	174



## MUNSON AND WALKER'S TABLE FOR CALCULATING DEXTROSE, INVERT SUGAR, LACTOSE, AND MALTOSE—(Continued).

[Weights in milligrams.]

Cuprous Oxide (Cu <sub>2</sub> O).	Copper (Cu).	Dextrose.	Invert Sugar.	Invert Sugar and Sucrose.		Lactose.			Maltose.		Cuprous Oxide (Cu <sub>2</sub> O).
				0.4 Gram Total Sugar.	2 Grams Total Sugar.	CuH <sub>2</sub> O <sub>11</sub> .	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + $\frac{1}{2}$ H <sub>2</sub> O.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + H <sub>2</sub> O.	CuH <sub>2</sub> O <sub>11</sub> .	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + H <sub>2</sub> O.	
175	155.5	77.4	80.1	78.4	72.2	112.8	115.7	118.7	136.7	143.9	175
176	156.3	77.8	80.6	78.8	72.7	113.4	116.4	119.4	137.5	144.7	176
177	157.2	78.3	81.0	79.3	73.2	114.1	117.1	120.1	138.3	145.5	177
178	158.1	78.8	81.5	79.8	73.7	114.8	117.8	120.8	139.1	146.4	178
179	159.0	79.2	82.0	80.3	74.2	115.4	118.4	121.5	139.8	147.2	179
180	159.9	79.7	82.5	80.8	74.6	116.1	119.1	122.2	140.6	148.0	180
181	160.8	80.1	82.9	81.3	75.1	116.7	119.8	122.9	141.4	148.9	181
182	161.7	80.6	83.4	81.7	75.6	117.4	120.5	123.6	142.2	149.7	182
183	162.6	81.1	83.9	82.2	76.1	118.1	121.2	124.3	143.0	150.5	183
184	163.4	81.5	84.4	82.7	76.6	118.7	121.8	125.0	143.8	151.4	184
185	164.3	82.0	84.9	83.2	77.1	119.4	122.5	125.7	144.6	152.2	185
186	165.2	82.5	85.3	83.7	77.6	120.1	123.2	126.4	145.4	153.0	186
187	166.1	82.9	85.8	84.2	78.0	120.7	123.9	127.1	146.2	153.9	187
188	167.0	83.4	86.3	84.6	78.5	121.4	124.6	127.8	147.0	154.7	188
189	167.9	83.9	86.8	85.1	79.0	122.1	125.3	128.5	147.8	155.5	189
190	168.8	84.3	87.2	85.6	79.5	122.7	125.9	129.2	148.6	156.4	190
191	169.7	84.8	87.7	86.1	80.0	123.4	126.6	129.9	149.3	157.2	191
192	170.5	85.3	88.2	86.6	80.5	124.1	127.3	130.6	150.1	158.0	192
193	171.4	85.7	88.7	87.1	81.0	124.7	128.0	131.3	150.9	158.9	193
194	172.3	86.2	89.2	87.6	81.4	125.4	128.7	132.0	151.7	159.7	194
195	173.2	86.7	89.6	88.0	81.9	126.1	129.4	132.7	152.5	160.5	195
196	174.1	87.1	90.1	88.5	82.4	126.7	130.0	133.4	153.3	161.4	196
197	175.0	87.6	90.6	89.0	82.9	127.4	130.7	134.1	154.1	162.2	197
198	175.9	88.1	91.1	89.5	83.4	128.1	131.4	134.8	154.9	163.0	198
199	176.8	88.5	91.6	90.0	83.9	128.7	132.1	135.5	155.7	163.9	199
200	177.7	89.0	92.0	90.5	84.4	129.4	132.8	136.2	156.5	164.7	200
201	178.5	89.5	92.5	91.0	84.8	130.0	133.5	136.9	157.3	165.5	201
202	179.4	89.9	93.0	91.4	85.3	130.7	134.1	137.6	158.1	166.4	202
203	180.3	90.4	93.5	91.9	85.8	131.4	134.8	138.3	158.8	167.2	203
204	181.2	90.9	94.0	92.4	86.3	132.0	135.5	139.0	159.6	168.0	204
205	182.1	91.4	94.5	92.9	86.8	132.7	136.2	139.7	160.4	168.9	205
206	183.0	91.8	94.9	93.4	87.3	133.4	136.9	140.4	161.2	169.7	206
207	183.9	92.3	95.4	93.9	87.8	134.0	137.6	141.1	162.0	170.5	207
208	184.8	92.8	95.9	94.4	88.3	134.7	138.3	141.8	162.8	171.4	208
209	185.6	93.2	96.4	94.9	88.8	135.4	138.9	142.5	163.6	172.2	209
210	186.5	93.7	96.9	95.4	89.2	136.0	139.6	143.2	164.4	173.0	210
211	187.4	94.2	97.4	95.8	89.7	136.7	140.3	143.9	165.2	173.8	211
212	188.3	94.6	97.8	96.3	90.2	137.4	141.0	144.6	166.0	174.7	212
213	189.2	95.1	98.3	96.8	90.7	138.0	141.7	145.3	166.8	175.5	213
214	190.1	95.6	98.8	97.3	91.2	138.7	142.4	146.0	167.5	176.4	214
215	191.0	96.1	99.3	97.8	91.7	139.4	143.0	146.7	168.3	177.2	215
216	191.9	96.5	99.8	98.3	92.2	140.0	143.7	147.4	169.1	178.0	216
217	192.8	97.0	100.3	98.8	92.7	140.7	144.4	148.1	169.9	178.9	217
218	193.6	97.5	100.8	99.3	93.2	141.4	145.1	148.8	170.7	179.7	218
219	194.5	98.0	101.2	99.8	93.7	142.0	145.8	149.5	171.5	180.5	219
220	195.4	98.4	101.7	100.3	94.2	142.7	146.5	150.2	172.3	181.4	220
221	196.3	98.9	102.2	100.8	94.7	143.4	147.2	150.9	173.1	182.2	221
222	197.2	99.4	102.7	101.2	95.1	144.0	147.8	151.6	173.9	183.0	222
223	198.1	99.9	103.2	101.7	95.6	144.7	148.5	152.3	174.7	183.9	223
224	199.0	100.3	103.7	102.2	96.1	145.4	149.2	153.0	175.5	184.7	224
225	199.9	100.8	104.2	102.7	96.6	146.0	149.9	153.7	176.2	185.5	225
226	200.7	101.3	104.6	103.2	97.1	146.7	150.6	154.4	177.0	186.4	226
227	201.6	101.8	105.1	103.7	97.6	147.4	151.3	155.1	177.8	187.2	227
228	202.5	102.2	105.6	104.2	98.1	148.0	152.0	155.8	178.6	188.0	228
229	203.4	102.7	106.1	104.7	98.6	148.7	152.6	156.5	179.4	188.8	229



## MUNSON AND WALKER'S TABLE FOR CALCULATING DEXTROSE, INVERT SUGAR, LACTOSE, AND MALTOSE—(Continued).

[Weights in milligrams.]

Cuprous Oxide (CuO).	Copper (Cu).	Dextrose.	Invert Sugar.	Invert Sugar and Sucrose		Lactose.			Maltose.		Cuprous Oxide (CuO).
				0.4 Gram Total Sugar.	2 Grams Total Sugar.	CuH <sub>2</sub> O <sub>11</sub> .	CuH <sub>2</sub> O <sub>11</sub> + $\frac{1}{2}$ H <sub>2</sub> O.	CuH <sub>2</sub> O <sub>11</sub> + H <sub>2</sub> O.	CuH <sub>2</sub> O <sub>11</sub> .	CuH <sub>2</sub> O <sub>11</sub> + H <sub>2</sub> O.	
230	204.3	103.2	106.6	105.2	99.1	149.4	153.3	157.2	180.2	189.7	230
231	205.2	103.7	107.1	105.7	99.6	150.0	154.0	157.9	181.0	190.5	231
232	206.1	104.1	107.6	106.2	100.1	150.7	154.7	158.6	181.8	191.3	232
233	207.0	104.6	108.1	106.7	100.6	151.4	155.4	159.3	182.6	192.2	233
234	207.9	105.1	108.6	107.2	101.1	152.0	156.1	160.0	183.4	193.0	234
235	208.7	105.6	109.1	107.7	101.6	152.7	156.7	160.7	184.2	193.8	235
236	209.6	106.0	109.5	108.2	102.1	153.4	157.4	161.4	184.9	194.7	236
237	210.5	106.5	110.0	108.7	102.6	154.0	158.1	162.1	185.7	195.5	237
238	211.4	107.0	110.5	109.2	103.1	154.7	158.8	162.8	186.5	196.3	238
239	212.3	107.5	111.0	109.6	103.5	155.4	159.5	163.5	187.3	197.2	239
240	213.2	108.0	111.5	110.1	104.0	156.1	160.2	164.3	188.1	198.0	240
241	214.1	108.4	112.0	110.6	104.5	156.7	160.9	165.0	188.9	198.8	241
242	215.0	108.9	112.5	111.1	105.0	157.4	161.5	165.7	189.7	199.7	242
243	215.8	109.4	113.0	111.6	105.5	158.1	162.2	166.4	190.5	200.5	243
244	216.7	109.9	113.5	112.1	106.0	158.7	162.9	167.1	191.3	201.3	244
245	217.6	110.4	114.0	112.6	106.5	159.4	163.6	167.8	192.1	202.2	245
246	218.5	110.8	114.5	113.1	107.0	160.1	164.3	168.5	192.9	203.0	246
247	219.4	111.3	115.0	113.6	107.5	160.7	165.0	169.2	193.6	203.8	247
248	220.3	111.8	115.4	114.1	108.0	161.4	165.7	169.9	194.4	204.7	248
249	221.2	112.3	115.9	114.6	108.5	162.1	166.3	170.6	195.2	205.5	249
250	222.1	112.8	116.4	115.1	109.0	162.7	167.0	171.3	196.0	206.3	250
251	223.0	113.2	116.9	115.6	109.5	163.4	167.7	172.0	196.8	207.2	251
252	223.8	113.7	117.4	116.1	110.0	164.1	168.4	172.7	197.6	208.0	252
253	224.7	114.2	117.9	116.6	110.5	164.7	169.1	173.4	198.4	208.8	253
254	225.6	114.7	118.4	117.1	111.0	165.4	169.8	174.1	199.2	209.7	254
255	226.5	115.2	118.9	117.6	111.5	166.1	170.5	174.8	200.0	210.5	255
256	227.4	115.7	119.4	118.1	112.0	166.8	171.1	175.5	200.8	211.3	256
257	228.3	116.1	119.9	118.6	112.5	167.4	171.8	176.2	201.6	212.2	257
258	229.2	116.6	120.4	119.1	113.0	168.1	172.5	176.9	202.3	213.0	258
259	230.1	117.1	120.9	119.6	113.5	168.8	173.2	177.6	203.1	213.8	259
260	231.0	117.6	121.4	120.1	114.0	169.4	173.9	178.3	203.9	214.7	260
261	231.8	118.1	121.9	120.6	114.5	170.1	174.6	179.0	204.7	215.5	261
262	232.7	118.6	122.4	121.1	115.0	170.8	175.3	179.8	205.5	216.3	262
263	233.6	119.0	122.9	121.6	115.5	171.4	176.0	180.5	206.3	217.2	263
264	234.5	119.5	123.4	122.1	116.0	172.1	176.6	181.2	207.1	218.0	264
265	235.4	120.0	123.9	122.6	116.5	172.8	177.3	181.9	207.9	218.8	265
266	236.3	120.5	124.4	123.1	117.0	173.5	178.0	182.6	208.7	219.7	266
267	237.2	121.0	124.9	123.6	117.5	174.1	178.7	183.3	209.5	220.5	267
268	238.1	121.5	125.4	124.1	118.0	174.8	179.4	184.0	210.3	221.3	268
269	238.9	122.0	125.9	124.6	118.5	175.5	180.1	184.7	211.0	222.1	269
270	239.8	122.5	126.4	125.1	119.0	176.1	180.8	185.4	211.8	223.0	270
271	240.7	122.9	126.9	125.6	119.5	176.8	181.5	186.1	212.6	223.8	271
272	241.6	123.4	127.4	126.2	120.0	177.5	182.1	186.8	213.4	224.6	272
273	242.5	123.9	127.9	126.7	120.6	178.1	182.8	187.5	214.2	225.5	273
274	243.4	124.4	128.4	127.2	121.1	178.8	183.5	188.2	215.0	226.3	274
275	244.3	124.9	128.9	127.7	121.6	179.5	184.2	188.9	215.8	227.1	275
276	245.2	125.4	129.4	128.2	122.1	180.2	184.9	189.6	216.6	228.0	276
277	246.1	125.9	129.9	128.7	122.6	180.8	185.6	190.3	217.4	228.8	277
278	246.9	126.4	130.4	129.2	123.1	181.5	186.3	191.0	218.2	229.6	278
279	247.8	126.9	130.9	129.7	123.6	182.2	187.0	191.7	218.9	230.5	279
280	248.7	127.3	131.4	130.2	124.1	182.8	187.7	192.4	219.7	231.3	280
281	249.6	127.8	131.9	130.7	124.6	183.5	188.3	193.1	220.5	232.1	281
282	250.5	128.3	132.4	131.2	125.1	184.2	189.0	193.9	221.3	233.0	282
283	251.4	128.8	132.9	131.7	125.6	184.8	189.7	194.6	222.1	233.8	283
284	252.3	129.3	133.4	132.2	126.1	185.5	190.4	195.3	222.9	234.6	284



## MUNSON AND WALKER'S TABLE FOR CALCULATING DEXTROSE, INVERT SUGAR, LACTOSE, AND MALTOSE—(Continued).

[Weights in milligrams.]

Cuprous Oxide (Cu <sub>2</sub> O).	Copper (Cu).	Dextrose.	Invert Sugar.	Invert Sugar and Sucrose.		Lactose.			Maltose.		Cuprous Oxide (Cu <sub>2</sub> O).
				0.4 Gram Total Sugar.	2 Grams Total Sugar.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> .	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + $\frac{1}{2}$ H <sub>2</sub> O.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + H <sub>2</sub> O.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> .	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + H <sub>2</sub> O.	
285	253.2	129.8	133.9	132.7	126.6	186.2	191.1	196.0	223.7	235.5	285
286	254.0	130.3	134.4	133.2	127.1	186.9	191.8	196.7	224.5	236.3	286
287	254.9	130.8	134.9	133.7	127.6	187.5	192.5	197.4	225.3	237.1	287
288	255.8	131.3	135.4	134.3	128.1	188.2	193.2	198.1	226.1	238.0	288
289	256.7	131.8	135.9	134.8	128.6	188.9	193.8	198.8	226.9	238.8	289
290	257.6	132.3	136.4	135.3	129.2	189.5	194.5	199.5	227.6	239.6	290
291	258.5	132.7	136.9	135.8	129.7	190.2	195.2	200.2	228.4	240.5	291
292	259.4	133.2	137.4	136.3	130.2	190.9	195.9	200.9	229.2	241.3	292
293	260.3	133.7	137.9	136.8	130.7	191.5	196.6	201.6	230.0	242.1	293
294	261.2	134.2	138.4	137.3	131.2	192.2	197.3	202.3	230.8	242.9	294
295	262.0	134.7	138.9	137.8	131.7	192.9	198.0	203.0	231.6	243.8	295
296	262.9	135.2	139.4	138.3	132.2	193.6	198.7	203.7	232.4	244.6	296
297	263.8	135.7	140.0	138.8	132.7	194.2	199.3	204.4	233.2	245.4	297
298	264.7	136.2	140.5	139.4	133.2	194.9	200.0	205.1	234.0	246.3	298
299	265.6	136.7	141.0	139.9	133.7	195.6	200.7	205.8	234.8	247.1	299
300	266.5	137.2	141.5	140.4	134.2	196.2	201.4	206.6	235.5	247.9	300
301	267.4	137.7	142.0	140.9	134.8	196.9	202.1	207.3	236.3	248.8	301
302	268.3	138.2	142.5	141.4	135.3	197.6	202.8	208.0	237.1	249.6	302
303	269.1	138.7	143.0	141.9	135.8	198.3	203.5	208.7	237.9	250.4	303
304	270.0	139.2	143.5	142.4	136.3	198.9	204.2	209.4	238.7	251.3	304
305	270.9	139.7	144.0	142.9	136.8	199.6	204.9	210.1	239.5	252.1	305
306	271.8	140.2	144.5	143.4	137.3	200.3	205.5	210.8	240.3	252.9	306
307	272.7	140.7	145.0	144.0	137.8	201.0	206.2	211.5	241.1	253.8	307
308	273.6	141.2	145.5	144.5	138.3	201.6	206.9	212.2	241.9	254.6	308
309	274.5	141.7	146.1	145.0	138.8	202.3	207.6	212.9	242.7	255.4	309
310	275.4	142.2	146.6	145.5	139.4	203.0	208.3	213.7	243.5	256.3	310
311	276.3	142.7	147.1	146.0	139.9	203.6	209.0	214.4	244.2	257.1	311
312	277.1	143.2	147.6	146.5	140.4	204.3	209.7	215.1	245.0	257.9	312
313	278.0	143.7	148.1	147.0	140.9	205.0	210.4	215.8	245.8	258.8	313
314	278.9	144.2	148.6	147.6	141.4	205.7	211.1	216.5	246.6	259.6	314
315	279.8	144.7	149.1	148.1	141.9	206.3	211.8	217.2	247.4	260.4	315
316	280.7	145.2	149.6	148.6	142.4	207.0	212.5	217.9	248.2	261.2	316
317	281.6	145.7	150.1	149.1	143.0	207.7	213.1	218.6	249.0	262.1	317
318	282.5	146.2	150.7	149.6	143.5	208.4	213.8	219.3	249.8	262.9	318
319	283.4	146.7	151.2	150.1	144.0	209.0	214.5	220.0	250.6	263.7	319
320	284.2	147.2	151.7	150.7	144.5	209.7	215.2	220.7	251.3	264.6	320
321	285.1	147.7	152.2	151.2	145.0	210.4	215.9	221.4	252.1	265.4	321
322	286.0	148.2	152.7	151.7	145.5	211.0	216.6	222.2	252.9	266.2	322
323	286.9	148.7	153.2	152.2	146.0	211.7	217.3	222.9	253.7	267.1	323
324	287.8	149.2	153.7	152.7	146.6	212.4	218.0	223.6	254.5	267.9	324
325	288.7	149.7	154.3	153.2	147.1	213.1	218.7	224.3	255.3	268.7	325
326	289.6	150.2	154.8	153.8	147.6	213.7	219.4	225.0	256.1	269.6	326
327	290.5	150.7	155.3	154.3	148.1	214.4	220.1	225.7	256.9	270.4	327
328	291.4	151.2	155.8	154.8	148.6	215.1	220.7	226.4	257.7	271.2	328
329	292.2	151.7	156.3	155.3	149.1	215.8	221.4	227.1	258.5	272.1	329
330	293.1	152.2	156.8	155.8	149.7	216.4	222.1	227.8	259.3	272.9	330
331	294.0	152.7	157.3	156.4	150.2	217.1	222.8	228.5	260.0	273.7	331
332	294.9	153.2	157.9	156.9	150.7	217.8	223.5	229.2	260.8	274.6	332
333	295.8	153.7	158.4	157.4	151.2	218.4	224.2	230.0	261.6	275.4	333
334	296.7	154.2	158.9	157.9	151.7	219.1	224.9	230.7	262.4	276.2	334
335	297.6	154.7	159.4	158.4	152.3	219.8	225.6	231.4	263.2	277.0	335
336	298.5	155.2	159.9	159.0	152.8	220.5	226.3	232.1	264.0	277.9	336
337	299.3	155.8	160.5	159.5	153.3	221.1	227.0	232.8	264.8	278.7	337
338	300.2	156.3	161.0	160.0	153.8	221.8	227.7	233.5	265.6	279.5	338
339	301.1	156.8	161.5	160.5	154.3	222.5	228.3	234.2	266.4	280.4	339



## MUNSON AND WALKER'S TABLE FOR CALCULATING DEXTROSE, INVERT SUGAR, LACTOSE, AND MALTOSE—(Continued).

[Weights in milligrams.]

Cuprous Oxide (Cu <sub>2</sub> O).	Copper (Cu).	Dextrose.	Invert Sugar.	Invert Sugar and Sucrose.		Lactose.			Maltose.		Cuprous Oxide (Cu <sub>2</sub> O).
				0.4 Gram Total Sugar.	2 Grams Total Sugar.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> .	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + $\frac{1}{2}$ H <sub>2</sub> O.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + H <sub>2</sub> O.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> .	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + H <sub>2</sub> O.	
340	302.0	157.3	162.0	161.0	154.8	223.2	229.0	234.9	267.1	281.2	340
341	302.9	157.8	162.5	161.6	155.4	223.8	229.7	235.6	267.9	282.0	341
342	303.8	158.3	163.1	162.1	155.9	224.5	230.4	236.3	268.7	282.9	342
343	304.7	158.8	163.6	162.6	156.4	225.2	231.1	237.0	269.5	283.7	343
344	305.6	159.3	164.1	163.1	156.9	225.9	231.8	237.8	270.3	284.5	344
345	306.5	159.8	164.6	163.7	157.5	226.5	232.5	238.5	271.1	285.4	345
346	307.3	160.3	165.1	164.2	158.0	227.2	233.2	239.2	271.9	286.2	346
347	308.2	160.8	165.7	164.7	158.5	227.9	233.9	239.9	272.7	287.0	347
348	309.1	161.4	166.2	165.2	159.0	228.5	234.6	240.6	273.5	287.9	348
349	310.0	161.9	166.7	165.7	159.5	229.2	235.3	241.3	274.3	288.7	349
350	310.9	162.4	167.2	166.3	160.1	229.9	235.9	242.0	275.0	289.5	350
351	311.8	162.9	167.7	166.8	160.6	230.6	236.6	242.7	275.8	290.4	351
352	312.7	163.4	168.3	167.3	161.1	231.2	237.3	243.4	276.6	291.2	352
353	313.6	163.9	168.8	167.8	161.6	231.9	238.0	244.1	277.4	292.0	353
354	314.4	164.4	169.3	168.4	162.2	232.6	238.7	244.8	278.2	292.8	354
355	315.3	164.9	169.8	168.9	162.7	233.3	239.4	245.6	279.0	293.7	355
356	316.2	165.4	170.4	169.4	163.2	233.9	240.1	246.3	279.8	294.5	356
357	317.1	166.0	170.9	170.0	163.7	234.6	240.8	247.0	280.6	295.3	357
358	318.0	166.5	171.4	170.5	164.3	235.3	241.5	247.7	281.4	296.2	358
359	318.9	167.0	171.9	171.0	164.8	236.0	242.2	248.4	282.2	297.0	359
360	319.8	167.5	172.5	171.5	165.3	236.7	242.9	249.1	282.9	297.8	360
361	320.7	168.0	173.0	172.1	165.8	237.3	243.6	249.8	283.7	298.7	361
362	321.6	168.5	173.5	172.6	166.4	238.0	244.3	250.5	284.5	299.5	362
363	322.4	169.0	174.0	173.1	166.9	238.7	245.0	251.2	285.3	300.3	363
364	323.3	169.6	174.6	173.7	167.4	239.4	245.7	252.0	286.1	301.2	364
365	324.2	170.1	175.1	174.2	167.9	240.0	246.4	252.7	286.9	302.0	365
366	325.1	170.6	175.6	174.7	168.5	240.7	247.0	253.4	287.7	302.8	366
367	326.0	171.1	176.1	175.2	169.0	241.4	247.7	254.1	288.5	303.6	367
368	326.9	171.6	176.7	175.8	169.5	242.1	248.4	254.8	289.3	304.5	368
369	327.8	172.1	177.2	176.3	170.0	242.7	249.1	255.5	290.0	305.3	369
370	328.7	172.7	177.7	176.8	170.6	243.4	249.8	256.2	290.8	306.1	370
371	329.5	173.2	178.3	177.4	171.1	244.1	250.5	256.9	291.6	307.0	371
372	330.4	173.7	178.8	177.9	171.6	244.8	251.2	257.7	292.4	307.8	372
373	331.3	174.2	179.3	178.4	172.2	245.4	251.9	258.4	293.2	308.6	373
374	332.2	174.7	179.8	179.0	172.7	246.1	252.6	259.1	294.0	309.5	374
375	333.1	175.3	180.4	179.5	173.2	246.8	253.3	259.8	294.8	310.3	375
376	334.0	175.8	180.9	180.0	173.7	247.5	254.0	260.5	295.6	311.1	376
377	334.9	176.3	181.4	180.6	174.3	248.1	254.7	261.2	296.4	312.0	377
378	335.8	176.8	182.0	181.1	174.8	248.8	255.4	261.9	297.2	312.8	378
379	336.7	177.3	182.5	181.6	175.3	249.5	256.1	262.6	297.9	313.6	379
380	337.5	177.9	183.0	182.1	175.9	250.2	256.8	263.4	298.7	314.5	380
381	338.4	178.4	183.6	182.7	176.4	250.8	257.5	264.1	299.5	315.3	381
382	339.3	178.9	184.1	183.2	176.9	251.5	258.1	264.8	300.3	316.1	382
383	340.2	179.4	184.6	183.8	177.5	252.2	258.8	265.5	301.1	316.9	383
384	341.1	180.0	185.2	184.3	178.0	252.9	259.5	266.2	301.9	317.8	384
385	342.0	180.5	185.7	184.8	178.5	253.6	260.2	266.9	302.7	318.6	385
386	342.9	181.0	186.2	185.4	179.1	254.2	260.9	267.6	303.5	319.4	386
387	343.8	181.5	186.8	185.9	179.6	254.9	261.6	268.3	304.2	320.3	387
388	344.6	182.0	187.3	186.4	180.1	255.6	262.3	269.0	305.0	321.1	388
389	345.5	182.6	187.8	187.0	180.6	256.3	263.0	269.8	305.8	321.9	389
390	346.4	183.1	188.4	187.5	181.2	256.9	263.7	270.5	306.6	322.8	390
391	347.3	183.6	188.9	188.0	181.7	257.6	264.4	271.2	307.4	323.6	391
392	348.2	184.1	189.4	188.6	182.3	258.3	265.1	271.9	308.2	324.4	392
393	349.1	184.7	190.0	189.1	182.8	259.0	265.8	272.6	309.0	325.2	393
394	350.0	185.2	190.5	189.7	183.3	259.6	266.5	273.3	309.8	326.1	394



## MUNSON AND WALKER'S TABLE FOR CALCULATING DEXTROSE, INVERT SUGAR, LACTOSE, AND MALTOSE—(Continued).

[Weights in milligrams.]

Cuprous Oxide (Cu <sub>2</sub> O).	Copper (Cu).	Dextrose.	Invert Sugar.	Invert Sugar and Sucrose.		Lactose.			Maltose.		Cuprous Oxide (Cu <sub>2</sub> O).
				0.4 Gram Total Sugar.	2 Grams Total Sugar.	CuH <sub>2</sub> O <sub>11</sub> .	CuH <sub>2</sub> O <sub>11</sub> + $\frac{1}{2}$ H <sub>2</sub> O.	CuH <sub>2</sub> O <sub>11</sub> + H <sub>2</sub> O.	CuH <sub>2</sub> O <sub>11</sub> .	CuH <sub>2</sub> O <sub>11</sub> + H <sub>2</sub> O.	
395	350.9	185.7	191.0	190.2	183.9	260.3	267.2	274.0	310.6	326.9	395
396	351.8	186.2	191.6	190.7	184.4	261.0	267.9	274.7	311.4	327.7	396
397	352.6	186.8	192.1	191.3	184.9	261.7	268.6	275.5	312.1	328.6	397
398	353.5	187.3	192.7	191.8	185.5	262.3	269.3	276.2	312.9	329.4	398
399	354.4	187.8	193.2	192.3	186.0	263.0	269.9	276.9	313.7	330.2	399
400	355.3	188.4	193.7	192.9	186.5	263.7	270.6	277.6	314.5	331.1	400
401	356.2	188.9	194.3	193.4	187.1	264.4	271.3	278.3	315.3	331.9	401
402	357.1	189.4	194.8	194.0	187.6	265.0	272.0	279.0	316.1	332.7	402
403	358.0	189.9	195.4	194.5	188.1	265.7	272.7	279.7	316.9	333.6	403
404	358.9	190.5	195.9	195.0	188.7	266.4	273.4	280.4	317.7	334.4	404
405	359.7	191.0	196.4	195.6	189.2	267.1	274.1	281.1	318.5	335.2	405
406	360.6	191.5	197.0	196.1	189.8	267.8	274.8	281.9	319.2	336.0	406
407	361.5	192.1	197.5	196.7	190.3	268.4	275.5	282.6	320.0	336.9	407
408	362.4	192.6	198.1	197.2	190.8	269.1	276.2	283.3	320.8	337.7	408
409	363.3	193.1	198.6	197.7	191.4	269.8	276.9	284.0	321.6	338.5	409
410	364.2	193.7	199.1	198.3	191.9	270.5	277.6	284.7	322.4	339.4	410
411	365.1	194.2	199.7	198.8	192.5	271.2	278.3	285.4	323.2	340.2	411
412	366.0	194.7	200.2	199.4	193.0	271.8	279.0	286.2	324.0	341.0	412
413	366.9	195.2	200.8	199.9	193.5	272.5	279.7	286.9	324.8	341.9	413
414	367.7	195.8	201.3	200.5	194.1	273.2	280.4	287.6	325.6	342.7	414
415	368.6	196.3	201.8	201.0	194.6	273.9	281.1	288.3	326.3	343.5	415
416	369.5	196.8	202.4	201.6	195.2	274.6	281.8	289.0	327.1	344.4	416
417	370.4	197.4	202.9	202.1	195.7	275.2	282.5	289.7	327.9	345.2	417
418	371.3	197.9	203.5	202.6	196.2	275.9	283.2	290.4	328.7	346.0	418
419	372.2	198.4	204.0	203.2	196.8	276.6	283.9	291.2	329.5	346.8	419
420	373.1	199.0	204.6	203.7	197.3	277.3	284.6	291.9	330.3	347.7	420
421	374.0	199.5	205.1	204.3	197.9	277.9	285.3	292.6	331.1	348.5	421
422	374.8	200.1	205.7	204.8	198.4	278.6	286.0	293.3	331.9	349.3	422
423	375.7	200.6	206.2	205.4	198.9	279.3	286.7	294.0	332.7	350.2	423
424	376.6	201.1	206.7	205.9	199.5	280.0	287.4	294.7	333.4	351.0	424
425	377.5	201.7	207.3	206.5	200.0	280.7	288.1	295.4	334.2	351.8	425
426	378.4	202.2	207.8	207.0	200.6	281.3	288.8	296.2	335.0	352.7	426
427	379.3	202.8	208.4	207.6	201.1	282.0	289.4	296.9	335.8	353.5	427
428	380.2	203.3	208.9	208.1	201.7	282.7	290.1	297.6	336.6	354.3	428
429	381.1	203.8	209.5	208.7	202.2	283.4	290.8	298.3	337.4	355.1	429
430	382.0	204.4	210.0	209.2	202.7	284.1	291.5	299.0	338.2	356.0	430
431	382.8	204.9	210.6	209.8	203.3	284.7	292.2	299.7	339.0	356.8	431
432	383.7	205.5	211.1	210.3	203.8	285.4	292.9	300.5	339.7	357.6	432
433	384.6	206.0	211.7	210.9	204.4	286.1	293.6	301.2	340.5	358.5	433
434	385.5	206.5	212.2	211.4	204.9	286.8	294.3	301.9	341.3	359.3	434
435	386.4	207.1	212.8	212.0	205.5	287.5	295.0	302.6	342.1	360.1	435
436	387.3	207.6	213.3	212.5	206.0	288.1	295.7	303.3	342.9	361.0	436
437	388.2	208.2	213.9	213.1	206.6	288.8	296.4	304.0	343.7	361.8	437
438	389.1	208.7	214.4	213.6	207.1	289.5	297.1	304.7	344.5	362.6	438
439	390.0	209.2	215.0	214.2	207.7	290.2	297.8	305.5	345.3	363.4	439
440	390.8	209.8	215.5	214.7	208.2	290.9	298.5	306.2	346.1	364.3	440
441	391.7	210.3	216.1	215.3	208.8	291.5	299.2	306.9	346.8	365.1	441
442	392.6	210.9	216.6	215.8	209.3	292.2	299.9	307.6	347.6	365.9	442
443	393.5	211.4	217.2	216.4	209.9	292.9	300.6	308.3	348.4	366.8	443
444	394.4	212.0	217.8	216.9	210.4	293.6	301.3	309.0	349.2	367.6	444
445	395.3	212.5	218.3	217.5	211.0	294.2	302.0	309.7	350.0	368.4	445
446	396.2	213.1	218.9	218.0	211.5	294.9	302.7	310.5	350.8	369.3	446
447	397.1	213.6	219.4	218.6	212.1	295.6	303.4	311.2	351.6	370.1	447
448	397.9	214.1	220.0	219.1	212.6	296.3	304.1	311.9	352.4	370.9	448
449	398.8	214.7	220.5	219.7	213.2	297.0	304.8	312.6	353.2	371.7	449



## MUNSON AND WALKER'S TABLE FOR CALCULATING DEXTROSE, INVERT SUGAR, LACTOSE, AND MALTOSE—(Continued).

[Weights in milligrams.]

Cuprous Oxide (Cu <sub>2</sub> O).	Copper (Cu).	Dextrose.	Invert Sugar.	Invert Sugar and Sucrose.		Lactose.			Maltose.		Cuprous Oxide (Cu <sub>2</sub> O).
				0.4 Gram Total Sugar.	2 Grams Total Sugar.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> .	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + $\frac{1}{2}$ H <sub>2</sub> O.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + H <sub>2</sub> O.	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> .	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub> + H <sub>2</sub> O.	
450	399.7	215.2	221.1	220.2	213.7	297.6	305.5	313.3	353.9	372.6	450
451	400.6	215.8	221.6	220.8	214.3	298.3	306.2	314.0	354.7	373.4	451
452	401.5	216.3	222.2	221.4	214.8	299.0	306.9	314.7	355.5	374.2	452
453	402.4	216.9	222.8	221.9	215.4	299.7	307.6	315.5	356.3	375.1	453
454	403.3	217.4	223.3	222.5	215.9	300.4	308.3	316.2	357.1	375.9	454
455	404.2	218.0	223.9	223.0	216.5	301.1	309.0	316.9	357.9	376.7	455
456	405.1	218.5	224.4	223.6	217.0	301.7	309.7	317.6	358.7	377.6	456
457	405.9	219.1	225.0	224.1	217.6	302.4	310.4	318.3	359.5	378.4	457
458	406.8	219.6	225.5	224.7	218.1	303.1	311.1	319.0	360.3	379.2	458
459	407.7	220.2	226.1	225.3	218.7	303.8	311.8	319.8	361.0	380.0	459
460	408.6	220.7	226.7	225.8	219.2	304.5	312.5	320.5	361.8	380.9	460
461	409.5	221.3	227.2	226.4	219.8	305.1	313.2	321.2	362.6	381.7	461
462	410.4	221.8	227.8	226.9	220.3	305.8	313.9	321.9	363.4	382.5	462
463	411.3	222.4	228.3	227.5	220.9	306.5	314.6	322.6	364.2	383.4	463
464	412.2	222.9	228.9	228.1	221.4	307.2	315.3	323.4	365.0	384.2	464
465	413.0	223.5	229.5	228.6	222.0	307.9	316.0	324.1	365.8	385.0	465
466	413.9	224.0	230.0	229.2	222.5	308.6	316.7	324.8	366.6	385.9	466
467	414.8	224.6	230.6	229.7	223.1	309.2	317.4	325.5	367.3	386.7	467
468	415.7	225.1	231.2	230.3	223.7	309.9	318.1	326.2	368.1	387.5	468
469	416.6	225.7	231.7	230.9	224.2	310.6	318.8	326.9	368.9	388.3	469
470	417.5	226.2	232.3	231.4	224.8	311.3	319.5	327.7	369.7	389.2	470
471	418.4	226.8	232.8	232.0	225.3	312.0	320.2	328.4	370.5	390.0	471
472	419.3	227.4	233.4	232.5	225.9	312.6	320.9	329.1	371.3	390.8	472
473	420.2	227.9	234.0	233.1	226.4	313.3	321.6	329.8	372.1	391.7	473
474	421.0	228.5	234.5	233.7	227.0	314.0	322.3	330.5	372.9	392.5	474
475	421.9	229.0	235.1	234.2	227.6	314.7	323.0	331.3	373.7	393.3	475
476	422.8	229.6	235.7	234.8	228.1	315.4	323.7	332.0	374.4	394.2	476
477	423.7	230.1	236.2	235.4	228.7	316.1	324.4	332.7	375.2	395.0	477
478	424.6	230.7	236.8	235.9	229.2	316.7	325.1	333.4	376.0	395.8	478
479	425.5	231.3	237.4	236.5	229.8	317.4	325.8	334.1	376.8	396.6	479
480	426.4	231.8	237.9	237.1	230.3	318.1	326.5	334.8	377.6	397.5	480
481	427.3	232.4	238.5	237.6	230.9	318.8	327.2	335.6	378.4	398.3	481
482	428.1	232.9	239.1	238.2	231.5	319.5	327.9	336.3	379.2	399.1	482
483	429.0	233.5	239.6	238.8	232.0	320.1	328.6	337.0	380.0	400.0	483
484	429.9	234.1	240.2	239.3	232.6	320.8	329.3	337.7	380.7	400.8	484
485	430.8	234.6	240.8	239.9	233.2	321.5	330.0	338.4	381.5	401.6	485
486	431.7	235.2	241.4	240.5	233.7	322.2	330.7	339.1	382.3	402.4	486
487	432.6	235.7	241.9	241.0	234.3	322.9	331.4	339.9	383.1	403.3	487
488	433.5	236.3	242.5	241.6	234.8	323.6	332.1	340.6	383.9	404.1	488
489	434.4	236.9	243.1	242.2	235.4	324.2	332.8	341.3	384.7	404.9	489
490	435.3	237.4	243.6	242.7	236.0	324.9	333.5	342.0	385.5	405.8	490



**Allihn's Method for the Determination of Dextrose.\***—The solutions used are those described on page 615, except that 125 grams of potassium hydroxide are used in place of 50 grams of sodium hydroxide in preparing the alkaline tartrate solution. Place 30 cc. of Fehling's copper solution, 30 cc. of the alkaline tartrate solution, and 60 cc. of water in a beaker and heat to boiling. Add 25 cc. of the sugar solution, which must be so prepared as not to contain more than 1% dextrose, and boil over the flame for two minutes. Filter immediately without diluting through a Gooch crucible containing a layer of asbestos fiber, prepared as described on page 618, and wash thoroughly with hot water, using reduced pressure. Transfer the asbestos fiber and the adhering cuprous oxide by means of a glass rod to a beaker and rinse the crucible with about 30 cc. of a boiling mixture of dilute sulphuric and nitric acids containing 65 cc. of sulphuric acid (specific gravity 1.84) and 50 cc. of nitric acid (specific gravity 1.42) per liter. Heat and agitate till the solution is complete, then filter into a scrupulously clean, tared platinum dish of 100-cc. capacity, taking care to wash out all the copper solution from the filter into the dish. Deposit the copper electrolytically in the platinum dish and weigh. Determine the dextrose from Allihn's table, pages 633-634.

Or, the metallic copper may be calculated by means of the factor 0.7989 from the cupric oxide obtained as in Defren's method (page 618) and Allihn's table used.

Or, the cuprous oxide as directly obtained by either Allihn's or Defren's method may be washed with alcohol and ether, dried for twenty minutes at 100° C., and weighed, its equivalent in dextrose being ascertained from Allihn's table.

*Browne's Correction Formula,†* for use when the Allihn method is carried out on samples containing a considerable amount of sucrose, is as follows:

$$C = \frac{S}{D + 40},$$

in which  $C$  = correction in milligrams to be deducted from dextrose found,  $S$  = milligrams of sucrose, and  $D$  = milligrams of dextrose.

He found that the reducing action of sucrose is proportional (1) to the concentration of the sucrose and (2) to the amount of unreduced copper. In the volumetric methods and in the gravimetric methods when the amount

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\* Jour. prak. Chem., 22, 1880, p. 46.

† Jour. Amer. Chem. Soc., 1906, p. 451.



ALLIHN'S TABLE FOR THE DETERMINATION OF DEXTROSE.

Milli-grams of Cop-per.	Milli-grams of Cu-prous Oxide.	Milli-grams of Dex-trose.	Milli-grams of Cop-per.	Milli-grams of Cu-prous Oxide.	Milli-grams of Dex-trose.	Milli-grams of Cop-per.	Milli-grams of Cu-prous Oxide.	Milli-grams of Dex-trose.	Milli-grams of Cop-per.	Milli-grams of Cu-prous Oxide.	Milli-grams of Dex-trose.
11	12.4	6.6	76	85.6	38.8	141	158.7	71.8	206	231.9	105.8
12	13.5	7.1	77	86.7	39.3	142	159.9	72.3	207	233.0	106.3
13	14.6	7.6	78	87.8	39.8	143	161.0	72.9	208	234.2	106.8
14	15.8	8.1	79	88.9	40.3	144	162.1	73.4	209	235.3	107.4
15	16.9	8.6	80	90.1	40.8	145	163.2	73.9	210	236.4	107.9
16	18.0	9.0	81	91.2	41.3	146	164.4	74.4	211	237.6	108.4
17	19.1	9.5	82	92.3	41.8	147	165.5	74.9	212	238.7	109.0
18	20.3	10.0	83	93.4	42.3	148	166.6	75.5	213	239.8	109.5
19	21.4	10.5	84	94.6	42.8	149	167.7	76.0	214	240.9	110.0
20	22.5	11.0	85	95.7	43.4	150	168.9	76.5	215	242.1	110.6
21	23.6	11.5	86	96.8	43.9	151	170.0	77.0	216	243.2	111.1
22	24.8	12.0	87	97.9	44.4	152	171.1	77.5	217	244.3	111.6
23	25.9	12.5	88	99.1	44.9	153	172.3	78.1	218	245.4	112.1
24	27.0	13.0	89	100.2	45.4	154	173.4	78.6	219	246.6	112.7
25	28.1	13.5	90	101.3	45.9	155	174.5	79.1	220	247.7	113.2
26	29.3	14.0	91	102.4	46.4	156	175.6	79.6	221	248.7	113.7
27	30.4	14.5	92	103.6	46.9	157	176.8	80.1	222	249.9	114.3
28	31.5	15.0	93	104.7	47.4	158	177.9	80.7	223	251.0	114.8
29	32.7	15.5	94	105.8	47.9	159	179.0	81.2	224	252.4	115.3
30	33.8	16.0	95	107.0	48.4	160	180.1	81.7	225	253.3	115.9
31	34.9	16.5	96	108.1	48.9	161	181.3	82.2	226	254.4	116.4
32	36.0	17.0	97	109.2	49.4	162	182.4	82.7	227	255.6	116.9
33	37.2	17.5	98	110.3	49.9	163	183.5	83.3	228	256.7	117.4
34	38.3	18.0	99	111.5	50.4	164	184.6	83.8	229	257.8	118.0
35	39.4	18.5	100	112.6	50.9	165	185.8	84.3	230	258.9	118.5
36	40.5	18.9	101	113.7	51.4	166	186.9	84.8	231	260.1	119.0
37	41.7	19.4	102	114.8	51.9	167	188.0	85.3	232	261.2	119.6
38	42.8	19.9	103	116.0	52.4	168	189.1	85.9	233	262.3	120.1
39	43.9	20.4	104	117.1	52.9	169	190.3	86.4	234	263.4	120.7
40	45.0	20.9	105	118.2	53.5	170	191.4	86.9	235	264.6	121.2
41	46.2	21.4	106	119.3	54.0	171	192.5	87.4	236	265.7	121.7
42	47.3	21.9	107	120.5	54.5	172	193.6	87.9	237	266.8	122.3
43	48.4	22.4	108	121.6	55.0	173	194.8	88.5	238	268.0	122.8
44	49.5	22.9	109	122.7	55.5	174	195.9	89.0	239	269.1	123.4
45	50.7	23.4	110	123.8	56.0	175	197.0	89.5	240	270.2	123.9
46	51.8	23.9	111	125.0	56.5	176	198.1	90.0	241	271.3	124.4
47	52.9	24.4	112	126.1	57.0	177	199.3	90.5	242	272.5	125.0
48	54.0	24.9	113	127.2	57.5	178	200.4	91.1	243	273.6	125.5
49	55.2	25.4	114	128.3	58.0	179	201.5	91.6	244	274.7	126.0
50	56.3	25.9	115	129.6	58.6	180	202.6	92.1	245	275.8	126.6
51	57.4	26.4	116	130.6	59.1	181	203.8	92.6	246	277.0	127.1
52	58.5	26.9	117	131.7	59.6	182	204.9	93.1	247	278.1	127.6
53	59.7	27.4	118	132.8	60.1	183	206.0	93.7	248	279.2	128.1
54	60.8	27.9	119	134.0	60.6	184	207.1	94.2	249	280.3	128.7
55	61.9	28.4	120	135.1	61.1	185	208.3	94.7	250	281.5	129.2
56	63.0	28.8	121	136.2	61.6	186	209.4	95.2	251	282.6	129.7
57	64.2	29.3	122	137.4	62.1	187	210.5	95.7	252	283.7	130.3
58	65.3	29.8	123	138.5	62.6	188	211.7	96.3	253	284.8	130.8
59	66.4	30.3	124	139.6	63.1	189	212.8	96.8	254	286.0	131.4
60	67.6	30.8	125	140.7	63.7	190	213.9	97.3	255	287.1	131.9
61	68.7	31.3	126	141.9	64.2	191	215.0	97.8	256	288.2	132.4
62	69.8	31.8	127	143.0	64.7	192	216.2	98.4	257	289.3	133.0
63	70.9	32.3	128	144.1	65.2	193	217.3	98.9	258	290.5	133.5
64	72.1	32.8	129	145.2	65.7	194	218.4	99.4	259	291.6	134.1
65	73.2	33.3	130	146.4	66.2	195	219.5	100.0	260	292.7	134.6
66	74.3	33.8	131	147.5	66.7	196	220.7	100.5	261	293.8	135.1
67	75.4	34.3	132	148.6	67.2	197	221.8	101.0	262	295.0	135.7
68	76.6	34.8	133	149.7	67.7	198	222.9	101.5	263	296.1	136.2
69	77.7	35.3	134	150.9	68.2	199	224.0	102.0	264	297.2	136.8
70	78.8	35.8	135	152.0	68.8	200	225.2	102.6	265	298.3	137.3
71	79.9	36.3	136	153.1	69.3	201	226.3	103.1	266	299.5	137.8
72	81.1	36.8	137	154.2	69.8	202	227.4	103.7	267	300.6	138.4
73	82.2	37.3	138	155.4	70.3	203	228.5	104.2	268	301.7	138.9
74	83.3	37.8	139	156.5	70.8	204	229.7	104.7	269	302.8	139.5
75	84.4	38.3	140	157.6	71.3	205	230.8	105.3	270	304.0	140.0



ALLIHN'S TABLE FOR THE DETERMINATION OF DEXTROSE—(Continued).

Milli-grams of Copper.	Milli-grams of Cuprous Oxide.	Milli-grams of Dextrose.	Milli-grams of Copper.	Milli-grams of Cuprous Oxide.	Milli-grams of Dextrose.	Milli-grams of Copper.	Milli-grams of Cuprous Oxide.	Milli-grams of Dextrose.	Milli-grams of Copper.	Milli-grams of Cuprous Oxide.	Milli-grams of Dextrose.
271	305.1	140.6	321	361.4	168.1	371	417.7	196.3	421	474.0	225.1
272	306.2	141.1	322	362.5	168.6	372	418.8	196.8	422	475.6	225.7
273	307.3	141.7	323	363.7	169.2	373	420.0	197.4	423	476.2	226.3
274	308.5	142.2	324	364.8	169.7	374	421.1	198.0	424	477.4	226.9
275	309.6	142.8	325	365.9	170.3	375	422.2	198.6	425	478.5	227.5
276	310.7	143.3	326	367.0	170.9	376	423.3	199.1	426	479.6	228.0
277	311.9	143.9	327	368.2	171.4	377	424.5	199.7	427	480.7	228.6
278	313.0	144.4	328	369.3	172.0	378	425.6	200.3	428	481.9	229.2
279	314.1	145.0	329	370.4	172.5	379	426.7	200.8	429	483.0	229.8
280	315.2	145.5	330	371.5	173.1	380	427.8	201.4	430	484.1	230.4
281	316.4	146.1	331	372.7	173.7	381	429.0	202.0	431	485.3	231.0
282	317.5	146.6	332	373.8	174.2	382	430.1	202.5	432	486.4	231.6
283	318.6	147.2	333	374.9	174.8	383	431.2	203.1	433	487.5	232.2
284	319.7	147.7	334	376.0	175.3	384	432.3	203.7	434	488.6	232.8
285	320.9	148.3	335	377.2	175.9	385	433.5	204.3	435	489.7	233.4
286	322.0	148.8	336	378.3	176.5	386	434.6	204.8	436	490.9	233.9
287	323.1	149.4	337	379.4	177.0	387	435.7	205.4	437	492.0	234.5
288	324.2	149.9	338	380.5	177.6	388	436.8	206.0	438	493.1	235.1
289	325.4	150.5	339	381.7	178.1	389	438.0	206.5	439	494.3	235.7
290	326.5	151.0	340	382.8	178.7	390	439.1	207.1	440	495.4	236.3
291	327.4	151.6	341	383.9	179.3	391	440.2	207.7	441	496.5	236.9
292	328.7	152.1	342	385.0	179.8	392	441.3	208.3	442	497.6	237.5
293	329.9	152.7	343	386.2	180.4	393	442.4	208.8	443	498.8	238.1
294	331.0	153.2	344	387.3	180.9	394	443.6	209.4	444	499.9	238.7
295	332.1	153.8	345	388.4	181.5	395	444.7	210.0	445	501.0	239.3
296	333.3	154.3	346	389.6	182.1	396	445.9	210.6	446	502.1	239.8
297	334.4	154.9	347	390.7	182.6	397	447.0	211.2	447	503.2	240.4
298	335.5	155.4	348	391.8	183.2	398	448.1	211.7	448	504.4	241.0
299	336.6	156.0	349	392.9	183.7	399	449.2	212.3	449	505.5	241.6
300	337.8	156.5	350	394.0	184.3	400	450.3	212.9	450	506.6	242.2
301	338.9	157.1	351	395.2	184.9	401	451.5	213.5	451	507.8	242.8
302	340.0	157.6	352	396.3	185.4	402	452.6	214.1	452	508.9	243.4
303	341.1	158.2	353	397.4	186.0	403	453.7	214.6	453	510.0	244.0
304	342.3	158.7	354	398.6	186.6	404	454.8	215.2	454	511.1	244.6
305	343.4	159.3	355	399.7	187.2	405	456.0	215.8	455	512.3	245.2
306	344.5	159.8	356	400.8	187.7	406	457.1	216.4	456	513.4	245.7
307	345.6	160.4	357	401.9	188.3	407	458.2	217.0	457	514.5	246.3
308	346.8	160.9	358	403.1	188.9	408	459.4	217.5	458	515.6	246.9
309	347.9	161.5	359	404.2	189.4	409	460.5	218.1	459	516.8	247.5
310	349.0	162.0	360	405.3	190.0	410	461.6	218.7	460	517.9	248.1
311	350.1	162.6	361	406.4	190.6	411	462.7	219.3	461	519.0	248.7
312	351.3	163.1	362	407.6	191.1	412	463.8	219.9	462	520.1	249.3
313	352.4	163.7	363	408.7	191.7	413	465.0	220.4	463	521.3	249.9
314	353.5	164.2	364	409.8	192.3	414	466.1	221.0			
315	354.6	164.8	365	410.9	192.9	415	467.2	221.6			
316	355.8	165.3	366	412.1	193.4	416	468.4	222.2			
317	356.9	165.9	367	413.2	194.0	417	469.5	222.8			
318	358.0	166.4	368	414.3	194.6	418	470.6	223.3			
319	359.1	167.0	369	415.4	195.1	419	471.8	223.9			
320	360.3	167.5	370	416.6	195.7	420	472.9	224.5			

of reducing sugars is sufficient to remove nearly all the copper from the solution, the error due to sucrose is but slight.

**Electrolytic Apparatus.**—The author has devised the apparatus shown in Fig. 110 for the electrolytic deposition of copper in sugar analysis and for other work of like nature. *A*, Fig. 110, is a hard-rubber plate 50 cm. long and 25 cm. wide provided with four insulated metal binding posts, *B*, each carrying at the top a thumb screw by which a coiled platinum wire







electrode, *C*, may be attached. In front of each post is a copper plate about 4 cm. square covered with thin platinum foil, *P*, which is bent around the edges of the copper plate and so held in place, the copper plate being screwed to the rubber from beneath. On the square platinum-covered plate is set the platinum evaporating-dish which holds the solution from which the copper is to be deposited, the inside of the dish forming the cathode, while the electrode *C*, dipping below the surface of the solution, forms the anode. In front of each platinum-covered plate is a switch, *S*, and at either end of the hard-rubber plate is a binding post, *R*, for connection with the electric current. The wiring, which is on the under side of the rubber plate, is best illustrated by the diagram in Fig. 110.

Four determinations may be carried on simultaneously in four platinum dishes, if desired, the wiring and the switches being so arranged that beginning at one end of the plate either the first dish or the first two or three may be thrown in or out of circuit at will without interrupting the current through the remaining dishes. A cover with wooden sides and glass top fits closely over the whole apparatus as a protection from dust, but may be easily lifted off to manipulate the dishes when desired. The sides of the cover are perforated to permit the escape of the gas formed during the electrolysis.

The ordinary street current is used when available, and the strength of the current may be varied within wide limits by means of a number of 16 or 32 candle-power lamps, *K*, coupled in multiple, and a rheostat, *L*, consisting of a vertical glass tube sealed at the bottom, containing a column of dilute acid, the resistance being changed by varying the length of the acid column contained between the two platinum terminals immersed therein, one of which is movable. A gravity battery of four cells may be employed if the laboratory is not equipped with electric lights.

In using this apparatus for determining copper, as in sugar work the plating process should go on till all the copper is deposited, requiring several hours or over night with a current strength of about 0.25 ampere. Before stopping the process, the absence of copper in the solution should be proved by removing a few drops with a pipette, adding first ammonia, then acetic acid, and testing with ferrocyanide of potassium. If no brown coloration is produced, all the copper has been plated out. Throw the dish out of circuit by means of the switch, pour out the acid solution quickly before it has a chance to dissolve any of the copper, wash the dish first with water and then with alcohol, dry, and weigh.



The copper may be removed from the platinum dish by strong nitric acid.

*The Ross Apparatus*\* consists of a funnel tube provided with a stop cock and a spiral of platinum wire one end of which passes through and is fused into the glass at the constriction. In using the apparatus form an asbestos mat in the constriction with the aid of suction, reduce the cuprous oxide by a suitable method, and filter and wash through the asbestos mat in the same manner as on a Gooch crucible. Close the stop cock, add dilute nitric acid (4 : 100) sufficient to nearly fill the tube, introduce a platinum cylinder to serve as the cathode, and use the spiral as the anode. Employ a current yielding not more than 1 cc. of electrolytic gas per minute. When the copper is all deposited draw off the liquid, wash the platinum cylinder, dry, and weigh.

**Determination of Invert Sugar in the Presence of Cane Sugar.**—*Meissl and Hiller Method*.†—Defecate 40 grams of the sample dissolved in 100 cc. of water in a 200-cc. graduated flask with a slight excess of normal lead acetate, make up to the mark, shake, filter, delead with dry sodium carbonate or sulphate and again filter. Place 5 cc. of alkaline copper solution in each of five test-tubes or small beakers, add 1, 2, 3, 4, and 5 cc. of the sugar solution to form a series, heat each to boiling, boil 2 minutes, and filter. The solution which gives the highest shade of blue (not colorless) contains the proper proportion of sugars for the actual determination. Pipette 20 times the volume used in the preliminary test into a 100-cc. graduated flask, make up to the mark and shake. Prepare 50 cc. of alkaline copper solution by mixing 25 cc. of each of the two solutions (page 615), heat to boiling, add 50 cc. of the sugar solution, heat again to boiling and boil for exactly 2 minutes. Filter and weigh as metallic copper, cuprous oxide, or cupric oxide and calculate the results using the following formulæ and table as simplified by Rice:‡

$$Y = \frac{100 Cu}{W} \quad \text{or} \quad \frac{88.82 Cu_2O}{W} \quad \text{or} \quad \frac{79.89 CuO}{W}; \quad S = \frac{100 P}{P + Y};$$

$$I = 100 - S, \quad I' = 0.02 YF,$$

in which  $S$  and  $I$  = approximate per cents of sucrose and invert sugar in sugar solids,  $P$  = polarization of sample,  $Cu$ ,  $Cu_2O$ , and  $CuO$  = weights

\* B. B. Ross, 8th Int. Cong. App. Chem., 8, 1912, p. 75.

† Zeits. Ver. deutsch. Zucker-Ind., 14, 1889, p. 715.

‡ Personal communication.



of copper, cuprous oxide, and cupric oxide found,  $W$  = weight of sample in 100 cc.,  $F$  = factor found in following table, and  $I'$  = true per cent of invert sugar in the sample.

MEISSL AND HILLER TABLE OF FACTORS FOR INVERT SUGAR DETERMINATION.

$S : I$	Factors for Weights of $Cu$ , $Cu_2O$ , and $CuO$ in Milligrams.						
	$Cu$ 400	$Cu$ 350	$Cu$ 300	$Cu$ 250	$Cu$ 200	$Cu$ 150	$Cu$ 100
	$Cu_2O$ 450	$Cu_2O$ 394	$Cu_2O$ 338	$Cu_2O$ 281	$Cu_2O$ 225	$Cu_2O$ 169	$Cu_2O$ 113
	$CuO$ 500	$CuO$ 437	$CuO$ 375	$CuO$ 312	$CuO$ 250	$CuO$ 187	$CuO$ 125
0 : 100	56.4	55.4	54.5	53.8	53.2	53.0	53.0
10 : 90	56.3	55.3	54.4	53.8	53.2	52.9	52.9
20 : 80	56.2	55.2	54.3	53.7	53.2	52.7	52.7
30 : 70	56.1	55.1	54.2	53.7	53.2	52.6	52.6
40 : 60	55.9	55.0	54.1	53.6	53.1	52.5	52.4
50 : 50	55.7	54.9	54.0	53.5	53.1	52.3	52.2
60 : 40	55.6	54.7	53.8	53.2	52.8	52.1	51.9
70 : 30	55.5	54.5	53.5	52.9	52.5	51.9	51.6
80 : 20	55.4	54.3	53.3	52.7	52.2	51.7	51.3
90 : 10	54.6	53.6	53.1	52.6	52.1	51.6	52.2
91 : 9	54.1	53.6	52.6	52.1	51.6	51.2	50.7
92 : 8	53.6	53.1	52.1	51.6	51.2	50.7	50.3
93 : 7	53.6	53.1	52.1	51.2	50.7	50.3	49.8
94 : 6	53.1	52.6	51.6	50.7	50.3	49.8	48.9
95 : 5	52.6	52.1	51.2	50.3	49.4	48.9	48.5
96 : 4	52.1	51.2	50.7	49.8	48.9	47.7	46.9
97 : 3	50.7	50.3	49.8	48.9	47.7	46.2	45.1
98 : 2	49.9	48.9	48.5	47.3	45.8	43.3	40.0
99 : 1	47.7	47.3	46.5	45.1	43.3	41.2	38.1

*Rice's Expanded Meissl and Hiller Table*\* given on pages 639 to 641 greatly facilitates the calculation of invert sugar as it gives percentages corresponding to different weights of copper, cuprous oxide, and cupric oxide, different amounts of the sample, and different polarizations. Instead of the amounts directed by Meissl and Hiller use for the preliminary tests 0.25, 0.50, 1.25, 2.50, and 5 cc. These amounts multiplied by 20 represent 1, 2, 5, 10, and 20 grams of the sample per 100 cc. Rice states, however, that he makes no preliminary tests; if the quantity used is too much he

\* 8th Int. Cong. App. Chem., 8, 1912, p. 47.



RICE'S EXPANDED MEISSL AND HILLER TABLE GIVING PERCENTAGES OF  
INVERT SUGAR.

Wt. of Sample in 100 cc.			1 Gram.		2 Grams.		5 Grams.		10 Grams.		20 Grams.	
Polarization.			30°	35°	20°	30°	85°	95°	85°	95°	85°	95°
Wt. Obtained as												
Cu	Cu <sub>2</sub> O	CuO										
0.0999	0.1125	0.1250	10.28	10.26	5.13	5.12	1.661	1.600	0.76	0.76		
0.1019	0.1147	0.1275	10.48	10.46	5.24	5.23	1.708	1.648	0.78	0.78		
0.1039	0.1170	0.1300	10.69	10.67	5.34	5.33	1.755	1.696	0.80	0.80		
0.1059	0.1192	0.1325	10.89	10.87	5.44	5.43	1.802	1.744	0.82	0.82		
0.1070	0.1215	0.1350	11.10	11.08	5.54	5.53	1.849	1.792	0.84	0.84		
0.1099	0.1237	0.1375	11.31	11.29	5.65	5.63	1.896	1.839	0.86	0.86		
0.1119	0.1260	0.1400	11.52	11.50	5.76	5.74	1.942	1.886	0.88	0.88		
0.1138	0.1282	0.1425	11.73	11.71	5.86	5.84	1.989	1.933	0.90	0.90		
0.1158	0.1305	0.1450	11.94	11.92	5.96	5.95	2.036	1.980	0.92	0.92		
0.1178	0.1327	0.1475	12.15	12.13	6.07	6.05	2.082	2.027	0.94	0.94		
0.1198	0.1350	0.1500	12.36	12.34	6.18	6.16	2.128	2.074	0.96	0.96		
0.1218	0.1372	0.1525	12.57	12.55	6.28	6.26	2.175	2.121	0.98	0.98		
0.1238	0.1395	0.1550	12.78	12.76	6.38	6.36	2.221	2.168	1.00	1.00		
0.1258	0.1417	0.1575	12.99	12.97	6.49	6.47	2.267	2.215	1.02	1.02		
0.1278	0.1440	0.1600	13.21	13.19	6.60	6.58	2.313	2.262	1.05	1.04		
0.1298	0.1462	0.1625	13.42	13.40	6.70	6.68	2.359	2.309	1.07	1.06		
0.1318	0.1485	0.1650	13.64	13.62	6.81	6.79	2.405	2.356	1.09	1.08		
0.1338	0.1507	0.1675	13.85	13.83	6.92	6.90	2.451	2.403	1.11	1.10		
0.1358	0.1530	0.1700	14.07	14.05	7.03	7.01	2.497	2.449	1.13	1.12		
0.1378	0.1552	0.1725	14.28	14.26	7.13	7.11	2.543	2.496	1.15	1.14		
0.1398	0.1575	0.1750	14.50	14.48	7.24	7.22	2.589	2.543	1.17	1.16		
0.1418	0.1597	0.1775	14.72	14.69	7.35	7.33	2.635	2.589	1.19	1.18		
0.1438	0.1620	0.1800	14.93	14.91	7.46	7.44	2.680	2.635	1.22	1.21		
0.1458	0.1642	0.1825	15.15	15.12	7.56	7.54	2.726	2.682	1.24	1.23		
0.1478	0.1665	0.1850	15.37	15.34	7.67	7.65	2.772	2.728	1.27	1.25		
0.1498	0.1687	0.1875	15.59	15.56	7.78	7.76	2.817	2.774	1.29	1.27		
0.1518	0.1710	0.1900	15.81	15.78	7.89	7.87	2.862	2.820	1.31	1.30		
0.1538	0.1732	0.1925	16.03	16.00	7.99	7.97	2.907	2.867	1.33	1.32		
0.1558	0.1755	0.1950	16.25	16.22	8.10	8.08	2.952	2.913	1.35	1.34		
0.1578	0.1777	0.1975	16.47	16.44	8.21	8.19	2.997	2.959	1.37	1.36		
0.1598	0.1800	0.2000	16.69	16.66	8.32	8.30	3.042	3.005	1.40	1.38		
0.1618	0.1822	0.2025	16.91	16.88	8.43	8.40	3.087	3.051	1.42	1.40		
0.1638	0.1845	0.2050	17.13	17.10	8.54	8.51	3.132	3.097	1.44	1.42		
0.1658	0.1867	0.2075	17.35	17.32	8.65	8.62	3.177	3.143	1.46	1.44		
0.1678	0.1890	0.2100	17.57	17.54	8.76	8.73	3.221	3.188	1.49	1.47		
0.1698	0.1912	0.2125	17.79	17.76	8.87	8.83	3.266	3.234	1.51	1.49		
0.1718	0.1935	0.2150	18.01	17.98	8.98	8.94	3.310	3.280	1.53	1.51	0.72	
0.1738	0.1957	0.2175	18.23	18.20	9.09	9.05	3.354	3.325	1.55	1.53	0.73	
0.1758	0.1980	0.2200	18.45	18.42	9.20	9.16	3.398	3.370	1.58	1.56	0.75	
0.1778	0.2002	0.2225	18.67	18.64	9.31	9.26	3.433	3.416	1.60	1.58	0.76	
0.1798	0.2025	0.2250	18.89	18.86	9.42	9.37	3.488	3.461	1.62	1.60	0.77	
0.1817	0.2047	0.2275	19.11	19.08	9.53	9.48	3.532	3.506	1.64	1.62	0.78	
0.1837	0.2070	0.2300	19.34	19.30	9.64	9.59	3.576	3.551	1.67	1.65	0.79	



RICE'S EXPANDED MEISSL AND HILLER TABLE GIVING PERCENTAGES OF  
INVERT SUGAR—Continued.

Wt. of Sample in 100 cc.			1 Gram.		2 Grams.		5 Grams.		10 Grams.		20 Grams.	
Polarization.			30°	35°	20°	30°	85°	95°	85°	95°	85°	95°
Wt. Obtained as												
Cu	Cu <sub>2</sub> O	CuO										
0.1857	0.2092	0.2325	19.56	19.52	9.75	9.70	3.621	3.597	1.69	1.67	0.80	
0.1877	0.2115	0.2350	19.78	19.74	9.86	9.81	3.666	3.642	1.71	1.69	0.81	
0.1897	0.2137	0.2375	20.00	19.96	9.97	9.92	3.710	3.687	1.73	1.72	0.82	0.81
0.1917	0.2160	0.2400	20.23	20.19	10.08	10.03	3.754	3.732	1.76	1.75	0.83	0.83
0.1937	0.2182	0.2425	20.45	20.41	10.19	10.14	3.799	3.777	1.78	1.77	0.84	0.84
0.1957	0.2205	0.2450	20.67	20.63	10.30	10.25	3.844	3.822	1.80	1.79	0.85	0.85
0.1977	0.2227	0.2475	20.89	20.85	10.41	10.36	3.888	3.867	1.83	1.81	0.86	0.86
0.1997	0.2250	0.2500	21.12	21.08	10.52	10.47	2.932	3.912	1.86	1.84	0.88	0.87
0.2017	0.2272	0.2525	21.34	21.30	10.63	10.58	3.977	3.957	1.88	1.86	0.89	0.88
0.2037	0.2295	0.2550	21.56	21.52	10.74	10.69	4.022	4.002	1.90	1.88	0.90	0.89
0.2057	0.2317	0.2575	21.78	21.74	10.85	10.80	4.066	4.047	1.92	1.90	0.91	0.90
0.2077	0.2340	0.2600	22.00	21.96	10.96	10.91	4.110	4.092	1.95	1.93	0.92	0.91
0.2097	0.2362	0.2625	22.22	22.18	11.07	11.02	4.155	4.137	1.97	1.95	0.93	0.92
0.2117	0.2385	0.2650	22.44	22.40	11.18	11.13	4.200	4.182	1.99	1.97	0.94	0.93
0.2137	0.2407	0.2675	22.66	22.62	11.29	11.24	4.244	4.227	2.02	1.99	0.95	0.94
0.2157	0.2430	0.2700	22.89	22.85	11.40	11.35	4.288	4.271	2.05	2.02	0.97	0.96
0.2177	0.2452	0.2725	23.11	23.07	11.51	11.46	4.333	4.316	2.07	2.04	0.98	0.97
0.2197	0.2475	0.2750	23.33	23.29	11.62	11.57	4.378	4.361	2.09	2.06	0.99	0.98
0.2217	0.2497	0.2775	23.55	23.51	11.73	11.68	4.422	4.405	2.11	2.08	1.00	0.99
0.2237	0.2520	0.2800	23.78	23.74	11.84	11.79	4.466	4.449	2.14	2.12	1.01	1.00
0.2257	0.2542	0.2825	24.00	23.96	11.95	11.90	4.511	4.494	2.16	2.14	1.02	1.01
0.2277	0.2565	0.2850	24.22	24.18	12.06	12.01	4.556	4.538	2.18	2.16	1.03	1.02
0.2297	0.2587	0.2875	24.44	24.40	12.17	12.12	4.600	4.582	2.20	2.18	1.04	1.03
0.2317	0.2610	0.2900	24.67	24.63	12.28	12.23	4.644	4.626	2.23	2.21	1.06	1.05
0.2337	0.2633	0.2925	24.89	24.85	12.39	12.34	4.689	4.671	2.25	2.23	1.07	1.06
0.2357	0.2655	0.2950	25.11	25.07	12.50	12.45	4.734	4.716	2.27	2.25	1.08	1.07
0.2377	0.2677	0.2975	25.33	25.29	12.61	12.56	4.779	4.761	2.29	2.27	1.09	1.08
0.2397	0.2700	0.3000	25.56	25.52	12.73	12.67	4.823	4.805	2.32	2.30	1.10	1.09
0.2417	0.2722	0.3025	25.78	25.74	12.84	12.78	4.868	4.850	2.34	2.32	1.11	1.10
0.2437	0.2745	0.3050	26.00	25.96	12.95	12.89	4.913	4.895	2.36	2.34	1.12	1.11
0.2457	0.2767	0.3075	26.22	26.18	13.06	13.00	4.958	4.940	2.39	2.37	1.13	1.12
0.2477	0.2790	0.3100	26.45	26.41	13.18	13.11	5.003	4.985	2.42	2.40	1.15	1.14
0.2497	0.2812	0.3125	26.67	26.63	13.29	13.22	5.049	5.031	2.44	2.42	1.16	1.15
0.2517	0.2835	0.3150	26.90	26.85	13.40	13.33	5.095	5.076	2.46	2.44	1.17	1.16
0.2537	0.2857	0.3175	27.12	27.07	13.51	13.44	5.141	5.121	2.48	2.46	1.18	1.17
0.2556	0.2880	0.3200	27.35	27.30	13.63	13.56	5.186	5.166	2.51	2.49	1.19	1.18
0.2576	0.2902	0.3225	27.57	27.52	13.74	13.67	5.232	5.212	2.53	2.51	1.20	1.19
0.2596	0.2925	0.3250	27.80	27.75	13.85	13.78	5.278	5.258	2.55	2.53	1.21	1.20
0.2616	0.2947	0.3275	28.02	27.97	13.96	13.89	5.324	5.303	2.57	2.55	1.22	1.21
0.2636	0.2970	0.3300	28.25	28.20	14.08	14.01	5.371	5.348	2.60	2.58	1.24	1.23
0.2656	0.2992	0.3325	28.47	28.42	14.19	14.12	5.418	5.394	2.62	2.60	1.25	1.24
0.2676	0.3015	0.3350	28.70	28.65	14.30	14.23	5.465	5.440	2.64	2.62	1.26	1.25
0.2696	0.3037	0.3375	28.93	28.87	14.42	14.35	5.512	5.486	2.66	2.64	1.27	1.26



RICE'S EXPANDED MEISSL AND HILLER TABLE GIVING PERCENTAGES OF  
INVERT SUGAR—*Concluded.*

Wt. of Sample in 100 cc.			1 Gram.		2 Grams.		5 Grams.		10 Grams.		20 Grams.	
Polarization.			30°	35°	20°	30°	85°	95°	95°	85°	85°	95°
Wt. Obtained as												
Cu	Cu <sub>2</sub> O	CuO										
0.2716	0.3060	0.3400	29.16	29.10	14.54	14.47	5.558	5.532	2.69	2.67	1.28	1.27
0.2736	0.3082	0.3425	29.39	29.32	14.65	14.59	5.605	5.578	2.71	2.69	1.29	1.28
0.2756	0.3105	0.3450	29.62	29.55	14.76	14.70	5.652	5.624	2.73	2.71	1.30	1.29
0.2776	0.3127	0.3475	29.85	29.77	14.88	14.81	5.699	5.671	2.75	2.73	1.31	1.30
0.2796	0.3150	0.3500	30.08	30.00	15.00	14.93	5.746	5.718	2.78	2.76	1.33	1.32
0.2816	0.3172	0.3525	30.31	30.23	15.11	15.04	5.793	5.765	2.80	2.78	1.34	1.33
0.2836	0.3195	0.3550	30.54	30.46	15.22	15.15	5.840	5.812	2.82	2.80	1.35	1.34
0.2856	0.3217	0.3575	30.77	30.69	15.34	15.27	5.888	5.859	2.84	2.82	1.36	1.35
0.2876	0.3240	0.3600	31.00	30.93	15.46	15.39	5.936	5.906	2.87	2.85	1.37	1.36
0.2896	0.3262	0.3625	31.23	31.16	15.57	15.50	5.983	5.953	2.89	2.87	1.38	1.37
0.2916	0.3285	0.3650	31.46	31.40	15.69	15.61	6.031	6.000	2.91	2.88	1.39	1.38
0.2936	0.3307	0.3675	31.69	31.63	15.81	15.73	6.079	6.048	2.93	2.91	1.40	1.39
0.2956	0.3330	0.3700	31.93	31.87	15.93	15.85	6.127	6.096	2.96	2.94	1.42	1.41
0.2976	0.3352	0.3725	32.16	32.10	16.04	15.96	6.174	6.144	2.98	2.96	1.43	1.42
0.2996	0.3375	0.3750	32.40	32.34	16.16	16.08	6.222	6.192	3.00	2.98	1.44	1.43
0.3016	0.3397	0.3775	32.63	32.67	16.28	16.20	6.270	6.240	3.03	3.00	1.45	1.44
0.3036	0.3420	0.3800	32.87	32.81	16.40	16.32	6.318	6.288	3.06	3.03	1.46	1.45
0.3056	0.3442	0.3825	33.10	33.04	16.52	16.44	6.366	6.337	3.08	3.05	1.47	1.47
0.3076	0.3465	0.3850	33.34	33.28	16.64	16.56	6.414	6.386	3.10	3.07	1.48	1.48
0.3096	0.3487	0.3875	33.58	33.52	16.76	16.68	6.462	6.434	3.12	3.09	1.49	1.49
0.3116	0.3510	0.3900	33.82	33.76	16.88	16.80	6.510	6.482	3.15	3.12	1.51	1.50
0.3136	0.3532	0.3925	34.06	34.00	17.00	16.92	6.558	6.531	3.17	3.14	1.52	1.51
0.3156	0.3555	0.3950	34.30	34.24	17.12	17.04	6.608	6.580	3.19	3.16	1.53	1.52
0.3176	0.3577	0.3975	34.54	34.48	17.24	17.16	6.654	6.629	3.21	3.18	1.54	1.53
0.3196	0.3600	0.4000	34.78	34.72	17.36	17.28	6.703	6.678	3.24	3.21	1.55	1.54
0.3216	0.3622	0.4025	35.02	34.96	17.48	17.40	6.751	6.727	3.26	3.23	1.56	1.55
0.3236	0.3645	0.4050	35.26	35.20	17.60	17.52	6.799	6.776	3.28	3.26	1.57	1.56
0.3256	0.3667	0.4075	35.50	35.44	17.72	17.64	6.848	6.825	3.30	3.27	1.58	1.57
0.3275	0.3890	0.4100	35.75	35.68	17.84	17.76	6.897	6.875	3.33	3.30	1.60	1.59
0.3295	0.3712	0.4125	35.99	35.92	17.96	17.88	6.945	6.924	3.35	3.32	1.61	1.60
0.3315	0.3735	0.4150	36.24	36.16	18.08	18.00	6.993	6.973	3.37	3.34	1.62	1.60
0.3335	0.3757	0.4175	36.48	36.40	18.20	18.12	7.042	7.023	3.39	3.36	1.63	1.62
0.3355	0.3780	0.4200	36.73	36.65	18.33	18.25	7.091	7.073	3.42	3.39	1.64	1.63
0.3375	0.3802	0.4225	36.97	36.89	18.45	18.37	7.139	7.122	3.44	3.41	1.65	1.64
0.3395	0.3825	0.4250	37.22	37.13	18.57	18.49	7.188	7.172	3.46	3.43	1.66	1.65
0.3415	0.3847	0.4275	37.47	37.37	18.69	18.61	7.237	7.222	3.48	3.45	1.67	1.66
0.3435	0.3870	0.4300	37.72	37.62	18.82	18.74	7.286	7.272	3.51	3.48	1.69	1.68
0.3455	0.3892	0.4325	37.96	37.86	18.94	18.86	7.334	7.321	3.53	3.50	1.70	1.69
0.3475	0.3915	0.4350	38.21	38.10	19.06	18.99	7.383	7.371	3.55	3.52	1.71	1.70
0.3495	0.3937	0.4375	38.46	38.44	19.19	19.12	7.432	7.421	3.58	3.55	1.72	1.71



starts with another portion of half the dilution. Full details of the process as now conducted follow: \*

"Weigh out a quantity which from the direct polarization seems proper, always estimating low. Dissolve and make up to mark and if not clear pour onto a filter paper in which has been placed a level teaspoonful of dry kieselguhr. Pour back until the filtrate comes clear. Place 50 cc. of the solution and 50 cc. of mixed alkaline copper solution in a 350-cc. Griffin beaker and cover with clock glass. Heat on a piece of sheet asbestos, with a hole 5 cm. in diameter below which is a wire gauze, so as to reach boiling in 4 minutes or under 5 minutes. Boil exactly 2 minutes, then pour in 100 cc. of cold water. Remove from the flame immediately, filter through an ignited weighed porcelain Gooch crucible containing a layer of asbestos 3 mm. thick, previously treated for days with strong hydrochloric acid and alkaline copper solution. Heat  $\frac{1}{2}$  hour at dull redness, cool, and weigh as CuO."

**Determination of Sucrose by Fehling's Solution.**†—If a polariscope is not available, cane sugar can be determined as follows: First determine the percentage of invert sugar present in the sample by one of the Fehling methods already described. Then dissolve 1 gram of the sugar in about 100 cc. of water in a 500-cc. graduated flask, add 3 cc. of concentrated hydrochloric acid and invert by heating in water to 68° and cooling in the regular manner. Neutralize with sodium hydroxide or sodium carbonate, and make up to the mark with water. Determine the per cent of total reducing sugar as invert sugar either by the volumetric or gravimetric Fehling process. Subtract the invert sugar found present in the sugar by direct determination from the total found present after inversion, and the remainder is the invert sugar due to cane sugar. This figure multiplied by 0.95 gives the percentage of cane sugar.

For the determination of sucrose by the gravimetric Fehling process on the inverted sample, multiply the cupric oxide (CuO) by the factor 0.4307, or the copper (Cu) by the factor 0.5394.

#### ANALYSIS OF MOLASSES AND SYRUPS.

First insure a perfectly homogeneous sample by stirring with a rod to evenly distribute any separated sugar.

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\* Personal communication.

† Tucker, *Manual of Sugar Analysis*, p. 182.



**Determination of Total Solids.**—(1) *Asbestos Method*.—Weigh 20 grams into a 100-cc. graduated flask, dissolve in water, and make up to the mark. Insure a uniform solution by shaking. Measure 10 cc. of this solution into a tared platinum dish containing about 5 grams of freshly ignited, finely divided asbestos fiber, and dry to constant weight at 70° *in vacuo*, or in a McGill oven (see page 609).

(2) *Sand Method*.—Place a stirring rod in a flat-bottom metal dish, add ignited quartz sand sufficient to bring the total weight up to an even number of grams using not less than 12 to 15 grams, and weigh. Add 2 to 4 grams of the material, dilute with water, and mix thoroughly. Dry on a water bath with stirring and finally in a water oven until the loss is insignificant.

(3) *By Calculation from Refractive Index*.—Determine the refractive index by means of the Abbé refractometer (page 94), and calculate the total solids, using Geerlig's tables (p. 645).

This method is more accurate and convenient than the specific gravity method and employs a smaller quantity of material. The investigations of Stolle\* and of Tolman and Smith† have shown that sucrose, maltose, dextrose, levulose and lactose all have practically the same refractive index. Dextrin has a somewhat higher refractive index, nevertheless the solids of commercial glucose do not give a reading appreciably higher than the sugars named.

A. H. Bryan‡ has compared this method with the method of drying at 70° *in vacuo*, with the following results:

Material.	Number of Samples.	Difference compared with the Gravimetric Method.
Maple syrup .....	13	-1.34 to +0.72
Cane table syrup .....	10	-0.79 to +0.62
Cane molasses.....	17	-1.53 to +0.59
Beet molasses .....	15	-1.83 to -0.07
Honey .....	24	-2.52 to +0.91
Glucose.....	2	-0.27 to +0.27

(4) *By Calculation from Specific Gravity*.—Weigh 25 grams of the sample into a 100-cc. graduated flask, dissolve in water, and make up

\* Zeits. deutsch. Zucker-Ind., 1901, pp. 335, 469.

† Jour. Am. Chem. Soc., 28, 1906, p. 1476.

‡ Ibid., 30, 1908, p. 1443.



to the mark. Determine the specific gravity, at  $\frac{20^{\circ}}{4^{\circ}}$  C., of the diluted solution by means of a pycnometer or accurate hydrometer.

Ascertain from the table on pages 647 and 648 the percentage by weight of solids (sugar) corresponding to the specific gravity of the diluted solution, and calculate the total solids in the original sample by the following formula:

$$S' = 4DS,$$

in which  $S'$  is the total solids in original sample,  $D$  is the specific gravity of the diluted solution, and  $S$  is the per cent of solids in the diluted solution.

The solids may also be obtained directly by means of the saccharometer, also known as the Brix spindle. This instrument is a hydrometer graduated so as to show the per cent of sugar when the temperature of the liquid is  $20^{\circ}$  C.

If the specific gravity or saccharometer reading is taken at any other temperature than  $20^{\circ}$  C. the necessary correction may be found in the table on page 649.

**Determination of Ash.**—Weigh from 5 to 10 grams of the sample into a tared platinum dish, evaporate to dryness on the water-bath, and proceed as directed for ash of sugar (page 609).

**Polarization and Determination of Sucrose.**—Molasses and golden syrup require the application of clarifying reagents before a sufficiently clear solution can be obtained for reading on the polariscope. Even then it is not possible nor is it necessary to get a water-white solution, so that in this class of products greater accuracy can usually be attained by polarizing in a 100-mm. tube (half the standard length) and multiplying the reading by 2. In some cases it may be found necessary to use an even shorter tube.

When the sample contains a considerable amount of glucose the use of the shorter tube is absolutely necessary since otherwise the range of the scale would not permit of a reading.

The clarifier best adapted as a rule for molasses and golden syrup is lead subacetate either in the form of a solution as described on page 610, or, as first proposed by Horne,\* in the form of the anhydrous salt.

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\* Jour. Am. Chem. Soc., 26, 1904, p. 186.



GEERLIG'S TABLE FOR DRY SUBSTANCE IN SUGAR-HOUSE PRODUCTS  
BY THE ABBE REFRACTOMETER, AT 28° C.\*

Refractive Index.	Per Cent Dry Substance.	Decimals to be Added for Fractional Readings.†		Refractive Index.	Per Cent Dry Substance.	Decimals to be Added for Fractional Readings.†	
1.3335	1	0.0001=0.05	0.0010=0.75	1.4083	45	0.0004=0.2	0.0015=0.75
1.3349	2	0.0002=0.1	0.0011=0.8	1.4104	46	0.0005=0.25	0.0016=0.8
1.3364	3	0.0003=0.2	0.0012=0.8	1.4124	47	0.0006=0.3	0.0017=0.85
1.3379	4	0.0004=0.25	0.0013=0.85	1.4145	48	0.0007=0.35	0.0018=0.9
1.3394	5	0.0005=0.3	0.0014=0.9	1.4166	49	0.0008=0.4	0.0019=0.95
1.3409	6	0.0006=0.4	0.0015=1.0	1.4186	50	0.0009=0.45	0.0020=1.0
1.3424	7	0.0007=0.5		1.4207	51	0.0010=0.5	0.0021=1.0
1.3439	8	0.0008=0.6		1.4228	52	0.0011=0.55	
1.3454	9	0.0009=0.7		1.4249	53		
1.3469	10			1.4270	54		
1.3484	11	0.0001=0.05		1.4292	55	0.0001=0.05	0.0013=0.55
1.3500	12	0.0002=0.1		1.4314	56	0.0002=0.1	0.0014=0.6
1.3516	13	0.0003=0.2		1.4337	57	0.0003=0.1	0.0015=0.65
1.3530	14	0.0004=0.25		1.4359	58	0.0004=0.15	0.0016=0.7
1.3546	15	0.0005=0.3		1.4382	59	0.0005=0.2	0.0017=0.75
1.3562	16	0.0006=0.4		1.4405	60	0.0006=0.25	0.0018=0.8
1.3578	17	0.0007=0.45		1.4428	61	0.0007=0.3	0.0019=0.85
1.3594	18	0.0008=0.5		1.4451	62	0.0008=0.35	0.0020=0.9
1.3611	19	0.0009=0.6		1.4474	63	0.0009=0.4	0.0021=0.9
1.3627	20	0.0010=0.65		1.4497	64	0.0010=0.45	0.0022=0.95
1.3644	21	0.0011=0.7		1.4520	65	0.0011=0.5	0.0023=1.0
1.3661	22	0.0012=0.75		1.4543	66	0.0012=0.5	0.0024=1.0
1.3678	23	0.0013=0.8		1.4567	67		
1.3695	24	0.0014=0.85		1.4591	68		
1.3712	25	0.0015=0.9		1.4615	69		
1.3729	26	0.0016=0.95		1.4639	70		
				1.4663	71		
				1.4687	72		
1.3746	27	0.0001=0.05	0.0012=0.6				
1.3764	28	0.0002=0.1	0.0013=0.65				
1.3782	29	0.0003=0.15	0.0014=0.7	1.4711	73	0.0001=0.0	0.0015=0.55
1.3800	30	0.0004=0.2	0.0015=0.75	1.4736	74	0.0002=0.05	0.0016=0.6
1.3818	31	0.0005=0.25	0.0016=0.8	1.4761	75	0.0003=0.1	0.0017=0.65
1.3836	32	0.0006=0.3	0.0017=0.85	1.4786	76	0.0004=0.15	0.0018=0.65
1.3854	33	0.0007=0.35	0.0018=0.9	1.4811	77	0.0005=0.2	0.0019=0.7
1.3872	34	0.0008=0.45	0.0019=0.95	1.4836	78	0.0006=0.2	0.0020=0.75
1.3890	35	0.0009=0.4	0.0020=1.0	1.4862	79	0.0007=0.25	0.0021=0.8
1.3909	36	0.0010=0.5	0.0021=1.0	1.4888	80	0.0008=0.3	0.0022=0.8
1.3928	37	0.0011=0.55		1.4914	81	0.0009=0.35	0.0023=0.85
1.3947	38			1.4940	82	0.0010=0.35	0.0024=0.9
1.3966	39			1.4966	83	0.0011=0.4	0.0025=0.9
1.3984	40			1.4992	84	0.0012=0.45	0.0026=0.95
1.4003	41			1.5019	85	0.0013=0.5	0.0027=1.0
				1.5046	86	0.0014=0.5	0.0028=1.0
				1.5073	87		
1.4023	42	0.0001=0.05	0.0012=0.6	1.5100	88		
1.4043	43	0.0002=0.1	0.0013=0.65	1.5127	89		
1.4063	44	0.0003=0.15	0.0014=0.7	1.5155	90		

\* Intern. Sugar Jour., 10, pp. 69-70.

† Find in the table the refractive index which is next lower than the reading actually made and note the corresponding whole number for the per cent of dry substance. Subtract the refractive index obtained from the table from the observed reading; the decimal corresponding to this difference, as given in the column so marked, is added to the whole per cent of dry substance as first obtained.



## TEMPERATURE CORRECTIONS FOR USE WITH GEERLIG'S TABLE.

Temperature of the Prisms in °C.	Dry Substance.												
	0	5	10	15	20	25	30	40	50	60	70	80	90
	Subtract—												
20	0.53	0.54	0.55	0.56	0.57	0.58	0.60	0.62	0.64	0.62	0.61	0.60	0.58
21	.46	.47	.48	.49	.50	.51	.52	.54	.56	.54	.53	.52	.50
22	.40	.41	.42	.42	.43	.44	.45	.47	.48	.47	.46	.45	.44
23	.33	.33	.34	.35	.36	.37	.38	.39	.40	.39	.38	.38	.38
24	.26	.26	.27	.28	.28	.29	.30	.31	.32	.31	.31	.30	.30
25	.20	.20	.21	.21	.22	.22	.23	.23	.24	.23	.23	.23	.22
26	.12	.12	.13	.14	.14	.15	.15	.16	.16	.16	.15	.15	.14
27	.07	.07	.07	.07	.07	.07	.08	.08	.08	.08	.08	.08	.07
	Add—												
	0	5	10	15	20	25	30	40	50	60	70	80	90
29	0.07	0.07	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.08	0.08	0.07
30	.12	.12	.13	.14	.14	.14	.15	.15	.16	.16	.16	.15	.14
31	.20	.20	.21	.21	.22	.22	.23	.23	.24	.23	.23	.23	.22
32	.26	.26	.27	.28	.28	.29	.30	.31	.32	.31	.31	.30	.30
33	.33	.33	.34	.35	.36	.37	.38	.39	.40	.39	.38	.38	.38
34	.40	.41	.42	.42	.43	.44	.45	.47	.48	.47	.46	.45	.44
35	.46	.47	.48	.49	.50	.51	.52	.54	.56	.54	.53	.52	.50

*The Process.*—The normal weight, 26 grams, of the molasses or syrup is dissolved in water in a 100-cc. flask, and in the case of molasses and "golden," or "drip" syrup, sufficient subacetate of lead solution is added to precipitate the coloring matter. From 5 to 10 cc. of the clarifier usually suffice. The flask is then filled to the mark with water and the contents shaken thoroughly and filtered. If on account of air bubbles it is difficult to make up to the mark, the bubbles may usually be dispelled by a drop of ether. With maple syrup no clarifier is, as a rule, necessary, though sometimes alumina cream is helpful. With a very dark-colored molasses 20 to 30 cc. of lead subacetate are required for clarification and in extreme cases (though rarely with the grades of molasses used as food) it is necessary, after the ordinary filtration, to pass through from 5 to 6 grams of powdered, dried bone charcoal.\*

An excess of subacetate of lead should be avoided on account of the possibility of the filtrate becoming turbid through the formation of lead carbonate by exposure to the air. A drop of acetic acid will nearly always clear the solution, if the turbidity is due to carbonate. If cloudiness in the filtrate persists, weigh out a fresh portion of the sample, dilute, and add first the lead subacetate solution, and afterwards enough of a strong solution of sodium sulphate or common salt to precipitate the excess of lead; then fill to the mark and filter. Polarize, and conduct the inversion as directed on p. 610, using, however, a 100-mm. tube, and multi-

\* The treatment with bone char should be used only as a last resort, as, on account of slight absorption of sugar, observed readings are from 0.4° to 0.5° too low.



DENSITY OF SOLUTIONS OF CANE SUGAR AT  $\frac{20^{\circ}}{4^{\circ}}$  C.\*

Per Cent Sugar.	Tenths of Per Cent.									
	0	1	2	3	4	5	6	7	8	9
0	0.9982	0.9986	0.9990	0.9994	0.9998	1.0002	1.0006	1.0010	1.0013	1.0017
1	1.0021	1.0025	1.0029	1.0033	1.0037	1.0041	1.0045	1.0048	1.0052	1.0056
2	1.0060	1.0064	1.0068	1.0072	1.0076	1.0080	1.0084	1.0088	1.0091	1.0095
3	1.0099	1.0103	1.0107	1.0111	1.0115	1.0119	1.0123	1.0127	1.0131	1.0135
4	1.0139	1.0143	1.0147	1.0151	1.0155	1.0159	1.0163	1.0167	1.0171	1.0175
5	1.0179	1.0183	1.0187	1.0191	1.0195	1.0199	1.0203	1.0207	1.0211	1.0215
6	1.0219	1.0223	1.0227	1.0231	1.0235	1.0239	1.0243	1.0247	1.0251	1.0255
7	1.0259	1.0263	1.0267	1.0271	1.0276	1.0279	1.0283	1.0287	1.0291	1.0295
8	1.0299	1.0303	1.0308	1.0312	1.0316	1.0320	1.0324	1.0328	1.0332	1.0336
9	1.0340	1.0344	1.0349	1.0353	1.0357	1.0361	1.0365	1.0369	1.0373	1.0377
10	1.0381	1.0386	1.0390	1.0394	1.0398	1.0402	1.0406	1.0410	1.0415	1.0419
11	1.0423	1.0427	1.0431	1.0435	1.0440	1.0444	1.0448	1.0452	1.0456	1.0460
12	1.0465	1.0469	1.0473	1.0477	1.0481	1.0486	1.0490	1.0494	1.0498	1.0502
13	1.0507	1.0511	1.0515	1.0519	1.0524	1.0528	1.0532	1.0536	1.0540	1.0545
14	1.0549	1.0553	1.0558	1.0562	1.0566	1.0570	1.0575	1.0579	1.0583	1.0587
15	1.0592	1.0596	1.0600	1.0605	1.0609	1.0613	1.0617	1.0622	1.0626	1.0630
16	1.0635	1.0639	1.0643	1.0648	1.0652	1.0656	1.0661	1.0665	1.0669	1.0674
17	1.0678	1.0682	1.0687	1.0691	1.0695	1.0700	1.0704	1.0708	1.0713	1.0717
18	1.0721	1.0726	1.0730	1.0735	1.0739	1.0743	1.0748	1.0752	1.0757	1.0761
19	1.0765	1.0770	1.0774	1.0779	1.0783	1.0787	1.0792	1.0796	1.0801	1.0805
20	1.0810	1.0814	1.0818	1.0823	1.0827	1.0832	1.0836	1.0841	1.0845	1.0850
21	1.0854	1.0859	1.0863	1.0868	1.0872	1.0877	1.0881	1.0885	1.0890	1.0894
22	1.0899	1.0904	1.0908	1.0913	1.0917	1.0922	1.0926	1.0931	1.0935	1.0940
23	1.0944	1.0949	1.0953	1.0958	1.0962	1.0967	1.0971	1.0976	1.0981	1.0985
24	1.0990	1.0994	1.0999	1.1003	1.1008	1.1013	1.1017	1.1022	1.1026	1.1031
25	1.1036	1.1040	1.1045	1.1049	1.1054	1.1059	1.1063	1.1068	1.1072	1.1077
26	1.1082	1.1086	1.1091	1.1096	1.1100	1.1105	1.1110	1.1114	1.1119	1.1124
27	1.1128	1.1133	1.1138	1.1142	1.1147	1.1152	1.1156	1.1161	1.1166	1.1170
28	1.1175	1.1180	1.1185	1.1189	1.1194	1.1199	1.1203	1.1208	1.1213	1.1218
29	1.1222	1.1227	1.1232	1.1237	1.1241	1.1246	1.1251	1.1256	1.1260	1.1265
30	1.1270	1.1275	1.1279	1.1284	1.1289	1.1294	1.1299	1.1303	1.1308	1.1313
31	1.1318	1.1323	1.1327	1.1332	1.1337	1.1342	1.1347	1.1351	1.1356	1.1361
32	1.1366	1.1371	1.1376	1.1380	1.1385	1.1390	1.1395	1.1400	1.1405	1.1410
33	1.1415	1.1419	1.1424	1.1429	1.1434	1.1439	1.1444	1.1449	1.1454	1.1459
34	1.1463	1.1468	1.1473	1.1478	1.1483	1.1488	1.1493	1.1498	1.1503	1.1508
35	1.1513	1.1518	1.1523	1.1528	1.1533	1.1538	1.1542	1.1547	1.1552	1.1557
36	1.1562	1.1567	1.1572	1.1577	1.1582	1.1587	1.1592	1.1597	1.1602	1.1607
37	1.1612	1.1617	1.1622	1.1627	1.1632	1.1637	1.1643	1.1648	1.1653	1.1658
38	1.1663	1.1668	1.1673	1.1678	1.1683	1.1688	1.1693	1.1698	1.1703	1.1708
39	1.1713	1.1718	1.1724	1.1729	1.1734	1.1739	1.1744	1.1749	1.1754	1.1759
40	1.1764	1.1770	1.1775	1.1780	1.1785	1.1790	1.1795	1.1800	1.1806	1.1811
41	1.1816	1.1821	1.1826	1.1831	1.1837	1.1842	1.1847	1.1852	1.1857	1.1863
42	1.1868	1.1873	1.1878	1.1883	1.1889	1.1894	1.1899	1.1904	1.1909	1.1915
43	1.1920	1.1925	1.1930	1.1936	1.1941	1.1947	1.1951	1.1957	1.1962	1.1967
44	1.1972	1.1978	1.1983	1.1988	1.1994	1.1999	1.2004	1.2009	1.2015	1.2020
45	1.2025	1.2031	1.2036	1.2041	1.2047	1.2052	1.2057	1.2063	1.2068	1.2073
46	1.2079	1.2084	1.2089	1.2095	1.2100	1.2105	1.2111	1.2116	1.2122	1.2127
47	1.2132	1.2138	1.2143	1.2149	1.2154	1.2159	1.2165	1.2170	1.2176	1.2181
48	1.2186	1.2192	1.2197	1.2203	1.2208	1.2214	1.2219	1.2224	1.2230	1.2235
49	1.2241	1.2246	1.2252	1.2257	1.2263	1.2268	1.2274	1.2279	1.2285	1.2290
50	1.2296	1.2301	1.2307	1.2312	1.2318	1.2323	1.2329	1.2334	1.2340	1.2345

\* According to Dr. F. Plato (Kaiserlichen Normal-Eichungs-Kommission, Wiss. Abh., 2, 1900, page 153). This table (carried out to the 6th place of decimals) is given by the U. S. Bureau of Standards. (Circ. 44, pp. 137-139) as the basis for standardizing hydrometers, indicating per cent of sugar at 20°, known as saccharometers or Brix spindles. The table is also useful in calculating the per cent of sugar from the specific gravity as determined by the pycnometer. Temperature corrections are given on page 649.



Per Cent Sugar.	Tenths of Per Cent.									
	0	1	2	3	4	5	6	7	8	9
50	1.2296	1.2301	1.2307	1.2312	1.2318	1.2323	1.2329	1.2334	1.2340	1.2345
51	1.2351	1.2356	1.2362	1.2367	1.2373	1.2379	1.2384	1.2390	1.2395	1.2401
52	1.2406	1.2412	1.2418	1.2423	1.2429	1.2434	1.2440	1.2446	1.2451	1.2457
53	1.2462	1.2468	1.2474	1.2479	1.2485	1.2490	1.2496	1.2502	1.2507	1.2513
54	1.2519	1.2524	1.2530	1.2536	1.2541	1.2547	1.2553	1.2558	1.2564	1.2570
55	1.2575	1.2581	1.2587	1.2592	1.2598	1.2604	1.2610	1.2615	1.2621	1.2627
56	1.2632	1.2638	1.2644	1.2650	1.2655	1.2661	1.2667	1.2673	1.2678	1.2684
57	1.2690	1.2696	1.2701	1.2707	1.2713	1.2719	1.2725	1.2730	1.2736	1.2742
58	1.2748	1.2754	1.2759	1.2765	1.2771	1.2777	1.2783	1.2788	1.2794	1.2800
59	1.2806	1.2812	1.2818	1.2823	1.2829	1.2835	1.2841	1.2847	1.2853	1.2859
60	1.2865	1.2870	1.2876	1.2882	1.2888	1.2894	1.2900	1.2906	1.2912	1.2918
61	1.2924	1.2929	1.2935	1.2941	1.2947	1.2953	1.2959	1.2965	1.2971	1.2977
62	1.2983	1.2989	1.2995	1.3001	1.3007	1.3013	1.3019	1.3025	1.3031	1.3037
63	1.3043	1.3049	1.3055	1.3061	1.3067	1.3073	1.3079	1.3085	1.3091	1.3097
64	1.3103	1.3109	1.3115	1.3121	1.3127	1.3133	1.3139	1.3145	1.3151	1.3157
65	1.3163	1.3169	1.3175	1.3182	1.3188	1.3194	1.3200	1.3206	1.3212	1.3218
66	1.3224	1.3230	1.3236	1.3243	1.3249	1.3255	1.3261	1.3267	1.3273	1.3279
67	1.3286	1.3292	1.3298	1.3304	1.3310	1.3316	1.3322	1.3329	1.3335	1.3341
68	1.3347	1.3353	1.3360	1.3366	1.3372	1.3378	1.3384	1.3391	1.3397	1.3403
69	1.3409	1.3416	1.3422	1.3428	1.3434	1.3440	1.3447	1.3453	1.3459	1.3465
70	1.3472	1.3478	1.3484	1.3491	1.3497	1.3503	1.3509	1.3516	1.3522	1.3528
71	1.3535	1.3541	1.3547	1.3553	1.3560	1.3566	1.3572	1.3579	1.3585	1.3591
72	1.3598	1.3604	1.3610	1.3617	1.3623	1.3630	1.3636	1.3642	1.3649	1.3655
73	1.3661	1.3668	1.3674	1.3681	1.3687	1.3693	1.3700	1.3706	1.3713	1.3719
74	1.3725	1.3732	1.3738	1.3745	1.3751	1.3757	1.3764	1.3770	1.3777	1.3783
75	1.3790	1.3796	1.3803	1.3809	1.3816	1.3822	1.3829	1.3835	1.3841	1.3848
76	1.3854	1.3861	1.3867	1.3874	1.3880	1.3887	1.3893	1.3900	1.3907	1.3913
77	1.3920	1.3926	1.3933	1.3939	1.3946	1.3952	1.3959	1.3965	1.3972	1.3978
78	1.3985	1.3992	1.3998	1.4005	1.4011	1.4018	1.4025	1.4031	1.4038	1.4044
79	1.4051	1.4058	1.4064	1.4071	1.4077	1.4084	1.4091	1.4097	1.4104	1.4111
80	1.4117	1.4124	1.4130	1.4137	1.4144	1.4150	1.4157	1.4164	1.4170	1.4177
81	1.4184	1.4190	1.4197	1.4204	1.4210	1.4217	1.4224	1.4231	1.4237	1.4244
82	1.4251	1.4257	1.4264	1.4271	1.4278	1.4284	1.4291	1.4298	1.4305	1.4311
83	1.4318	1.4325	1.4332	1.4338	1.4345	1.4352	1.4359	1.4365	1.4372	1.4379
84	1.4386	1.4393	1.4399	1.4406	1.4413	1.4420	1.4427	1.4433	1.4440	1.4447
85	1.4454	1.4461	1.4468	1.4474	1.4481	1.4488	1.4495	1.4502	1.4509	1.4515
86	1.4522	1.4529	1.4536	1.4543	1.4550	1.4557	1.4564	1.4570	1.4577	1.4584
87	1.4591	1.4598	1.4605	1.4612	1.4619	1.4626	1.4633	1.4640	1.4646	1.4653
88	1.4660	1.4667	1.4674	1.4681	1.4688	1.4695	1.4702	1.4709	1.4716	1.4723
89	1.4730	1.4737	1.4744	1.4751	1.4758	1.4765	1.4772	1.4779	1.4786	1.4793
90	1.4800	1.4807	1.4814	1.4821	1.4828	1.4835	1.4842	1.4849	1.4856	1.4863
91	1.4870	1.4877	1.4884	1.4891	1.4898	1.4905	1.4912	1.4919	1.4926	1.4934
92	1.4941	1.4948	1.4955	1.4962	1.4969	1.4976	1.4983	1.4990	1.4997	1.5001
93	1.5012	1.5019	1.5026	1.5033	1.5040	1.5047	1.5054	1.5061	1.5069	1.5076
94	1.5083	1.5090	1.5097	1.5104	1.5112	1.5119	1.5126	1.5133	1.5140	1.5147
95	1.5155	1.5162	1.5169	1.5176	1.5183	1.5191	1.5198	1.5205	1.5212	1.5219
96	1.5227	1.5234	1.5241	1.5248	1.5255	1.5263	1.5270	1.5277	1.5284	1.5292
97	1.5299	1.5306	1.5313	1.5321	1.5328	1.5335	1.5342	1.5350	1.5357	1.5364
98	1.5372	1.5379	1.5386	1.5393	1.5401	1.5408	1.5415	1.5423	1.5430	1.5437
99	1.5445	1.5452	1.5459	1.5467	1.5474	1.5481	1.5489	1.5496	1.5503	1.5511
100	1.5518									



TEMPERATURE CORRECTIONS TO SACCHAROMETER READINGS  
(STANDARD AT 20° C.).\*

Temperature in Degrees Centigrade.	Observed Per Cent of Sugar.													
	0	5	10	15	20	25	30	35	40	45	50	55	60	70
	Subtract from Observed Per Cent.													
0	0.30	0.49	0.65	0.77	0.89	0.99	1.08	1.16	1.24	1.31	1.37	1.41	1.44	1.49
5	0.36	0.47	0.56	0.65	0.73	0.80	0.86	0.91	0.97	1.01	1.05	1.08	1.10	1.14
10	0.32	0.38	0.43	0.48	0.52	0.57	0.60	0.64	0.67	0.70	0.72	0.74	0.75	0.77
11	0.31	0.35	0.40	0.44	0.48	0.51	0.55	0.58	0.60	0.63	0.65	0.66	0.68	0.70
12	0.29	0.32	0.36	0.40	0.43	0.46	0.50	0.52	0.54	0.56	0.58	0.59	0.60	0.62
13	0.26	0.29	0.32	0.35	0.38	0.41	0.44	0.46	0.48	0.49	0.51	0.52	0.53	0.55
14	0.24	0.26	0.29	0.31	0.34	0.36	0.38	0.40	0.41	0.42	0.44	0.45	0.46	0.47
15	0.20	0.22	0.24	0.26	0.28	0.30	0.32	0.33	0.34	0.36	0.36	0.37	0.38	0.39
16	0.17	0.18	0.20	0.22	0.23	0.25	0.26	0.27	0.28	0.28	0.29	0.30	0.31	0.32
17	0.13	0.14	0.15	0.16	0.18	0.19	0.20	0.20	0.21	0.21	0.22	0.23	0.23	0.24
18	0.09	0.10	0.10	0.11	0.12	0.13	0.13	0.14	0.14	0.14	0.15	0.15	0.15	0.16
19	0.05	0.05	0.05	0.06	0.06	0.06	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08
17.5	0.11	0.12	0.12	0.14	0.15	0.16	0.16	0.17	0.17	0.18	0.18	0.19	0.19	0.20
15.56 (60° F.)	0.18	0.20	0.22	0.24	0.26	0.28	0.29	0.30	0.30	0.32	0.33	0.33	0.34	0.34
	Add to Observed Per Cent.													
	21	22	23	24	25	26	27	28	29	30	35	40	45	50
	55	60	65	70	75	80								
21	0.04	0.05	0.06	0.06	0.06	0.07	0.07	0.07	0.07	0.08	0.08	0.08	0.08	0.09
22	0.10	0.10	0.11	0.12	0.12	0.13	0.14	0.14	0.15	0.15	0.16	0.16	0.16	0.16
23	0.16	0.16	0.17	0.17	0.19	0.20	0.21	0.21	0.22	0.23	0.24	0.24	0.24	0.24
24	0.21	0.22	0.23	0.24	0.26	0.27	0.28	0.29	0.30	0.31	0.32	0.32	0.32	0.32
25	0.27	0.28	0.30	0.31	0.32	0.34	0.35	0.36	0.38	0.38	0.39	0.39	0.40	0.39
26	0.33	0.34	0.36	0.37	0.40	0.40	0.42	0.44	0.46	0.47	0.47	0.48	0.48	0.48
27	0.40	0.41	0.41	0.44	0.46	0.48	0.50	0.52	0.54	0.54	0.55	0.56	0.56	0.56
28	0.46	0.47	0.49	0.51	0.54	0.56	0.58	0.60	0.61	0.62	0.63	0.64	0.64	0.64
29	0.54	0.55	0.56	0.59	0.61	0.63	0.66	0.68	0.70	0.70	0.71	0.72	0.72	0.72
30	0.61	0.62	0.63	0.66	0.68	0.71	0.73	0.76	0.78	0.78	0.79	0.80	0.80	0.81
35	0.99	1.01	1.02	1.06	1.10	1.13	1.16	1.18	1.20	1.21	1.22	1.22	1.23	1.22
40	1.42	1.45	1.47	1.51	1.54	1.57	1.60	1.62	1.64	1.65	1.65	1.65	1.66	1.65
45	1.91	1.94	1.96	2.00	2.03	2.05	2.07	2.09	2.10	2.10	2.10	2.10	2.10	2.08
50	2.46	2.48	2.50	2.53	2.56	2.57	2.58	2.59	2.59	2.58	2.58	2.57	2.56	2.52
55	3.05	3.07	3.09	3.12	3.12	3.12	3.12	3.11	3.10	3.08	3.07	3.05	3.03	2.97
60	3.69	3.72	3.73	3.73	3.72	3.70	3.67	3.65	3.62	3.60	3.57	3.54	3.50	3.43
65	4.4	4.4	4.4	4.4	4.4	4.4	4.3	4.2	4.2	4.1	4.1	4.0	4.0	3.9
70	5.1	5.1	5.1	5.0	5.0	5.0	4.9	4.8	4.8	4.7	4.7	4.6	4.6	4.4
75	6.1	6.0	6.0	5.9	5.8	5.8	5.7	5.6	5.5	5.4	5.4	5.3	5.2	5.0
80	7.1	7.0	7.0	6.9	6.8	6.7	6.6	6.4	6.3	6.2	6.1	6.0	5.9	5.6

\* U. S. Dept. of Commerce and Labor, Bur. of Standards, Circular 44, 1913, p. 129. This table is calculated using the data on thermal expansion of sugar solutions by Plato (Wiss. Abh. der Kaiserlichen Normal-Eichungs-Kommission, 2, 1900, p. 140), assuming the instrument to be of Jena 16<sup>III</sup> glass. The table should be used with caution and only for approximate results when the temperature differs much from the standard temperature or from the temperature of the surrounding air.



plying the reading by 2, both direct and invert. Use the Clerget-Herzfeld formula for calculation of the sucrose.

For medium- or light-colored grades of molasses, which yield but a small precipitate with lead subacetate, the above method of simple polarization, both direct and invert, gives results sufficiently accurate for ordinary work. For dark-colored, or "black-strap" molasses, or wherever extreme accuracy is required, the solution should be first made up to the mark and then clarified by the addition of a slight excess of anhydrous lead subacetate (p. 610), as proposed by Horne, or else the double dilution method of Wiley should be employed. Both methods make due allowance for the volume of the precipitate.

*Double Dilution Method.\**—Take half the normal weight of the sample and make up the solution to 100 cc., using the appropriate clarifier. Take the normal weight of the sample and make up a second solution with the clarifier to 100 cc. Filter and obtain direct polariscopic readings of both solutions. Invert each in the usual manner and obtain the invert reading of the two.

The true direct polarization of the sample is the product of the two direct readings divided by their difference. The true invert polarization is the product of the two invert readings divided by their difference.

**Determination of Raffinose in Beet Sugar Molasses.**—For the determination of sucrose and raffinose when present in the same solution, use the following formulas of Creydt as modified by Browne † to correspond with the Clerget-Herzfeld method of inversion:

$$S = \frac{0.5124a - b}{0.839},$$

and

$$R = \frac{a - S}{1.852},$$

or

$$R = \frac{0.3266a + b}{1.554},$$

where  $S$  = per cent of sucrose,  $R$  = per cent of raffinose,  $a$  = direct reading, and  $b$  = reading after inversion.

\* Wiley and Elwell, *Analyst*, 1896, 21, p. 184.

† *Handbook of Sugar Analysis*, New York, 1912, p. 283.



Davoll \* recommends for purposes of clarification of the molasses the use of powdered zinc after inversion of the molasses sample according to Clerget's method. He adds 1 gram of the zinc to the sample after inversion while at the temperature of 69° C., allowing it to act for three to four minutes at that temperature, after which he cools and filters, with the production of an almost colorless solution.

**Determination of Reducing Sugar.**—(*Estimated as Dextrose.*)—Dilute 5 grams of molasses or syrup with water in a 100-cc. graduated flask, using 2 to 5 cc. normal lead acetate. Make up to 100 cc., filter, take an aliquot part of the filtrate (25 to 50 cc.) and make this up to 100 cc., the amount taken being such that, when diluted, the solution will contain not more than  $\frac{1}{2}\%$  of dextrose. Since lead acetate has been used to clarify, add to the aliquot part taken and before dilution, enough sodium sulphate to precipitate the excess of lead, then filter and make up to the 100 cc. mark.

Determine the reducing sugar in this solution by either volumetric or gravimetric Fehling processes.

**U. S. Standard Molasses** is molasses containing not more than 25% of water, nor more than 5% of ash.

**Adulteration of Molasses and Syrups.**—A common adulterant of all these products is commercial glucose. From its water-white color and inert sweetness, no less than from its cheapness, it forms an admirable adulterant for dark-colored or low-grade molasses and syrups, counteracting to a great extent by its smoothness the strong and often disagreeable taste of the inferior products with which it is mixed. Thus a grade of molasses too cheap to be ordinarily used for food purposes can be made to assume the appearance, and to some extent the taste, of the higher-priced and light-colored grades, by admixture with commercial glucose.

Tin salts are also used to improve the color of low-grade or dark molasses, and bleaching agents, such as sulphurous acid, are frequently employed. Copper is sometimes found, due to utensils or vessels used in processes of manufacture.

Lead may occur in maple syrup, due to the leaden plugs or spigots through which the sap is sometimes drawn from the trees.

**Detection and Determination of Commercial Glucose.**†—From the direct polarization of a normal solution of molasses or syrup the presence

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\* Jour. Am. Chem. Soc., 25 (1903), p. 1019.

† Leach, *ibid.*, p. 982.



or absence of commercial glucose can usually be established. The direct polarization of a normal solution of pure molasses should not be much in excess of  $50^{\circ}$  on the Soleil-Ventzke scale, while a pure, dark-colored molasses should polarize well under  $40^{\circ}$ . Golden syrup and maple syrup read higher than molasses, and a normal solution of pure maple syrup may have a direct polarization as high as  $65^{\circ}$ , being more often than not above  $60^{\circ}$ .

An excessively high direct polarization is at once an indication of the presence of commercial glucose, while an invert reading at ordinary room temperature to the right of the zero-point is an almost positive proof of its presence in either of the above products.

The optically active constituents of commercial glucose, viz., dextrin, maltose, and dextrose, are present in such varying amounts, that it is impossible to determine accurately the exact amount of this adulterant in complex saccharine products which themselves contain components common to glucose. Its approximate amount can, however, be very satisfactorily estimated in molasses and syrups by the use of the following formula:

$$G = \frac{(a - S)100}{175}, *$$

where  $G$  = per cent of commercial glucose,  $a$  = direct polarization, and  $S$  = per cent of cane sugar previously obtained from the Clerget-Herzfeld formula. A large amount of invert sugar present affects the accuracy of this formula. It is especially applicable to maple syrup, wherein the per cent of invert sugar is small, but may be applied also to molasses and golden syrup, wherein the amount of invert sugar is not so large but that results may be obtained as close as it could be expected from an empirical formula.†

In saccharine products containing considerable invert sugar the invert reading at  $87^{\circ}$  C. obtained as directed on page 671, is divided by

\* Leach, U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 48.

† This formula is based on the assumption that  $42^{\circ}$  Bé. mixing glucose, the grade specially made and used for admixture with molasses, syrups, and honey, has a maximum polarization of  $175^{\circ}$  V. It was adopted as a result of investigations made some years ago by the author, but subsequently it appeared that  $42^{\circ}$  Bé. mixing glucose polarizes lower than formerly. Thus a sample recently examined by the author polarized at  $162.4^{\circ}$  V. Pending further investigations it seems best for the present to retain the old formula, for, while it undoubtedly gives low results, especially with higher admixtures of glucose, it approximates the truth more closely than would be expected, perhaps because it tends to compensate for the error due to substances in genuine molasses and honey that polarize to the right after inversion. Furthermore, it has been adopted by the A. O. A. C. To avoid misunderstanding, express results in terms of glucose polarizing at that factor.



the appropriate factor (163) to obtain the percentage of commercial glucose.

While theoretically pure molasses and syrups would be expected to show no rotation when polarized at 87° C. after inversion, as a matter of fact most samples exhibit a decidedly right-handed reading at that temperature. Occasionally a zero reading is noted, and in rare instances a slight left-handed rotation occurs under the above conditions.

Dextro-rotation is undoubtedly caused by some form of decomposition or fermentation. It may be due to a preponderance of dextrose in the reducing sugars, since levulose is more easily decomposed than dextrose, or it may be caused by the decomposition products formed when the raw juice is being defecated with lime, or again it might result from a special fermentation forming dextran.

The following table shows results by A. H. Bryan\* of polarization of samples of Louisiana molasses and syrup of known purity, showing especially the invert readings at 87° C.:

POLARIZATION OF LOUISIANA MOLASSES AND SYRUP.

MOLASSES.				SYRUP.			
Direct Polarization at 20° C.	Corrected Invert Polarization—		Dry Substance.	Direct Polarization at 20° C.	Corrected Invert Polarization—		Dry Substance.
	At 20° C.	At 87° C.			At 20° C.	At 87° C.	
° V.	° V.	° V.	Per Cent.	° V.	° V.	° V.	Per Cent.
40.8	-20.24	+2.2	80.8	48.4	-17.6	+1.98	74.3
24.6	-20.9	+2.2	76.8	54.0	-18.7	+3.30	68.3
26.0	-18.26	+3.52	76.8	50.2	-12.1	+6.16†	
42.4	-16.94	+2.42	78.2	50.4	-14.3	+1.76	
52.4	-16.28	+2.20	69.1	61.8	-16.5	+2.20	
55.6	-13.59	+4.18	69.6				
39.6	-18.04	+2.20	80.8				
39.6	-17.82	+2.20	79.0				
44.0	-17.16	+2.64	72.0				
42.0	-17.60	+2.42	73.8				
42.4	-17.27	+3.52	76.1				
41.6	-16.94	+3.96	74.0				
52.4	-17.60	+3.52	76.1				
26.6	-19.8	0.00	78.1				
50.8	-25.08	+1.10	87.5				
22.6	-16.72	+3.96	84.1				
41.6	-14.74	+1.10	75.0				
45.6	-15.4	+2.20	78.0				
				Average.....		+2.65	
				Maximum . . . . .		+6.16	
				Minimum .....		0.00	

\* A. O. A. C. Proc., 1908, U. S. Dept. of Agric., Bur. of Chem., Bul. 122, p. 182.

† Sampleropy and badly fermented.



**TYPICAL ANALYSES OF MOLASSES AND SYRUPS ADULTERATED WITH COMMERCIAL GLUCOSE.**

	Polarization.			Per Cent Sucrose (Clerget's Formula).	Reducing Sugar (Dex- trose).	Commercial Glucose (Leach's Formula).	Moisture.	Ash.
	Direct.	Invert.	Tempera- ture.					
(a) Molasses, .....	62	+36.3	18°	19	30.03	24.6	29.36	3.8
(b) " .....	98.7	+71.9	18°	19.9	27.62	45.0	27.98	3.5
(c) " .....	109.7	+90	17°	14.5	33.11	54.4	22.02	2.6
(a) Golden drip syrup..	73.5	+39.8	18°	25	31.61	27.7	23.67	3.9
(b) " " " ..	109.4	+87.6	17°	16.9	33.44	52.8	24.48	2.5
(c) " " " ..	143.6	+136.0	18.4°	5.6	38.17	78.5	21.52	1.0
(a) Maple syrup.....	76.3	+7.6	18.6°	51	10.55	14.4	31.91	0.6
(b) " " .....	77.9	+24	19°	40.1	.....	21.6	23.44	.....
(c) " " .....	87.0	+30.6	22.4°	42.5	16.90	25.4	28.80	1.0

**Determination of Dextrin.**—According to Beckman's method weighed amount of the honey or molasses is diluted with an equal volume of water and from ten to twelve times its volume of methyl alcohol added. The precipitated dextrin is collected in a tared filter and thoroughly washed with methyl alcohol, after which it is dried and weighed.

**Reduction of Saccharine Products to an Ash for Mineral Analysis.**—If a considerable quantity of molasses, syrup, or other saccharine substance is to be burnt to an ash, it is both tedious and annoying to ignite directly, by reason of the excessive swelling and frothing of such substance during ignition. Small quantities of molasses, syrup, or honey may with care be reduced to an ash by the method described on page 609.

If a readily controlled electric current is available, it may be utilized as follows: \* Mix 100 grams of molasses, syrup, or other saccharine solution, which should be evaporated to syrupy consistency if not already such, with about 35 grams of concentrated sulphuric acid in a large porcelain evaporating-dish. An electric current is then passed through it while stirring, by placing one platinum electrode in the bottom of the dish near one side and attaching the other to the lower end of the glass rod, with which the contents are stirred. Begin with a current of about 1 ampere and gradually increase to 4.† In from ten to fifteen minutes

\* Leach, 32d An. Rept. Mass. State Board of Health (1900), p. 653. Reprint, p. 37. This method is preferred to the ordinary method of heating with sulphuric acid, especially in case of molasses, because, if properly manipulated, it so quietly comes into the form of a very finely divided char or powder, especially adapted for subsequent quick ignition.

† Modified from method of Budde and Schou for determining nitrogen electrolytically. Ztschr. anal. Chem., 38 (1899), p. 345.



the mass is reduced to a fine, dry char, which may then be readily burnt to a white ash in the original dish over a free flame or in a muffle.

Or, 100 grams of the molasses or syrupy solution to be ashed may be first evaporated to dryness and afterward mixed with from 10 to 20 cc. of concentrated sulphuric acid in a porcelain evaporating-dish, or if the substance to be ashed be a dry sugar or confectionery, 20 grams are mixed with the above amount of acid. Heat is gently applied by means of the gas flame till the swelling and frothing have ceased, which usually requires only a few minutes. The final ignition is then accomplished in the usual manner, nitric acid being added if necessary to completely destroy the organic matter.

**Determination of Tin in Molasses.**—Fuse the ash from a weighed portion of the sample with sodium hydroxide in a silver crucible, dissolve in water, and acidulate with hydrochloric acid; filter and precipitate the tin from this solution with hydrogen sulphide; wash the precipitate on a filter and dissolve it in an excess of ammonium sulphide. Filter this solution into a tared platinum dish, and deposit the tin directly in the dish by electrolysis, using a current of 0.05 ampere and the apparatus described on page 634.

**Distinction between Invert Sugar, Maltose, and Lactose.\***—All these sugars reduce Fehling's solution. Dextrose and levulose (invert sugar) when boiled with Barfoed's copper acetate solution (14 grams crystallized copper acetate and 5 cc. acetic acid in 200 cc. water) will form a precipitate of cuprous oxide, while neither maltose nor lactose will do this. The solution, which has thus been tested for invert sugar and found to be free, or the filtrate from the cuprous oxide precipitate, is treated with an excess of basic lead acetate, filtered, and to the filtrate is added an excess of sodium sulphate solution to precipitate the lead. The solution is again filtered and treated with copper sulphate solution, if not already blue. It is then made alkaline with sodium hydroxide and heated to boiling. A red precipitate of cuprous oxide at this stage indicates either lactose or maltose or both.

A solution of the sugar, made strongly ammoniacal, is then mixed with alkaline bismuth solution† and the container is set in a water-bath at 60° C. Maltose soon reduces the bismuth, but lactose does not.

To test for lactose, add strong nitric acid to the solid sugar residue

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\* Bartley and Mayer, Merck's Report, 12 (1903), p. 100.

† This reagent is prepared as follows: Bismuth subnitrate, 2 grams; Rochelle salt, 4 grams; sodium hydroxide, 8 grams; dissolved in 100 cc. of water by the aid of heat.



and warm gently till red fumes come off. Then set the container in hot water and cool gradually. Crystals of mucic acid appear after a time if any appreciable amount of lactose be present.

**Determination of Lactose or Maltose.**—Either sugar, if in solution free from other reducing sugars, may be determined by the volumetric Fehling method (page 615) or by the Defren method, using the table on page 619.

For the determination of maltose in commercial glucose, see page 661.

**Estimation of Cane Sugar and Dextrose in Mixtures.**—Obtain true direct and invert readings of a normal solution of the mixture. Determine the per cent of sucrose by Clerget-Herzfeld formula. This figure represents the right-handed rotation due to a sucrose. Subtracting this from the direct polarization, the difference represents the right-handed rotation due to dextrose. The specific rotatory power of sucrose is 66.5 and that of dextrose 52.76.

Calling  $d$  the percentage of dextrose and  $R'$  the right-handed rotation due to dextrose as above obtained, if the Soleil-Ventzke scale is used,

$$66.5 : 52.76 = d : R',$$

whence

$$d = \frac{66.5R'}{52.76}.$$

#### ANALYSIS OF MAPLE PRODUCTS.

**Preparation of Sample.**—Syrups are analyzed in the condition they are placed on the market, rejecting any sediment which may have settled out. Jones has noted that if maple sugar is analyzed in its commercial form the results would include mineral matter and other insoluble constituents which might invite a considerable admixture of ordinary sugar. It is therefore important to carry out the analysis on a syrup prepared according to Bryan\* as follows:

Dissolve 100 grams of the sugar in at least 200 cc. of water and boil down to 65% of solids. If the solution is cloudy, filter after the liquid has been boiled down to about 30% of solids, then complete the concentration. Allow to stand at 20° C. for two days and decant from the sediment.

**Determination of Moisture.**—This is accomplished by direct drying with sand, or by calculation from the specific gravity, or, preferably from the refractive index. See molasses methods, page 643.

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\* A. W. Bryan, U. S. Dept. of Agric., Bur. of Chem., Circ. 40, p. 6.



**Determination of Ash.**—Burn 5 grams in a platinum dish by the usual method, observing the precautions given for molasses, page 644.

*Soluble and Insoluble Ash.\**—To the platinum dish containing the ash add 40 cc. of hot water and boil gently for two minutes. Filter through a small ashless filter, and wash with hot water until the filtrate amounts to 100 cc. Return the filter to the dish used for ashing, burn at a low red heat, cool and weigh, thus obtaining the insoluble ash. The soluble ash is obtained by difference, subtracting the weight of insoluble from that of total ash.

*Alkalinity of Soluble Ash.\**—Allow the filtrate from the above determination to cool, then titrate with tenth-normal hydrochloric acid, using methyl orange as an indicator.

*Alkalinity of Insoluble Ash.\**—Add excess of tenth-normal hydrochloric acid (usually 10 cc.) to the ignited insoluble ash in the platinum dish, boil gently, cool and titrate with tenth-normal sodium hydroxide, using methyl orange as an indicator.

Express the alkalinity in each case as the number of cubic centimeters of tenth-normal acid on the ash of 1 gram of sample.

**Determination of Sucrose.**—Calculate by Clerget-Herzfeld formula (page 610). Use 5 cc. of alumina cream but no lead subacetate except when necessary and then but 1 cc.

**Determination of Reducing Sugar.**—Follow Defren-O'Sullivan or Munson and Walker method (pages 618 and 622).

**Determination of Malic Acid Value.**—*Leach and Lythgoe Method,† modified by Cowles.‡*—The modified method differs from the original chiefly in that no ammonia is added and calcium acetate is substituted for calcium chloride; it gives slightly higher results.

Weigh 6.7 grams of the sample in a sugar dish, transfer to a 200-cc. beaker with 5 cc. of water, add 2 cc. of a 10% calcium acetate solution, and shake. Stir in 100 cc. of 95 per cent alcohol and warm the solution until the precipitate settles, leaving the supernatant liquid clear. Filter off the precipitate and wash with 75 cc. of 85% alcohol, dry the filter paper, and ignite in a platinum dish. Add 10 cc. of tenth-normal hydrochloric acid and warm gently until all the lime dissolves. Cool and titrate back with tenth-normal sodium hydroxide, using methyl orange as an indicator. One-tenth of the number of cubic centimeters of tenth-normal acid is the

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\* A. H. Bryan, U. S. Dept. of Agric., Bur. of Chem., Circ. 40, p. 6.

† Jour. Amer. Chem. Soc., 26, 1904, pp. 380, 1536.

‡ Ibid., 30, 1908, p. 1285; U. S. Dept. of Agr., Bul. 466, 1917, p. 11.



malic acid value. Run a blank determination with each set of determinations, using the same amount of reagents, and subtract the result obtained from the malic acid number.

**Determination of Lead Number.**—*Winton Method.*\*—Weigh 25 grams of the material (or 26 grams if a portion of the filtrate is to be used for polarization) and transfer by means of boiled water into a 100-cc. flask. Add 25 cc. of standard lead subacetate solution, fill to the mark, shake, allow to stand at least three hours and filter through a dry filter. From the clear filtrate, pipette off 10 cc., dilute to 50 cc., add a moderate excess of sulphuric acid, and 100 cc. of 95% alcohol. Let stand over night, filter on a Gooch crucible, wash with 95% alcohol, dry at a moderate heat, ignite at low redness for three minutes, taking care to avoid the reducing cone of the flame, cool, and weigh. Calculate the amount of lead in the precipitate, using the factor 0.6831, subtract this from the amount of lead in 2.5 cc. of the standard solution, multiply the remainder by 100, and divide by 2.5, thus obtaining the lead number.

The standard lead subacetate is prepared by diluting one part of the ordinary solution (page 610) with four volumes of water, filtering if not clear. It is standardized by a blank determination conducted as above described, but acidifying with a few drops of acetic acid before making up to volume, as recommended by A. H. Bryan. The solution deposits a slight precipitate on standing, but this does not usually appreciably affect its strength.

The range in lead number of maple products calculated to the dry basis is given on pages 593 and 594; Snell and Scott have shown, however, that the range in the case of syrups is narrower when the comparison is made on the wet basis.

Adding of cane sugar reduces the lead number to a greater degree than the percentage of admixture thus rendering the fact more apparent.

*Ross Modification.*†—This process yields higher results than the original methods (see page 594). The number of mixtures of maple and cane syrups (or sugars) is proportional to the admixture.

Transfer 25 grams of the syrup to a 100-cc. flask, using about 25 cc. of freshly boiled water, add 10 cc. of potassium sulphate solution (7 grams per liter), then 25 cc. of lead subacetate solution of the strength employed in the foregoing method. Make up to the mark with boiled water and proceed as in the Winton method.

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\* Jour. Am. Chem. Soc., 28, 1906, p. 1204.

† U. S. Dept. of Agric., Bur. of Chem., Circ. 53.



Run the blank in exactly the same way, substituting 25 grams of pure cane sugar syrup (66 grams of sucrose dissolved in 34 grams of water) for the maple syrup.

*McGill or Canadian Method.\**—To 5 grams of the dry sugar, or its equivalent in syrup, dissolved in water and made up to 20 cc., add 2 cc. of lead subacetate solution, mix and allow to stand for two hours. Filter on a Gooch crucible, or sugar tube packed with asbestos, wash 4 to 5 times with hot water, dry, and weigh. Multiply the weight by 20 to obtain the lead number.

In genuine maple syrups McGill found a range of 1.37 to 6.56 for 456 samples (using 5 grams of syrup), Snell and Scott a range of 1.74 to 7.50 for 126 samples (using 5 grams of dry matter).

*Snell, MacFarlane, and Von Zoeren Volumetric Method.†*—Dilute the syrups with water, boil until the temperature reaches 219° F. and filter through cotton wool. After cooling, dilute 10 cc. to 100 cc. with distilled water, and measure 60 cc. of the diluted solution into a 100-cc. beaker.

Measure the electrical resistance using a dip electrode as described for the Snell method of determining electrical resistance (page 661). Maintaining the temperature constant, add 1 cc. of lead acetate solution (a filtered solution of Horne's lead subacetate sp.gr. 1.033) from a burette, stir well, and again measure the electrical resistance. Continue the addition in this manner, 1 cc. at a time, until 10 cc. have been added. Plot the resistances found against the quantities of subacetate solution used. If the syrup is genuine the results of the plot will be two intersecting straight lines. In 70 genuine maple syrups examined by the originators of the method the intersections fell between 4.8 and 6.6 cc.

Pure maple sugars converted into syrups give practically the same values as pure syrups.‡

**Determination of Volume of Lead Precipitate.§—Hortvet Method.**—The apparatus consists of (1) a tube, 15.3 cm. in length, made up of a wide cylindrical portion 3 cm. in diameter, narrowed at the top to a neck 2 cm. in diameter, and at the bottom to a stem graduated in tenths to 5 cc. and (2) a holder, made of pine or white wood, of a size adapted to carry the tube in the shield of the centrifuge. The holders and tubes should be arranged in balanced pairs in the centrifuge.

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\* Lab. Ind. Rev. Dept., Ottawa, Bul. 228, 1911, p. 5.

† Jour. Ind. Eng. Chem., 8, 1916, p. 241.

‡ Ibid., 8, 1916, p. 421.

§ Jour. Am. Chem. Soc., 26, 1904, p. 1532.



Introduce 5 cc. of syrup or 5 grams of sugar into the tube. Add 10 cc. of water, and dissolve completely. Next add 10 drops of alumina cream, and 1.5 of lead subacetate. Shake thoroughly, and allow to stand from forty-five to sixty minutes. Place the tube in its holder in the centrifuge shield, and run six minutes. If, after the end of this time, any material adheres to the sides of the wide part of the tube, loosen with a small wire or by giving the tube a slight twist, then run the tube six additional minutes, and finally read the volume of the precipitate in the stem, estimating to 0.01 cc.

Run a blank with the above reagents in water, subtracting the blank reading from that of the precipitate. In the case of syrup, reduce to the 5-gram basis by dividing by the specific gravity of the sample. If the sugar content of the sample is known, the specific gravity can be calculated from the table on page 648. For pure maple syrup 1.33 is very nearly correct.

The centrifuge used by Hortvet had a radius of 18.5 cm. and was run at a speed of 1600 revolutions per minute. The corresponding velocity in cm. per second ( $v$ ) and revolutions per minute ( $R$ ) for any given centrifuge with a radius of  $r$  cm. may be calculated by the following formulae:

$$v = \sqrt{520,000r}, \quad R = 60v / 2\pi r.$$

Results by Hortvet on pure maple syrups vary from 1.2 cc. to about 2.5 cc., and on pure maple sugars from 1.8 to 4 cc.

Commercial brands of adulterated syrups and sugars give such precipitates as 0.00 cc., 0.02 cc., 0.05 cc., and 0.08 cc. Hortvet regards with suspicion a syrup testing lower than 1.2 cc., and when the result is below 1 cc., the sample is positively condemned as being mixed with refined cane sugar. In the case of sugar, a somewhat higher minimum figure is adopted than with syrup. In view of the fact that the speed has much to do with the volume of the precipitate, the analyst should make a series of similar experiments with his own centrifuge, and work out his own standards. Results may be better compared with each other, if calculated on the water-free basis.

In case of doubt, and in fact in all cases at first, it would be well to make confirmatory tests, such as determining the ash and reducing sugar.

**Sy's Lead Method.\***—In a 25-cc. graduated cylinder introduce 5 cc. of syrup, or 5 grams of sugar which is afterwards dissolved in a little

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\* Jour. Am. Chem. Soc., 30, 1908, p. 1430.



water. Add water to the 15 cc. mark and 2 cc. of lead subacetate solution. Shake thoroughly and allow the mixture to stand twenty hours. Then read the volume of the precipitate, which for pure maple products should be at least 3 cc. and is usually over 5 cc.

**Determination of Electrical Conductivity Value.**—*Snell Method.\**—Measure out into a small beaker (or directly into the conductivity cell) 20 cc. of the syrup, allowing thorough draining. Using the same graduate, add two successive portions of water, each equal in volume to the syrup taken. Mix thoroughly, pour into conductivity cell, bring to 25° C., and make the measurement. Divide the constant of the cell by the observed number of ohms and multiply the result by 100,000.

Genuine syrups examined by Snell and co-worker have given values of 96 to 230.

The essential features of the apparatus are:

1. A low voltage electrical current operating an induction coil.
2. A conductivity cell of a form suitable for liquids of low conductivity, and with electrodes not easily displaced.†
3. A Wheatstone bridge with telephone.
4. A device for exact regulation of temperature.

#### ANALYSIS OF COMMERCIAL GLUCOSE.

Wiley ‡ has worked out a method for calculating the percentage of dextrin, maltose, and dextrose present in commercial glucose, based on the specific rotatory power of these substances and on the reducing power of maltose and dextrose. To apply this method, the operator, if he has a polariscope reading in sugar scale degrees, must ascertain the equivalent readings in angular degrees from the table on page 606, and calculate the specific rotatory power in each case from the formula

$$(\alpha)_D = \frac{100a}{cl}, \text{ page 607.}$$

Thus, if he possesses a Schmidt and Haensch instrument, he should multiply the true reading, as obtained on that instrument, with a normal solution of the given sugar or mixture, by the factor 0.3468, to convert the reading into circular degrees from which to figure the specific rotatory power as above.

\* Jour. Ind. Eng. Chem., 5, 1913, p. 740.

† Van Zoeren, Jour. Amer. Chem. Soc., 38, 1916, p. 652.

‡ Chem. News, 46, p. 175; Agric. Anal., 3, pp. 288-290.



The specific rotatory power of dextrin is fixed at 193, that of maltose at 138, and that of dextrose at 53.

Then if  $P$  = total polarization of the mixture in terms of specific rotatory power,  $d$  = per cent dextrose,  $m$  = per cent maltose, and  $d'$  = per cent dextrin,

$$P = 53d + 138m + 193d'. \quad \dots \dots \dots (1)$$

The value of  $P$  is obtained from observation and calculation as above described on a known solution of the sample, say 10 grams in 100 cc. The reducing sugars, maltose and dextrose, are then removed, preferably by oxidation with cyanide of mercury, as follows.\*

Prepare the reagent by dissolving 120 grams mercuric cyanide and 120 grams sodium hydroxide in water, mixing the two solutions, and making up to 1000 cc. Remove any precipitate that may gather by filtration.

Make a solution of 10 grams of the glucose sample in 100 cc. and take 10 cc. of this solution in a 50-cc. graduated flask. Add sufficient mercuric cyanide solution to have an excess of reagent after the oxidation (from 20 to 25 cc.), and boil for three minutes under a hood with a good draft. Cool and neutralize the alkali with concentrated hydrochloric acid, adding the latter till the brown color is discharged. By this method the optical activity of the maltose and dextrose is discharged, while that of the dextrin remains unaffected. From the polariscope reading calculate as above the specific rotatory power of the dextrin ( $P'$ ). Then

$$P' = 193d'. \quad \dots \dots \dots (2)$$

The reducing power on Fehling's solution of dextrose is to that of maltose as 100 is to 62. Whence, if  $R$  = reducing sugar (reckoned as dextrose) we have

$$R = d + 0.62m. \quad \dots \dots \dots (3)$$

Subtracting equation (2) from equation (1) we have

$$P - P' = 53d + 138m. \quad \dots \dots \dots (4)$$

Multiplying equation (3) by 53 and subtracting from equation (4),

$$\begin{aligned} P - P' &= 53d + 138m, \\ 53R &= 53d + 32.86m, \\ \hline P - P' - 53R &= 105.14m. \quad \dots \dots \dots (5) \end{aligned}$$

\* Wiley, Agric. Anal., p. 290.



Therefore

$$m = \frac{P - P' - 53R}{105.14}, \quad \dots \dots \dots (6)$$

$$d = R - 0.62m, \quad \dots \dots \dots (7)$$

$$d' = \frac{P'}{193}, \quad \dots \dots \dots (8)$$

**Determination of Dextrin in Commercial Glucose.**—One volume of the sample is well shaken with about 10 volumes of 90% alcohol, and the precipitated dextrin is separated by filtration through a tared filter, washed thoroughly with strong alcohol, dried at 100°, and weighed.

**Qualitative Tests for Commercial Glucose.**—Several confirmatory chemical tests may be employed for commercial glucose, aside from the optical test with the polariscope. Thus a precipitate of dextrin by treatment of the sample with an excess of strong alcohol, in the absence of mineral salts insoluble in alcohol, is strongly indicative of commercial glucose. An excess of sodium chloride in the ash also points strongly to the presence of glucose.

**Determination of Ash.**—Formerly, when sulphuric acid was used for conversion of the starch, the ash consisted largely of calcium sulphate, but at present when hydrochloric acid is almost exclusively used the mineral matter is almost entirely common salt, formed by the neutralization of the acid.

Determine ash by burning in a platinum dish at dull redness as in the case of other saccharine products. Qualitative or quantitative tests may be made for chloride, in the latter case calculating the equivalent amount of sodium chloride. If the amount of sodium chloride found does not equal the total ash, sulphates may be looked for.

**Determination of Sulphurous Acid.**—At the present time glucose usually is free from an appreciable amount of sulphurous acid which formerly was extensively employed for bleaching. It may be determined by distillation, oxidation to sulphuric acid, and precipitation with barium chloride as described on page 313.

**Detection of Arsenic.**—Since the Manchester epidemic of arsenical poisoning, due to the consumption of beer prepared from glucose contaminated through the sulphuric acid with this poison, it is highly important that both the acid used for conversion and the glucose be frequently tested for this contamination.



The tests may be made on 2 to 5 grams of the materials without charring or destruction of the organic matter, by the Marsh test or the Sanger-Black-Gutzeit test as described under general methods on pages 63 to 66.

The English limit of one and one-half parts per million calculated as metallic arsenic should not be exceeded.

### HONEY.

**Composition and Occurrence.**—Honey is the saccharine product deposited by bees (*Apis mellifica* and *A. dorsata*) in the cells of honey-comb, which the insect forms out of wax secreted by its body. Honey has its source chiefly in the nectares of flowers, from which the bees abstract it, also in the juices of ripe fruits and the exudations of leaves (honeydew). While in the honey-sac of the bee, the sucrose, which forms the chief constituent of the fruit juice or nectar, becomes for the most part inverted, forming, in the honey, dextrose and levulose. The evaporation to a syrupy consistency is effected in the hive by exposure to a current of air, produced by fanning of the wings of the bees.

The flavor of honey varies considerably, according to its source. Besides water and the sugars named honey contains dextrin and small amounts of protein, mineral matter (including phosphates), and organic acids. Pollen is usually present, also as a rule a small quantity of wax. Fincke \* states real honey may or may not contain formic acid.

**European Honey.**—Neufeld † gives the following limits for pure honey:

Water.....	8.30 to 33.59%
Protein.....	0.03 to 2.67%
Invert sugar.....	49.59 to 93.96%
Sucrose.....	0.10 to 10.12%
Dextrin.....	0.99 to 9.70%
Formic acid.....	0.03 to 0.21%
Ash.....	0.02 to 0.68%

**Canadian Honey.**—A large number of samples of genuine honey analyzed in 1897 for the Department of Inland Revenue, Canada (Bul. 47), showed the following variations:

\* Zeits. Unters. Nahr. Genussm., 23, 1912, p. 255.

† Der Nahrungsmittelchemiker als Sachverständiger, Berlin, 1907, p. 275.



Direct polarization.....	— 2.4	to	— 19
Invert “ .....	— 10.2	“	— 28
Sucrose (by Clerget).....	0.5	“	7.64%
Invert sugar.....	60.37	“	78.8%
Water.....	12	“	33%
Ash.....	0.03	“	0.50%

**American Honey.**—Browne\* has examined 97 samples of American and Hawaiian honey, representing the product made from the nectar of numerous flowers as well as honeydew. Maxima and minima of polarizations and analyses of some of the more important kinds, and of all the levorotatory and the dextrorotatory samples are given in the table on page 666.

As regards the chemical characteristics of honey from different flowers, Browne states that alfalfa honey usually has less dextrin and undetermined matter—the so-called ‘impurities’—and more sucrose than the other varieties, although the low amount of impurities is, to some extent, characteristic of the honey of the whole family (leguminosæ). The compositæ yield honey with about the average amount of organic non-sugars; the rosaceæ yield a product low in dextrin, but high in undetermined matter. Buckwheat and other polygonaceous honeys contain almost no sucrose, but give tests for tannins. Basswood honey is relatively high in dextrin, and that from poplar, oak, hickory and other trees, all of which contain considerable quantities of honeydew, are rich in both dextrin and ash. Pronounced tannin reactions are obtained in honey gathered from the flowers or plants of the sumac, hop and others rich in tannin. Tupelo, mangrove and sage honeys are distinguished by their high levulose content.

Browne found the average per cent of water in honey from the arid states of Arizona, Nevada, Utah, and Colorado was 15.60, and from the humid states of Minnesota, Wisconsin, Illinois, Missouri and Iowa was 18.88.

**Hawaiian Honey.**—This is characterized by its high ash and the presence of decided amounts of chlorides in the ash. Van Dine† states that the floral honey of Hawaii is largely from the blossoms of the algarroba (*Prosopis julifera*), while the honeydew honey, which, together with mixtures of honeydew and floral honey forms about two-thirds of the

\* U. S. Dept. of Agric., Bur. of Chem., Bul. 110 (1908).

† Ibid., p. 52.



POLARIZATIONS AND ANALYSES OF AMERICAN AND HAWAIIAN HONEYS.

Source of Honey, according to Flower.	Number of Samples.	Polarization.				Complete Analysis.							Free Acid as Formic. %	Reducing Sugar as Dextrose. %
		Direct.		Invert.		Water. %	Invert Sugar. %	Sucrose. %	Ash. %	Dextrin. %	Undeter- mined. %			
		Immediate at 20° C. ° V.	Constant at 20° ° V.	87° C. ° V.	20° C. ° V.							87° C. ° V.		
Alfalfa ( <i>Medicago sativa</i> ) . . . . .	8	-17.5	-20.5	+15.3	-24.5	+7.7	79.18	10.01	0.16	0.65	3.12	0.17	75.85	
Maximum . . . . .		-5.0	-9.4	+4.6	-21.0	+3.5	72.65	0.28	0.03	0.04	0.45	0.04	69.60	
Minimum . . . . .														
White clover ( <i>Trifolium repens</i> ) . . . . .	15													
Maximum . . . . .		-16.8	-18.2	+16.4	-23.0	+13.8	78.15	7.09	0.20	2.46	7.45	0.10	74.86	
Minimum . . . . .		-3.2	-6.0	+7.4	-12.9	+5.1	70.32	0.00	0.04	0.07	3.11	0.05	67.37	
Buckwheat ( <i>Fagopyrum fagopyrum</i> ) . . . . .	2													
Maximum . . . . .		-14.7	-17.4	+8.4	-21.0	+6.1	77.48	0.06	0.07	1.41	3.30	0.22	74.23	
Minimum . . . . .		-14.5	-16.2	+8.0	-19.8	+5.8	76.21	0.00	0.11	1.04	3.25	0.20	73.01	
Cotton ( <i>Gossypium herbaceum</i> ) . . . . .	3													
Maximum . . . . .		-16.5	-18.2	+15.0	-22.0	+13.8	80.69	1.64	0.28	1.83	4.25	0.20	77.30	
Minimum . . . . .		-2.3	-5.0	+6.6	-7.5	+5.1	74.70	0.48	0.13	0.40	0.48	0.12	71.56	
Basswood ( <i>Tilia sp.</i> ) . . . . .	6													
Maximum . . . . .		-9.3	-13.7	+23.7	-16.8	+23.2	78.55	2.03	0.35	7.58	4.21	0.18	75.24	
Minimum . . . . .		+3.7	-0.3	+9.7	-1.3	+8.6	69.85	0.00	0.11	1.50	2.83	0.05	66.92	
Sumac ( <i>Rhus</i> ) . . . . .	3													
Maximum . . . . .		-11.1	-13.8	+14.6	-17.6	+14.4	73.73	2.01	0.90	6.42	6.68	0.18	70.63	
Minimum . . . . .		-4.6	-8.5	+8.5	-11.4	+7.8	68.61	0.36	0.21	1.66	3.89	0.09	65.73	
Orange ( <i>Citrus aurantium</i> ) . . . . .	1	-11.7	-15.5	+8.8	-19.3	+6.6	77.57	0.60	0.08	0.45	4.31	0.08	74.31	
Tupelo ( <i>Nyssa aquatica</i> ) . . . . .	2													
Maximum . . . . .		-21.9	-24.6	+6.2	-29.3	+4.6	72.40	4.36	0.08	2.69	6.02	0.06	69.36	
Minimum . . . . .		-20.6	-23.4	+5.9	-27.5	+4.3	72.09	1.65	0.07	1.47	4.50	0.05	69.06	
Hawaiian honeydew and flowers . . . . .	5													
Maximum . . . . .		-10.6	-15.1	.....	-18.2	+34.8	76.55	5.27	1.29	10.01	4.18	0.15	73.33	
Minimum . . . . .		+24.9	+17.8	.....	+13.5	+7.8	64.84	0.36	0.48	1.77	2.66	0.07	62.12	
Levorotatory honeys . . . . .	92													
Maximum . . . . .		-21.9	-24.8	+23.7	-29.3	+23.2	83.36	10.01	0.90	7.58	7.45	0.25	79.86	
Minimum . . . . .		+3.7	-0.3	+0.5	-1.3	-0.7	62.23	0.00	0.03	0.04	0.04	0.04	59.61	
Dextrotratory honeys . . . . .	7													
Maximum . . . . .		+24.9	+17.8	+35.8	+15.0	+35.0	71.69	5.28	1.29	12.95	4.95	0.19	68.68	
Minimum . . . . .		+6.7	+3.6	+28.5	-2.5	+20.9	64.84	0.61	0.29	6.02	1.57	0.05	62.12	



product of the Hawaiian Islands, comes largely from the exudations of the sugar-cane leaf-hopper (*Perkinsiella saccharicida*), and the sugar-cane aphid (*Aphis sacchari*): Honeydew honey is dextrorotatory, and for this reason has often been condemned as adulterated. It has a strong molasses-like odor, and often a very dark color. Bakers prefer it to algarroba honey, because of its baking and boiling properties.

The variation in the composition of Hawaiian honey is shown in the table on page 666, compiled from Browne's data.

**Cuban, Mexican, and Haitian Honey.**—The following table contains analyses of 33 Cuban, 23 Mexican, and 16 Haitian honeys by A. H. Bryan.\* One of the samples gave a faint color with Browne's test, but not sufficient to confuse the sample with honey containing an appreciable amount of commercial invert sugar. Fiehe's test gave faint reactions in five samples.

	Cuban.	Mexican.	Haitian.
Direct polarization:			
Immediate at 20° C.....	— 6.1 to —20.0	— 7.2 to —22.9	—11.3 to —19.6
Constant at 20° C.....	— 8.6 to —21.1	— 8.5 to —24.2	—12.5 to —20.7
Constant at 87° C.....	+ 6.0 to +17.0	+ 3.2 to +15.7	+ 4.3 to +10.7
Invert polarization:			
At 20° C.....	— 8.9 to —23.4	— 9.3 to —26.1	—13.3 to —22.7
At 87° C.....	+ 4.5 to +15.4	+ 2.9 to +13.4	+ 3.5 to +10.1
Water.....per cent	16.05 to 27.00	19.43 to 24.40	18.60 to 22.05
Invert sugar.....	68.09 to 77.56	69.27 to 75.04	69.15 to 76.73
Sucrose.....	00.00 to 2.99	0.00 to 3.98	0.00 to 2.44
Ash.....	0.07 to 0.39	0.13 to 0.58	0.06 to 0.45
Dextrin.....	0.29 to 3.96	0.52 to 3.48	0.26 to 1.65
Undetermined.....	1.23 to 8.07	1.35 to 6.30	0.66 to 5.46
Free acid as formic... "	0.00 to 0.43	0.07 to 0.35	0.03 to 0.28

**Dextrorotatory Honey.**—The U. S. standards define honey as lævorotatory, thus excluding the larger part of the Hawaiian product, and also unimportant kinds of honey made from certain trees. Pure floral honey with no admixture of honeydew is seldom if ever dextrorotatory.

The following are the results obtained by Browne in the examination of dextrorotatory honeys:

\* U. S. Dept. of Agric. Bur. of Chem., Bul. 154, 1912.



	Wild Penny-royal.	Poplar.	Hickory.	White Oak	Hawaiian.		
					Sugar Cane Honey-dew.	Honeydew and Flowers.	Honeydew and Flowers.
Direct polarization at 20° C.*...	+17.0	+ 3.6	+ 7.8	+11.0	+17.8	+ 3.6	+ 5.3
Invert polarization at 20° C....	+15.0	- 2.5	+ 3.4	+ 5.2	+13.5	+ 1.9	+ 1.9
Invert polarization at 87° C....	+35.0	+20.9	+26.6	+28.6	+34.8	+23.7	+23.4
Water. ....per cent	16.44	17.02	16.05	13.56	15.46	16.29	17.80
Invert sugar .....	71.69	65.80	65.89	65.87	64.84	67.81	66.85
Sucrose .....	0.61	3.10	2.76	4.31	5.27	2.57	2.41
Ash .....	0.29	0.76	0.78	0.79	1.29	1.02	0.80
Dextrin .....	6.02	10.19	12.95	10.49	10.01	9.65	8.62
Undertermined .....	4.95	3.13	1.57	4.98	3.13	2.66	3.52
Free acid as formic ....	0.05	0.19	0.12	0.08	0.15	0.14	0.13
Reducing sugar as dextrose, per cent.....	68.68	63.04	63.12	63.11	62.12	64.96	64.04

\* Constant.

**Adulteration of Honey.**—The most common adulterant is commercial invert sugar. Cane sugar and glucose were formerly used. Gelatin is also said to be used. It appears to be a fact that bees may be made to feed upon cane syrup or commercial glucose, if these materials are placed in proximity to their hives, so that in some instances the adulterant may be supplied through the medium of the bee. Sophisticated honey is often put up in tumblers or jars containing pieces of honeycomb, so that presence of the comb is by no means proof of its purity. Comb-honey, sold in the frame as sealed by the bees, is never adulterated, except when the bees are fed upon glucose or cane sugar.

**Cane Sugar.**—The following are typical analyses of honey adulterated with cane sugar:

	A.	B.	C.
Direct polarization. . . .	+34.7	+12	+ 1.2
Invert " . . . .	-24	-17.6	-21.5
Temperature. ....	14°	15°	19.5°
Sucrose (Clerget) . . . .	43.16%	21.8%	17.07%
Invert sugar. ....	42.48%	60.03%	67.2%
Water. ....	42.42%	21.15%	15.56%
Ash. ....		.11%	0.06%

A strong right-handed polarization before inversion, coupled with a left-handed invert reading at 20°, is evidence of adulteration with cane sugar, or a product containing cane sugar.



Honey stored by bees fed on cane sugar is also characterized by its right-handed polarization. Although the bee inverts the larger part of the cane sugar in its body, this inversion is never as complete as in the case of nectar honey.

*Glucose.*—The following are typical analyses of honey adulterated with commercial glucose:

	A.*	B.	C.
Direct polarization. . .	+147	+66.9	+101.5
Invert " . . .	+135.2	+61.9	+99.0
Temperature. . . . .	18°	20°	22°
Sucrose (Clerget). . . .	8.83%	3.76%	0.0%
Invert sugar. . . . .	46.18%	74.66%	49.87%
Water. . . . .	15.19%	21.40%	23.7%
Ash. . . . .	0.03%		

Care should be taken not to confuse honeydew honey with honey adulterated with glucose. Browne gives the following means of distinction: (1) the difference in invert polarization between 20 and 87°, corrected to 77% invert sugar, (2) Beckman's iodine test (page 673), and (3) the König and Karsch test (page 674). He also finds the quantity and character of the ash, the acidity, and microscopic examination of value.

The following analyses of mixtures of commercial glucose and honey were made by A. H. Bryan.†

Mixture.		Constant Direct Polarization at 20° C.	Invert Polarization—		Polarization Difference (87°—20°).	Invert Sugar		Calculated Glucose.		
Glucose	Honey.		At 20° C.	At 87° C.		Before Inversion.	After Inversion.	Invert Polarization at 87° ÷ 1.63.	Invert Polarization at (20° C. + 17.5) ÷ 1.93.	100—(Corrected Polarization Difference X 100 ÷ 26.7)
%	%	° V.	° V.	° V.	° V.	%	%	%	%	%
100	.....	+153.8	+153.34	+144.32	.....	30.02	30.45	88.5	88.5	
50	50	+67.0	+65.67	+73.81	8.14	53.67	54.50	45.3	43.1	56.9
20	80	+15.4	+13.42	+33.00	19.58	69.00	70.35	20.2	16.0	19.2
10	90	—2.4	—4.84	+18.59	23.43	74.42	74.12	11.4	6.6	8.8
5	95	—11.5	—14.31	+11.66	25.96	75.74	77.80	7.2	1.6	3.8
3	97	—14.2	—16.94	+9.13	26.07	76.62	78.01	5.6	0.29	3.7
2	98	—16.0	—18.70	+8.14	26.84	76.64	78.34	5.0	0.00	1.2
1	99	—18.2	—20.90	+6.93	27.83	77.20	78.87	4.2	0.00	0.0
.....	100	—19.5	—22.11	+5.94	28.05	77.68	78.9	3.2	0.00	0.0

\* Both commercial glucose and added cane sugar.

† A. O. A. C. Proc., 1908, U. S. Dept. of Agric., Bur. of Chem., Bul. 122, p. 181.



*Commercial Invert Sugar* is the most difficult of detection of all the adulterants. Herzfeld's process\* for the manufacture of invert sugar syrups consists in boiling for thirty to forty-five minutes 1 kilogram of refined sugar in 300 cc. of water with 1.1 gram of tartaric acid. Browne† gives the following analysis of the product made by this process:

Direct polarization at 20°.....	- 6.2
Constant polarization at 20°.....	- 9.5
Invert polarization at 20°.....	-16.9
Invert polarization at 87°.....	+ 4.8
Water.....	16.32%
Invert sugar.....	73.38%
Sucrose.....	4.36%
Ash.....	0.00%
Dextrin.....	4.86%
	100.00%
Acids as formic.....	0.06%

This adulterant is best detected by Browne's and Fiehe's tests (page 674). Ley's test‡ has value as a confirmatory test, but should be used with caution, as American honeys do not react like the European.

*Gelatin* is indicated if a precipitate occurs in the diluted sample with a solution of tannic acid.

#### ANALYSIS OF HONEY.

**Preparation of Sample.**—In the case of strained honey, stir with a rod, till any separated sugars are evenly distributed throughout the mass, or, if the honey has become solidified wholly or in part by crystallization, use a gentle heat on a closed water-bath to restore to it fluid form.

In the case of comb honey, cut with a knife across the top of the comb if sealed, and separate completely from the comb by straining through a 40-mesh sieve.

**Determination of Moisture.**§—Weigh 2 grams into a flat-bottom metal dish 2½ inches in diameter, which, together with 10 to 15 grams

\* Zeits. ver. d. Zucker-Ind., 31, p. 1988.

† Loc. cit., p. 64.

‡ Pharm. Zeits., 47, 1902, p. 603.

§ Browne, U. S. Dept. of Agric., Bur. of Chem., Bul. 110, p. 18.



of fine quartz sand and a short stirring rod, has been previously tared, add 5 to 10 cc. of water, stir until the whole has been thoroughly incorporated, and dry to constant weight at 65 to 70° C. in a vacuum oven. Honeys of high purity usually dry in twelve hours, while those of the honeydew class, rich in dextrin and gum, require thirty-six hours, or longer.

**Determination of Ash.**—See page 644.

**Polarization.**—*Direct and Invert at 20° C.*—Proceed as directed under molasses (page 644), except that only alumina cream is used as a clarifier. To destroy birotation add a drop or two of ammonia before making up to the mark.\*

*Invert at 87° C.*—Invert a half normal portion in the usual manner in a 100-cc. flask, cool, add a few drops of phenolphthalein and enough sodium hydroxide to neutralize; discharge the pink color with a few drops of dilute hydrochloric acid, add from 5 to 10 cc. of alumina cream, make up to the mark and filter. Polarize in a 200-mm. tube at 87°, and multiply reading by 2.

Polarization at the temperature of 87° can most readily be effected by the use of a water-jacketed tube, as shown in Fig. III. An all-metal tube, the interior of which is heavily gold-plated to avoid corrosion by acid, is preferable to one in which the inner tube is glass with a metal jacket, as in the latter leaky joints are liable to occur, due to uneven expansion. A tubulure is provided in the outer tube for a thermometer, so that the exact temperature may be noted. A tank of boiling water placed on a shelf above the polariscope is connected by rubber tubing with the jacketed tube as it rests in the polariscope, as shown in Fig. III.

**Determination of Reducing Sugars.**—Determine by Allihn's method (page 632) in an aliquot of 25 cc. of a solution obtained by making 10 cc. of the solution prepared for polarization up to 250 cc. If desired the sugar may be determined by the volumetric Fehling process (page 615).

The reducing sugars may be calculated as dextrose as obtained from Allihn's table, or as levulose by multiplying the dextrose by 1.044.

**Determination of Levulose.**—*Wiley's Method.*†—This may be calculated approximately by the following formula:

$$l = \frac{100(1.0315A - a)}{(2.3919)26} = \frac{100(1.0315A - a)}{62.19},$$

\* Frühling, Zeits. öffentl. Chemie, 4, 1898, p. 410.

† Principles and Practice of Agricultural Analysis, 1897, III, p. 267. Browne, loc. cit., p. 17.



in which  $l$  = levulose,  $a$  = the direct polarization at  $20^{\circ}$  of a solution of the normal quantity of honey made up to 100 cc. at  $20^{\circ}$ , and  $A$  = the direct polarization of the same solution at  $87^{\circ}$  C., 2.3919 = the variation in polarization of 1 gram of levulose in 100 cc. of solution between  $20$  and  $87^{\circ}$  C., and 1.0315 = the factor for converting the volume of the solution at  $20^{\circ}$  into that at  $87^{\circ}$  C.

**Determination of Dextrose.\***—Multiply the percentage of levulose as obtained in the preceding section by 0.915, thus obtaining the equivalent dextrose, and subtract this from the per cent of reducing sugars expressed as dextrose.

**Determination of Sucrose.**—Owing to the inaccuracies of Clerget's method as applied to honey, Browne recommends the following: Neutralize the free acid of 10 cc. of the solution used for invert polarization with sodium carbonate, make up to 250 cc. and determine the reducing sugars by Allihn's method. Subtract from the invert sugar thus obtained the invert sugar found before inversion, and multiply the difference by 0.95.

**Determination of Dextrin.—Browne's Method.\***—Weigh 8 grams of honey directly into a 100-cc. flask, add 4 cc. of water, and finally with continued agitation sufficient absolute alcohol to fill to the mark. Shake thoroughly and allow to stand twenty-four hours, or until the dextrin is deposited on the bottom and sides of the flask and the liquid is perfectly clear. Decant on a filter and wash the precipitate in the flask with 10 cc. of cold 95% alcohol, pouring the liquid finally on the filter. Dissolve the precipitate in the flask and on the filter in a little boiling, distilled water, collecting the solution in a tared platinum dish. Evaporate the liquid, and dry to constant weight at  $100^{\circ}$  C. If the alcohol precipitate is considerable, it should be dried at  $70^{\circ}$  C. *in vacuo*. After weighing, dissolve in water and make up to a definite volume according to the weight as follows:

Residue, grams.	0-0.5	0.5-1.0	1.0-1.5	1.5-2.0	2.0-2.5	2.5-3.0
Volume, cc. . . .	50	100	150	200	250	300

Filter, determine invert sugar and sucrose in aliquots by copper reduction before and after inversion, and subtract the sum of these sugars from the total alcohol precipitate.

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\* Browne, loc. cit., p. 17. Jour. Am. Chem. Soc., 28, 1906, p. 446.



**Determination of Acids.**—Dissolve 10 grams of the honey in water and titrate with tenth-normal sodium hydroxide, using phenolphthalein as indicator. Express result as formic acid.

**Beckman's Test for Glucose.\***—Treat a mixture of equal parts of honey and water with a solution of iodine in potassium iodide. If glucose is present, a red or violet color (due to erythro- or amylo-dextrin) appears, the shade and intensity depending on the nature and amount of the glucose present.

**Determination of Commercial Glucose in Honey.**—Except for rough work, the method described on page 651 for calculating the per cent of commercial glucose from the sucrose and from the direct polarization is not recommended for use with honey and other products wherein the invert sugar is so large as to considerably affect its accuracy. In this case, it is best after inversion to polarize the sample at  $87^{\circ}$  C., the temperature at which the reading due to invert sugar would theoretically be 0. At this temperature, any considerable right-handed polarization can be accounted as due to commercial glucose. (See page 671.)

As in the case of molasses, the writer advocates assuming  $175^{\circ}$  as the direct polarization of the glucose used, this being about the maximum reading for a normal solution of  $42^{\circ}$ -Bé. glucose. Lythgoe has shown that in polarizing at high temperatures samples of saccharine products containing commercial glucose, certain precautions have to be observed not necessary when cane or invert sugar are the only sugars present. Thus, a normal solution of glucose, when polarized at  $87^{\circ}$  C., has a lower reading than in the cold, the difference being doubtless due partly at least to the expansion of the liquid. Again, on subjecting a normal solution of glucose to inversion with acid, as in Clerget's process, and heating to  $87^{\circ}$  C., it will be found impossible to get a constant reading, but the reading will drop rapidly, due to a partial hydrolysis of the maltose or dextrin.

In honey and other preparations containing much invert sugar and commercial glucose, it is best to proceed as follows: Divide the polarization at  $87^{\circ}$  by  $163^{\circ}\dagger$  and multiply the result by 100 for the percentage of commercial glucose in terms of glucose polarizing at  $175^{\circ}$ . It should be

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\* Zeits. Anal. Chem., 35, 1896, p. 267.

$\dagger$  The true polarization at  $87^{\circ}$  C. of a normal solution of glucose subjected to inversion and neutralization as above (but without the use of the clarifier), will be about 93% that of the direct polarization of the sample in the cold. Hence  $175 \times 0.93 = 162.7$ .



borne in mind that the results by even this method are only approximate, as genuine honey is more or less dextrorotatory at 87° C.

The following formula is used by European chemists:  $G = \frac{b - 17.5}{1.93}$ , in which  $G$  = the per cent of commercial glucose, and  $b$  = the polarization after inversion at 20° C.

**Browne's Test for Commercial Invert Sugar.\*—Reagent.**—This should be freshly prepared each time before using. Shake 5 cc. of c. p. anilin with 5 cc. of water, and add sufficient glacial acetic acid (2 cc.) to just clear the emulsion.

**Process.**—Treat 5 cc. of a 1 : 1 solution of the honey in a test-tube with 1 to 2 cc. of the anilin reagent, allowing the latter to flow down the walls of the tube so as to form a layer upon the honey solution. If, when the tube is gently agitated, a red ring forms beneath the anilin solution, this color becoming gradually imparted to the whole layer, artificial invert sugar is present. This reaction is due to furfural formed during the high temperature employed in the commercial processes of inversion. Boiling genuine honey also causes the formation of furfural, but this treatment impairs the flavor and is probably never practiced.

**Fiehe's Test for Commercial Invert Sugar Modified by Bryan.†—**Place 10 cc. of a 50% solution of the sample in a test tube, add 5 cc. of ether, shake vigorously, allow to stand until the ether is clear, then transfer 2 cc. to a small test-tube and add a large drop of a solution of 1 gram of resorcin in 100 cc. of hydrochloric acid and shake. A cherry-red color indicates commercial invert sugar while a faint orange to rose color, disappearing after a short time, may be due to heating of the honey.

**Ley's Ammoniacal Silver Nitrate Test‡** is not so reliable as the two preceding tests.

**Distinction of Honeydew and Glucose Honeys.—Method of König and Karsch.§**—Dissolve 40 grams of honey in a cylinder in water, and make up to 40 cc. Transfer 20 cc. of the homogeneous solution to a 250-cc. flask and fill to mark with absolute alcohol with slow addition and constant shaking, and then allow to stand two or three days, with occasional agitation. At the end of this time all the dextrin has settled out. After shaking the solution, filter and evaporate 100 cc. of the filtrate until free

\* U. S. Dept. of Agric., Bur. of Chem., Bul. 110, p. 68.

† Zeits. angew. Chem., 21, 1898, p. 2315; Bur. of Chem., Bul. 154, p. 15.

‡ Pharm. Ztg., 1902, p. 603; Zeits. angew. Chem., 1907, p. 993.

§ Zeits. anal. Chem., 34, 1895, p. 1. U. S. Dept. of Agric., Bur. of Chem., Bul. 110, p. 63.



from alcohol. To the liquid residue add a little subacetate of lead and sodium sulphate, make up to 20 cc. with water, and polarize the filtered solution. Dextrorotatory natural honeys show by this method a lævoration; honeys adulterated with dextrose of glucose to the extent of 25% or more, a dextrorotation. In case the honey contains a large amount of sucrose, the solution should be inverted with hydrochloric acid before polarizing.

**BEESWAX.**—The purity of beeswax is best established by determining its melting-point, its specific gravity, its saponification equivalent, and its refractometric reading. The melting-point of pure wax is about 64° C., while that of paraffin, its chief adulterant, is from 52 to 55° C. Its saponification equivalent should be from 87.8 to 107, while that of paraffin is 0.

*Method of Determining Specific Gravity of Beeswax.\**—Place a weighed rod of the wax, about 1 to 1.5 cm. long by 0.5 cm. diameter, in an accurately marked 50 cc. flask, and run in water from a burette till the water level reaches the mark. 50 cc. minus the burette reading represent the volume occupied by the wax. The rod should be made to lie flat on the bottom of the flask, so that the incoming water will force its end against the sides and prevent the end from rising above the mark. The weight of the rod, divided by its volume gives its specific gravity. The specific gravity of various mixtures of wax of 0.969 specific gravity and paraffin of 0.871 are given in the following table, prepared by Wagner, so that from the specific gravity of the mixture the percentage of paraffin can be calculated:

Wax (Percentage).	Paraffin (Percentage).	Specific Gravity.	Wax (Percentage).	Paraffin (Percentage).	Specific Gravity.
—	100	.871	75	25	.942
25	75	.893	80	20	.948
50	50	.920	100	—	.969

**The Refractometer Reading** is most useful in establishing the purity of wax. Observations with this instrument are best made at 65° and great care should be taken in the case of the Zeiss butyro-refractometer not to exceed this temperature, or injury to the instrument may result.

The Abbé refractometer may be used with perfect safety and, when available, is to be preferred for the examination of beeswax. Many

\* Gawalowski, Chem. Centrbl., 1890, p. 502.



food laboratories are, however, not equipped with the Abbé, but nearly all find the butyro-refractometer indispensable. The latter instrument was primarily designed for such substances as butter and lard, so that the manufacturers did not intend it to be subjected to as high a temperature as  $65^{\circ}$ . They have, however, assured the author that if care be taken

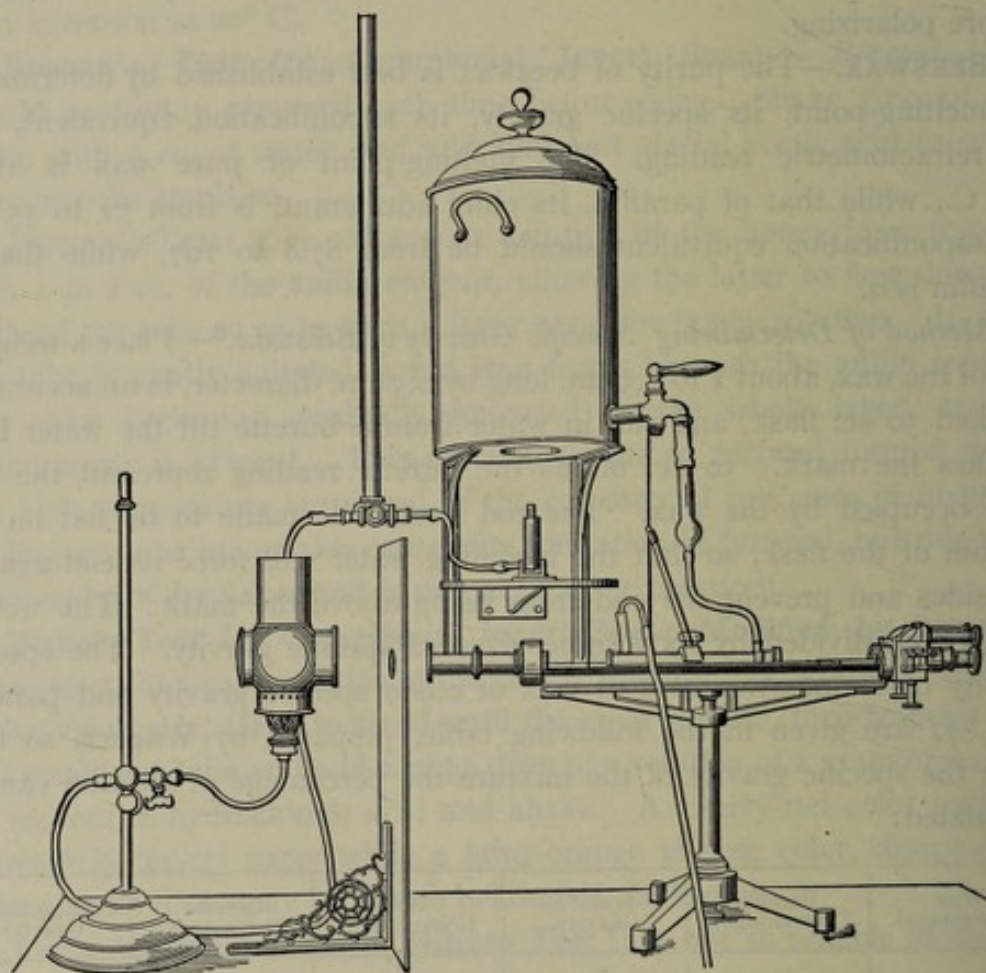


FIG. 111.—Apparatus for Polarizing at High Temperatures.

to bring the temperature very slowly and gradually to the required degree  $65^{\circ}$ , and to avoid also sudden cooling, the cement that secures the prisms in place will not be appreciably affected; otherwise cracking or loosening of the cement would be liable to occur after a time.

At  $65^{\circ}$  C. pure beeswax should have a reading on the butyro-refractometer of 30 to 31.5,\* while that of paraffin is from 11 to 14.5.†

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\*  $n_D$ , 1.4452 to 1.4463.

†  $n_D$ , 1.4310 to 1.4335.



### CONFECTIONERY.

The composition of confectionery is more complex than that of the saccharine products hitherto considered. As a rule, cane sugar, or one of its products, as molasses, forms the basis of most of the confections. Commercial glucose is also a common ingredient, while a large variety of such materials as eggs, butter, chocolate, various flavoring extracts, spices, nuts, and fruits, enter into the composition of confectionery.

**U. S. Standard Candy** is candy containing no terra alba, barytes, talc, chrome, yellow, or other mineral substances or poisonous colors or flavors, or other ingredients injurious to health.

**Adulteration.**—Of late the adulteration of confectionery has been held largely in check by the National Confectioners' Association of the United States, which has fixed high standards of purity, and has been very zealous in restricting the use of harmful adulterants.

Commercial glucose is not regarded as an adulterant of confectionery by the above-named association and by but few food authorities. On the contrary, any ingredient, other than color, that has no food value, may logically be considered as an adulterant. Under this head are included such substances as paraffin, as well as make-weight mineral matters, such as terra alba, talc, or calcium sulphate.

B. H. Smith \* has called attention to the presence of arsenic in shellac used to coat certain kinds of confectionery.

**Colors in Confectionery.**—A very wide range of colors is necessarily employed in the manufacture of confectionery, and the almost endless variety of coal-tar dyes now available lend themselves most readily to the confectioner's needs. Elsewhere, under "colors," lists of injurious and non-injurious dyes are given as compiled by the National Confectioners' Association, though it is not always readily apparent how the lines are drawn.

The tinctorial power of these dyes is so high that the actual amount of substance contained in a thin coating of the color on the outside of the candy is exceedingly small, so that it is doubtful whether serious cases of injury have ever arisen from their use.

This was not the case formerly when such poisonous mineral pigments as chromate of lead were frequently used.

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\* U. S. Dept. of Agric., Bur. of Chem., Circ. 91, 1912.



## ANALYSIS OF CONFECTIONERY.

The following methods are largely those submitted by the author as provisional methods of the A. O. A. C.:\*

(1) **Products of Practically Uniform Composition Throughout.**—

(a) *Lozenges and Other Pulverizable Products.*—Grind in a mortar or mill to a fine powder. For *total solids*, weigh from 2 to 5 grams of the powdered sample in a tared platinum dish, and dry in a McGill oven to constant weight.

For *Ash*, ignite the residue from total solids in the original dish, observing the precautions given under sugar (page 609), and molasses (page 644).

(b) *Semi-plastic, Syrupy, or Pasty Products.*—Weigh 50 grams of the sample into a 50-cc. graduated flask, mix thoroughly or dissolve, if soluble in water, and fill to the mark. Be sure that the solution is uniform, or, if insoluble material is present, that it is evenly mixed by shaking before taking aliquot parts for the various determinations. For *total solids* and *ash*, measure 25 cc. of the above solution or mixture into a tared platinum dish, and proceed as directed under (a).

(2) **Confectionery in Layers or Sections of Different Composition.**—

When it is desired to examine the different portions separately, they should be separated mechanically with a knife, when possible, and treated as directed under (1).

(3) **Sugar-coated Fruit, Nuts, etc.**—In case of a saccharine coating inclosing fruit, nuts, or any less readily soluble material, dissolve or wash off the exterior coating in water, which may, if desired, be evaporated to dryness for weighing, and proceed as in (1).

(4) **Candied or Sugared Fruits.**—Proceed as in the examination of fruits (Chapter XXI).

**Detection of Mineral Adulterant.**—As in the case of molasses, a considerable quantity, say 100 grams, should be reduced to an ash for examination for mineral adulterants, such as talc, calcium sulphate, and iron oxide, which are detected by regular qualitative tests.

**Detection of Lead Chromate.**—Fuse the ash in a porcelain crucible with a mixture of sodium carbonate and potassium chlorate, boil the fused residue with water, neutralize with acetic acid, filter, and treat the filtrate with barium chloride or lead acetate solution. A yellow pre-

\* U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 44.



precipitate indicates a chromate. Treat the insoluble part of the fusion with nitric acid, and test for lead in the usual manner.

If a drop of ammonium sulphide be applied to a piece of confectionery colored with lead chromate, it will produce a black coloration.

**Determination of Ether Extract.**—The ether extract includes the fat derived from chocolate, eggs, or butter, as well as any paraffin present. Measure 25 cc. of the 20% solution (1) (b) (page 678) into a very thin, readily frangible glass evaporating-shell (*Hoffmeister's Schälchen*), containing 5 to 7 grams of freshly ignited asbestos fiber; or, if impossible to thus obtain a uniform sample, weigh out 5 grams of the mixed, finely divided sample into a dish, and wash with water into the asbestos in the evaporating-shell, using, if necessary, a small portion of the asbestos fiber on a stirring-rod to transfer the last traces of the sample from dish to shell. Dry to constant weight at 100°, after which cool, wrap loosely in smooth paper, and crush into rather small fragments between the fingers, carefully transferring the pieces with the aid of a camel's-hair brush to an extraction-tube, or to a Schleicher and Schull cartridge for fat extraction. Extract with anhydrous ether or with petroleum ether in a continuous extraction apparatus for at least twenty-five hours. Transfer the solution to a tared flask, evaporate the ether, dry in an oven at 100° C. to constant weight, and weigh.

More recently the association adopted the Röse-Gottlieb method for butter scotch.

**Determination of Paraffin.**—Add to the ether extract in the flask, as above obtained, 10 cc. of 95% alcohol, and 2 cc. of 1 : 1 sodium hydroxide solution, connect the flask with a reflux condenser, and heat for an hour on the water-bath or until saponification is complete. Remove the condenser, and allow the flask to remain on the bath till the alcohol is evaporated off, and a dry residue is left. Treat the residue with about 40 cc. of water, and heat on the bath, with frequent shaking, till everything soluble is in solution. Wash into a separatory funnel, cool, and extract with four successive portions of petroleum ether, which are collected in a tared flask or capsule. Remove the petroleum ether by evaporation, and dry in the oven to constant weight.

It should be noted that any phytosterol or cholesterol present in the fat would come down with the paraffin, but the amount would be so insignificant that, except in the most exacting work, it may be disregarded. The character of the final residue should, however, be confirmed by determining its melting-point and specific gravity, and by subjecting it



to examination in the butyro-refractometer. The melting-point of paraffin is about  $54.5^{\circ}\text{C}.$ ; its specific gravity at  $15.5^{\circ}\text{C}.$  is from 0.868 to 0.915, and on the butyro-refractometer the reading at  $65^{\circ}\text{C}.$  is from 11 to 14.5.

**Determination of Starch.**—Measure gradually 25 cc. of a 20% aqueous solution or uniform mixture of the sample into a hardened filter or Gooch crucible, or transfer by washing 5 grams of the finely powdered substance to the filter or Gooch, and allow the residue on the filter to become air-dried. Extract with five successive portions of 10 cc. of ether, then wash with 150 cc. of 10% alcohol, and finally with 20 cc. of strong alcohol. Transfer the residue to a large flask and boil gently for four hours with 200 cc. of water and 20 cc. of hydrochloric acid (specific gravity 1.125), the flask being provided with a reflux condenser. Cool, neutralize with sodium hydroxide, add 5 cc. of alumina cream, and make up the volume to 250 cc. with water. Filter and determine the dextrose in an aliquot part of the filtrate by any of the various Fehling methods. The weight of the dextrose multiplied by 0.9 gives the weight of the starch.

**Polarization of Confectionery.**—As a clarifier use either alumina cream or subacetate of lead, according to the nature and capacity of the sample. Ordinarily alumina cream is best, but in dark-colored samples, or those in which molasses has been used, it is sometimes necessary to employ the subacetate. When starch is absent, and the sample is practically soluble, polarize and invert in the usual manner (page 610). Where considerable starch or insoluble matter is present, use the double-dilution method of Wiley and Ewell (page 650), thus making due allowance for the volume of the precipitate.

*Cane sugar, invert sugar, and dextrin,* are determined as directed for honey.

*Commercial glucose* is roughly determined by polarizing the sample at  $87^{\circ}\text{C}.$ , as in the case of honey (page 671).

Confectionery is made in such a wide variety of forms, and these differ in consistency to such an extent that commercial glucose of all available degrees of density can be utilized to advantage in one product or another. In this respect confectionery is unlike honey and molasses, wherein a fairly uniform grade of commercial glucose is necessarily used for mixing, this grade being naturally selected with reference to its similarity in density to the molasses. On this account the glucose factor used for honey and molasses (175) may in some varieties of confectionery be too high.



**Determination of Alcohol in Syrups Used in Confectionery.**—(*Brandy-drops.*)—Open each drop by cutting off a section with a sharp knife, and collect in a beaker the syrup of from 15 to 25 of the drops, which will usually yield from 30 to 50 grams of syrup. Strain the syrup into a tared beaker through a perforated porcelain filter-plate in a funnel to separate from particles of the inclosing shell, and ascertain the weight of the syrup. Wash into a distilling-flask, dilute with half its volume of water, and distil off into a tared receiving-flask a volume equal to the original volume of syrup taken. Ascertain the weight of the distillate and its specific gravity by means of a pycnometer. Multiply the percentage by weight of alcohol corresponding to the specific gravity, as found in the tables on page 690 *et seq.*, by the weight of the distillate, and divide this by the weight of syrup taken. The result is the per cent by weight of alcohol in the syrup.

**Detection of Colors.**—It is sometimes necessary to macerate a considerable mass of the material to remove the color, which is, however, in the majority of cases readily soluble. The insoluble colors are nearly all mineral pigments to be looked for in the ash, as in the case of chromate of lead (page 678). Frequently the coloring matter is confined to a thin outer layer, which is readily washed off.

The solution of the dyestuff is examined as directed under colors.

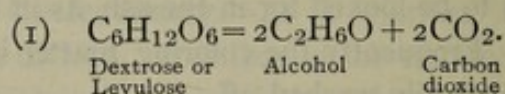
**Detection of Arsenic.**—Arsenic may be present through impure glucose, shellac, or coloring-matter. If the color is confined to an exterior coating, this should be washed off and examined. If distributed through the mass, a solution of the whole should be taken. Examine for arsenic by the Gutzeit or Marsh method, as directed on pages 63 to 66.



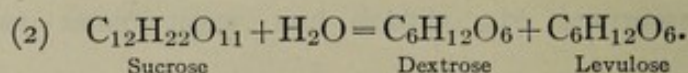
## CHAPTER XV.

### ALCOHOLIC BEVERAGES.

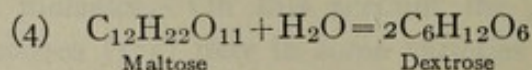
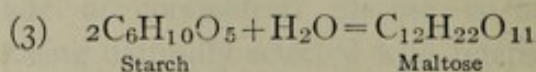
**Alcoholic Fermentation.**—In a broad sense all alcoholic liquors are saccharine products, in that they are essentially the result of the fermentation of sugars. In the case of fruits, the sugars already exist as such in their juices, which, when expressed, almost immediately begin to undergo spontaneously the process of alcoholic fermentation, through the agency of the enzyme zymase of the wild yeasts introduced with the skins of the fruit or from the air. The reaction is as follows:



While the foregoing reaction applies to the dextrose and levulose of invert sugar, which sugar usually predominates in fruit juices, being formed by the inversion of sucrose, the reaction with sucrose itself, which is not directly fermentable, involves a preliminary inversion through the agency of the enzyme invertase present in yeast, thus:



In the case of grains the process is more complex, involving hydrolization of the starch into maltose through the action of the diastase of malt and the further hydrolization of the maltose, which, like sucrose, is not directly fermentable, into dextrose by means of an enzyme of yeast known as maltase or maltoglucose. These reactions may be expressed as follows:





The above reaction, No. 1, illustrating the splitting up of grape sugar into alcohol and carbon dioxide, does not represent the practical yield of alcohol under ordinary conditions that occur in vinous fermentation, for, as a matter of fact, instead of 51.11 parts of alcohol and 48.89 parts carbon dioxide, which would theoretically result as above from the fermentation of 100 parts of dextrose, only about 95% of the theoretical yield can be obtained, so that in practice it is possible to form but about 48.5% alcohol and 46.5% carbon dioxide. The balance, amounting to some 5%, consists chiefly of glycerin, succinic acid, and traces of various compounds, including some of the higher-boiling alcohols (propyl, butyl, and amyl) and their ethers, which form the fusel oil of the distilled liquors.

Vinous fermentation takes place most readily in slightly acid liquids, at a temperature ranging from 25° to 30° C.

It is convenient to divide alcoholic beverages into two main groups, first the fermented and second the distilled liquors. The fermented liquors naturally subdivide themselves into (a) the products of the direct spontaneous fermentation of saccharine fruit juices, such, for example, as those of the apple and the grape, to form cider and wine respectively, and (b) the malted and brewed liquors, such as beer and ale, produced by the conversion of the starch of grain into sugar, and the final alcoholic fermentation of the latter.

The distilled liquors include such products as whiskey, brandy, rum, and gin, wherein alcoholic infusions prepared by previous fermentation in various ways are further subjected to distillation.

**Alcoholic Liquors and State (or Municipal) Control.**—The mere adulteration of liquors does not constitute the only feature which brings them within the scope of the public analyst's work and renders them especially amenable to stringent laws. Indeed, it is often a far more important question for the analyst to decide by his results whether or not the samples submitted to him, by police seizure or otherwise, are sold in violation of the regulations in force in his particular locality governing the liquor traffic.

A common regulation in no-license localities fixes the maximum per cent of alcohol which shall decide whether or not a liquor is legally a temperance drink, and can be sold as such with impunity. From its low content in alcohol, an analyst's findings regarding a certain sample may exonerate the dealer suspected of violating this law, while yet by the very reason of its being low in alcohol the same sample would be placed



in the adulterated list as regards non-conformance to a standard of purity. While the raising of revenue is one purpose for the existence of these laws bearing on liquor license, an equally important object sought to be gained is doubtless the repression of intemperance.

**Toxic Effects.**—A popular impression seems to exist that the toxic effects of an adulterated liquor are far worse from a temperance standpoint than those of a sample of good standard quality, and it is a common experience of the public analyst to have submitted to him by well-meaning temperance advocates samples which are alleged to have caused the worst forms of intoxication, and are thus suspected of being impure. As a matter of fact the chief adulterants of liquors are water, sugar, and, in the case of beer, various bitter principles and vegetable extractives, none of which are on record as being in themselves actively toxic.\*

Alcohol is the one ingredient of liquor which, more than any other, produces a marked physiological effect. Many liquors, especially those of the distilled variety classed as adulterated, are so considered by reason of their low alcoholic content through watering or otherwise, hence this commonest form of adulteration, far from being detrimental in itself, is actually helpful to the temperance cause.

**Details of Liquor Inspection.**—The same precautions should be carefully observed by officers making seizures of liquors for analysis, as by food inspectors, regarding safe delivery of the samples to the analyst. The following instructions are circulated by the State Board of Health of Massachusetts, which has in charge the inspection of liquors, concerning the taking of samples in that state and the transmission to the analyst:

#### DIRECTIONS FOR TAKING SAMPLES FOR ANALYSES.

The officer making a seizure, or taking samples of beer, should note at the time of such seizure the general appearance of the liquor,—as to whether it is clear or cloudy, whether it is still or has a strong head.

If the liquor is in bottles, take at least one pint bottle; if in barrels, draw a pint bottle from each. Request the owner to seal each sample taken. If the bottles have cork stoppers, cut the stoppers off level with the top of the bottle and cover with wax; if with patent stoppers, a little wax placed upon the wire at the point where it lays against the neck of the bottle is sufficient. If the owner refuses to seal it, then the officer

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\* The writer refers to substances intentionally added, and not to accidental impurities, such as arsenic, etc., that are occasionally found.



should seal it in his presence, calling his attention to the fact. Before leaving the premises, place upon the bottle a label or tag, with the date, the name of the owner, and the name of the officer upon it, and also the name of the town or city. Then place in a box, with the certificate required by law, and forward without delay to the analyst.

## FORM OF LABEL.

Town .....  
 Date of seizure ..... 19  
 Owner .....  
 Kind of liquor .....  
 Brewer .....

Accompanying each sample is a certificate like the following, the first part of which is filled out and signed by the officer, while the second part, containing the data of analysis, is filled out and signed by the analyst and returned by him to the officer. Such a certificate is nearly always accepted as evidence in court without the personal appearance of the analyst.

.....ss ..... 19 .

To the State Board of Health:

I send herewith a sample of .....  
 taken from liquors seized by me ..... 19 .

Ascertain the percentage of alcohol it contains, by volume, at sixty degrees Fahrenheit, and return to me a certificate herewith upon the annexed form.

Seized from.....

.....  
 .....

Officer.

## COMMONWEALTH OF MASSACHUSETTS.

No.....

OFFICE OF THE STATE BOARD OF HEALTH.

BOSTON, ..... 19 .

This is to certify that the ..... received by me  
 with the above statement contains ..... per cent of alcohol,  
 by volume, at sixty degrees Fahrenheit. <sup>100</sup>

Received. .... 19 .

Analysis made. .... 19 .

.....

[SEAL.]

Analyst State Board of Health.



A convenient method for recording analyses is by the employment of numbered library cards, which bear the same number as the certificates and are kept by the analyst.

The following is a convenient form:

No. ....	Analyzed.....
County .....	Wt. flask and alc .....
City or town, .....	Wt. flask, .....
Officer, .....	Wt. alc.....
Defendant, .....	Sp. gr. alc. (60°) .....
Address, .....	Per cent alcohol.....
Kind of liquor, .....	Reported.....
Seized, .....	
Received.....	
How delivered .....	
Sealed, .....	
Condition.....	
Kind of bottle .....	
Registered.....	

#### METHODS OF ANALYSIS COMMON TO ALL LIQUORS.

**Specific Gravity.**—This should be taken at 15.6° or calculated to that temperature. The most convenient mode of procedure is to bring the temperature of the sample somewhat below that point by allowing the flask containing it to stand in cold water, and to have everything in readiness to make the determination when 15.6° temperature has been reached, either by the hydrometer spindle in a glass cylinder, by the Westphal balance, or by the pycnometer. The latter is by far the most accurate, especially if it is of the form which is fitted with a thermometer-stopper.

**Detection of Alcohol.**—It is rarely necessary to make a qualitative test for alcohol in liquors, since it is almost invariably present even in many of the so-called temperance drinks, at least in small amount. Indeed in many localities a beverage is legally a temperance drink that contains not more than 1% alcohol by volume.

**The Iodoform Test.**—Alcohol, when present in aqueous solution to the extent of 0.1% or more, may be detected by the iodoform test. The solution is warmed in a test-tube with a few drops of a strong solution of iodine in potassium iodide, after which enough sodium hydroxide solution is added to nearly decolorize. On standing for some time a yellow precipitate of iodoform will appear if alcohol be present, or at once if there is a considerable amount, and the characteristic odor of iodoform will be rendered apparent, even when the precipitate is so slight as to be almost imperceptible. This iodoform precipitate is crystalline, showing under the microscope as star-shaped groups or hexagonal tablets.



It should not be forgotten that other substances than alcohol give the reaction, as lactic acid, acetone, and various aldehydes and ketones.

Pure methyl or amyl alcohol or acetic acid do not thus react.

*Berthelot* recommends benzoyl chloride as a reagent for detecting alcohol. By warming a mixture of a few drops of benzoyl chloride with the solution to be tested, and adding a little sodium hydroxide, ethyl benzoate is formed, recognizable by its distinctive odor. This reaction is delicate to 0.1% alcohol. The presence of other alcohols than ethyl produces ethers of characteristic odor.

*Hardy's Test for Alcohol* consists in shaking the aqueous solution with some powdered guaiacum resin, filtering, and adding to the filtrate a little hydrocyanic acid and a drop of dilute copper sulphate solution. A blue coloration considerably deeper than that due to the copper salt is indicative of alcohol.

*Methyl Alcohol* in spirits is tested for as described on pp. 781-784.

**Determination of Alcohol.**—In the case of carbonated liquids it is necessary to first expel the free carbon dioxide, which is readily accomplished by pouring the liquor back and forth from one beaker to another, from time to time removing the excess of froth from the top of the vessel by the aid of the hand. Or, the sample may be shaken vigorously in a large separatory funnel, and the still liquor drawn off from below the froth, repeating the operation several times if necessary. In either case the mechanical treatment should be continued till the liquor is comparatively quiet and free from foam.

(1) *By Distillation.*—This is by far the most accurate method of determining alcohol, and should be carried out in all cases where any legal controversy is apt to be involved. Into a flask of 250 to 400 cc. capacity introduce a convenient quantity of the liquor, which should be accurately weighed or measured, according to whether the percentage by weight or measure is desired. The following are suitable quantities: Distilled liquors, 25 grams or cc.; cordials, 25 to 50 grams or cc.; wines, ciders, and malt liquors, 100 grams or cc. In the case of wines or ciders which have undergone acetic fermentation, add 0.1 to 0.2 gram of precipitated calcium carbonate or neutralize with standard alkali.

Dilute the liquid to 150 cc. and distil into a 100-cc. flask. Nearly all alcoholic liquors, if comparatively free from carbon dioxide, will boil without undue frothing or foaming. New wine will occasionally give trouble in this regard, but foaming may usually be prevented in this



case by the addition of tannic acid. In case of wine, cider, and beer all the alcohol will have passed over in the first 75 cc. of the distillate, or three-fourths the original measured volume, but with distilled liquors high in alcohol the process had better be continued till nearly 100 cc. or the original volume taken have passed over. If the condenser is of glass, one can observe when all the alcohol has been distilled over, for the reason that the mixed alcohol and water vapors in the upper portion of the condenser present a striated or wavy appearance, readily apparent so long as the alcohol is passing over, while after all the alcohol has been distilled, the condenser-tube appears perfectly clear. The distillation is thus continued for some time after this striated appearance has ceased. The distillate in the receiving glass is finally made up to the mark or to the original volume of the liquor taken. Strictly speaking, the measurements before and after distillation should be made at  $15.6^{\circ}\text{C.}$ , but, excepting in case of distilled liquors, no appreciable error results from making both measurements at the same or room temperature. Another precaution formerly thought necessary was to have the delivery-tube from the condenser pass below the level of a little water in the receiving-flask from the start, but equally accurate results have been obtained by simply allowing the end of the condenser-tube to enter the narrow-necked flask.

Fig. 112 shows a bank of six stills of the kind used in the author's laboratory for alcohol determination in liquors. In each still the vertical glass worm-condenser, the round-bottomed distilling-flask, and the lamp, are supported by rings held by a single upright rod. The receiving flask is readily connected with the condenser by means of a single bent tube provided with a rubber stopper. The cold-water pipe supplying the condensers is shown at the top, and the gas-supply pipe at the bottom.

The distillate, made up to 100 cc., is thoroughly shaken and its specific gravity taken at exactly  $15.6^{\circ}$  in a pycnometer, or by the Westphal balance. From the specific gravity the corresponding percentage of alcohol by weight or volume, or the grams per 100 cc. in the distillate, is ascertained by reference to the accompanying tables.

To obtain percentage of alcohol by weight in the sample, multiply the per cent by weight in the distillate by the weight of the distillate, and divide by the weight of the sample taken; to obtain per cent by volume, multiply the per cent by volume in the distillate by 100, and divide by the volume of the sample used.

(2) *From the Specific Gravity of the Sample.*—In the case of distilled liquors having very little residue, an approximation to the true



percentage of alcohol may be obtained by using the alcohol table in connection with the specific gravity of the liquor itself. The accuracy of this method depends largely on the freedom from residue, being absolutely correct for mixtures of alcohol and water only.

(3) *By Evaporation.*—Determine the specific gravity of the sample, evaporate a measured portion of the liquor (50 or 100 cc.) in a porcelain

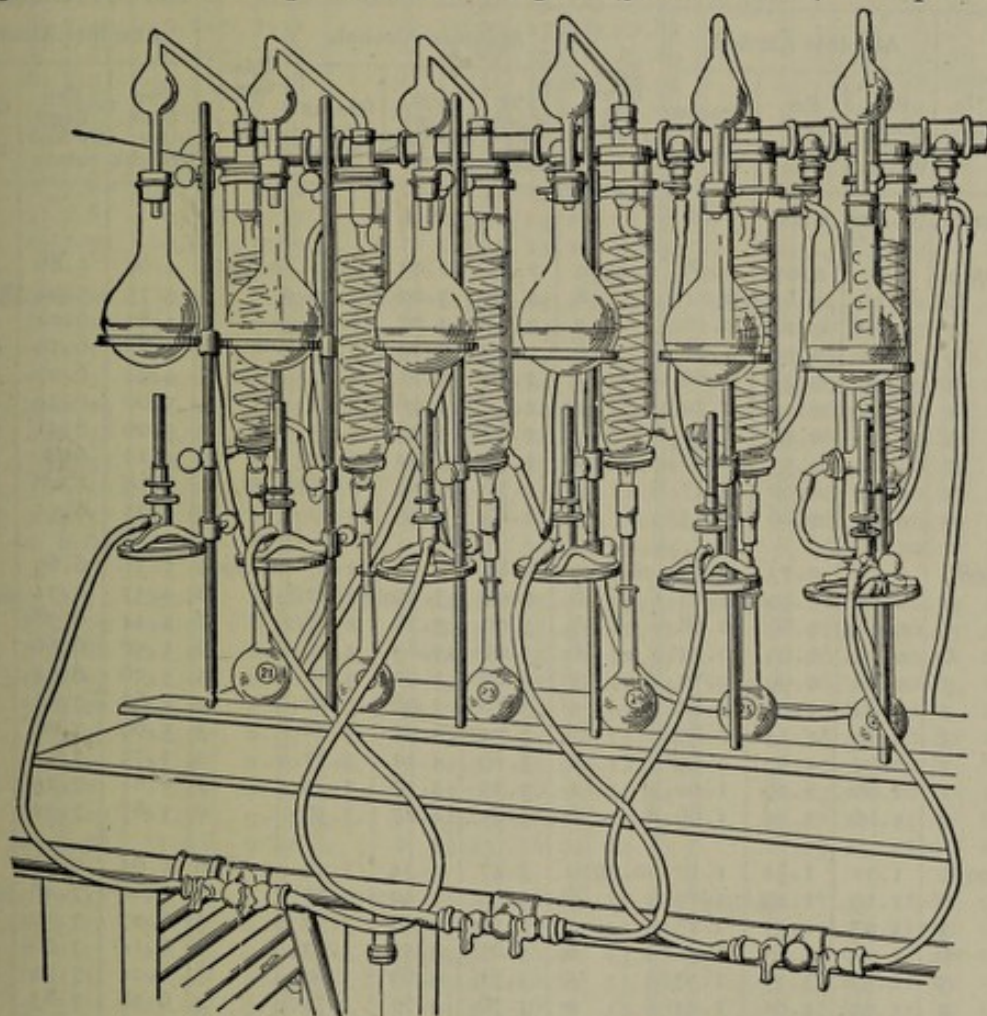


FIG. 112.—Bank of Stills for Alcohol Determination.

dish over the water-bath to one-fourth its bulk, make up to its original volume with distilled water, and determine the specific gravity of this second or dealcoholized portion. Add 1 to the original specific gravity, and from this subtract the second specific gravity. The difference is the specific gravity corresponding to the alcohol in the liquor, the per cent of which is found from the table.

*Example.*—Suppose the specific gravity of the original sample to be 0.9900 while that of the dealcoholized sample is 1.0009. Then  $1.9900 - 1.0009 = 0.9891$ .  $\therefore$  Per Cent by volume of alcohol = 8.10.



## SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL.

(ACCORDING TO HEHNER.)

Spec. Grav. at 15.6° C.	Absolute Alcohol.			Spec. Grav. at 15.6° C.	Absolute Alcohol.			Spec. Grav. at 15.6° C.	Absolute Alcohol.		
	Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.		Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.		Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.
1.0000	0.00	0.00	0.00								
0.9999	0.05	0.07	0.05	0.9959	2.33	2.93	2.32	0.9919	4.69	5.86	4.65
8	0.11	0.13	0.11	8	2.39	3.00	2.38	8	4.75	5.94	4.71
7	0.16	0.20	0.16	7	2.44	3.07	2.43	7	4.81	6.02	4.77
6	0.21	0.26	0.21	6	2.50	3.14	2.49	6	4.87	6.10	4.83
5	0.26	0.33	0.26	5	2.56	3.21	2.55	5	4.94	6.17	4.90
4	0.32	0.40	0.32	4	2.61	3.28	2.60	4	5.00	6.24	4.95
3	0.37	0.46	0.37	3	2.67	3.35	2.65	3	5.06	6.32	5.01
2	0.42	0.53	0.42	2	2.72	3.42	2.70	2	5.12	6.40	5.07
1	0.47	0.60	0.47	1	2.78	3.49	2.76	1	5.19	6.48	5.14
0	0.53	0.66	0.53	0	2.83	3.55	2.81	0	5.25	6.55	5.20
0.9989	0.58	0.73	0.58	0.9949	2.89	3.62	2.87	0.9909	5.31	6.63	5.26
8	0.63	0.79	0.63	8	2.94	3.69	2.92	8	5.37	6.71	5.32
7	0.68	0.86	0.68	7	3.00	3.76	2.98	7	5.44	6.78	5.39
6	0.74	0.93	0.74	6	3.06	3.83	3.04	6	5.50	6.86	5.45
5	0.79	0.99	0.79	5	3.12	3.90	3.10	5	5.56	6.94	5.51
4	0.84	1.06	0.84	4	3.18	3.98	3.16	4	5.62	7.01	5.57
3	0.89	1.13	0.89	3	3.24	4.05	3.22	3	5.69	7.09	5.64
2	0.95	1.19	0.95	2	3.29	4.12	3.27	2	5.75	7.17	5.70
1	1.00	1.26	1.00	1	3.35	4.20	3.33	1	5.81	7.25	5.76
0	1.06	1.34	1.06	0	3.41	4.27	3.39	0	5.87	7.32	5.81
0.9979	1.12	1.42	1.12	0.9939	3.47	4.34	3.45	0.9899	5.94	7.40	5.88
8	1.19	1.49	1.19	8	3.53	4.42	3.51	8	6.00	7.48	5.94
7	1.25	1.57	1.25	7	3.59	4.49	3.57	7	6.07	7.57	6.01
6	1.31	1.65	1.31	6	3.65	4.56	3.63	6	6.14	7.66	6.07
5	1.37	1.73	1.37	5	3.71	4.63	3.69	5	6.21	7.74	6.14
4	1.44	1.81	1.44	4	3.76	4.71	3.74	4	6.28	7.83	6.21
3	1.50	1.88	1.50	3	3.82	4.78	3.80	3	6.36	7.92	6.29
2	1.56	1.96	1.56	2	3.88	4.85	3.85	2	6.43	8.01	6.36
1	1.62	2.04	1.61	1	3.94	4.93	3.91	1	6.50	8.10	6.43
0	1.69	2.12	1.68	0	4.00	5.00	3.97	0	6.57	8.18	6.50
0.9969	1.75	2.20	1.74	0.9929	4.06	5.08	4.03	0.9889	6.64	8.27	6.57
8	1.81	2.27	1.80	8	4.12	5.16	4.09	8	6.71	8.36	6.63
7	1.87	2.35	1.86	7	4.19	5.24	4.16	7	6.78	8.45	6.70
6	1.94	2.43	1.93	6	4.25	5.32	4.22	6	6.86	8.54	6.78
5	2.00	2.51	1.99	5	4.31	5.39	4.28	5	6.93	8.63	6.85
4	2.06	2.58	2.05	4	4.37	5.47	4.34	4	7.00	8.72	6.92
3	2.11	2.62	2.10	3	4.44	5.55	4.40	3	7.07	8.80	6.99
2	2.17	2.72	2.16	2	4.50	5.63	4.46	2	7.13	8.88	7.05
1	2.22	2.79	2.21	1	4.56	5.71	4.52	1	7.20	8.96	7.12
0	2.28	2.86	2.27	0	4.62	5.78	4.58	0	7.27	9.04	7.19



## SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(Continued).

Spec. Grav. at 15.6° C.	Absolute Alcohol.			Spec. Grav. at 15.6° C.	Absolute Alcohol.			Spec. Grav. at 15.6° C.	Absolute Alcohol.		
	Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.		Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.		Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.
0.9879	7.33	9.13	7.24	0.9829	10.92	13.52	10.73	0.9779	14.91	18.36	14.58
8	7.40	9.21	7.31	8	11.00	13.62	10.81	8	15.00	18.48	14.66
7	7.47	9.29	7.37	7	11.08	13.71	10.89	7	15.08	18.58	14.74
6	7.53	9.37	7.43	6	11.15	13.81	10.95	6	15.17	18.68	14.83
5	7.60	9.45	7.50	5	11.23	13.90	11.03	5	15.25	18.78	14.90
4	7.67	9.54	7.57	4	11.31	13.99	11.11	4	15.33	18.88	14.98
3	7.73	9.62	7.63	3	11.38	14.09	11.18	3	15.42	18.98	15.07
2	7.80	9.70	7.70	2	11.46	14.18	11.26	2	15.50	19.08	15.14
1	7.87	9.78	7.77	1	11.54	14.27	11.33	1	15.58	19.18	15.21
0	7.93	9.86	7.83	0	11.62	14.37	11.41	0	15.67	19.28	15.30
0.9860	8.00	9.95	7.89	0.9819	11.69	14.46	11.48	0.9769	15.75	19.39	15.38
8	8.07	10.03	7.96	8	11.77	14.56	11.56	8	15.83	19.49	15.46
7	8.14	10.12	8.04	7	11.85	14.65	11.64	7	15.92	19.59	15.54
6	8.21	10.21	8.10	6	11.92	14.74	11.70	6	16.00	19.68	15.62
5	8.29	10.30	8.17	5	12.00	14.84	11.78	5	16.08	19.78	15.70
4	8.36	10.38	8.24	4	12.08	14.93	11.85	4	16.15	19.87	15.76
3	8.43	10.47	8.31	3	12.15	15.02	11.92	3	16.23	19.96	15.84
2	8.50	10.56	8.38	2	12.23	15.12	12.00	2	16.31	20.06	15.90
1	8.57	10.65	8.45	1	12.31	15.21	12.08	1	16.38	20.15	15.99
0	8.64	10.73	8.52	0	12.38	15.30	12.14	0	16.46	20.24	16.06
0.9850	8.71	10.82	8.58	0.9809	12.46	15.40	12.22	0.9759	16.54	20.33	16.13
8	8.79	10.91	8.66	8	12.54	15.49	12.30	8	16.62	20.43	16.21
7	8.86	11.00	8.73	7	12.62	15.58	12.37	7	16.69	20.52	16.28
6	8.93	11.08	8.80	6	12.69	15.68	12.44	6	16.77	20.61	16.35
5	9.00	11.17	8.87	5	12.77	15.77	12.51	5	16.85	20.71	16.43
4	9.07	11.26	8.93	4	12.85	15.86	12.59	4	16.92	20.80	16.50
3	9.14	11.35	9.00	3	12.92	15.96	12.66	3	17.00	20.89	16.57
2	9.21	11.44	9.07	2	13.00	16.05	12.74	2	17.08	20.99	16.65
1	9.29	11.52	9.14	1	13.08	16.15	12.81	1	17.17	21.09	16.74
0	9.36	11.61	9.22	0	13.15	16.24	12.89	0	17.25	21.19	16.81
0.9840	9.43	11.70	9.29	0.9799	13.23	16.33	12.96	0.9749	17.33	21.29	16.89
8	9.50	11.79	9.35	8	13.31	16.43	13.03	8	17.42	21.39	16.97
7	9.57	11.87	9.42	7	13.38	16.52	13.10	7	17.50	21.49	17.05
6	9.64	11.96	9.49	6	13.46	16.61	13.18	6	17.58	21.59	17.13
5	9.71	12.05	9.56	5	13.54	16.70	13.26	5	17.67	21.69	17.20
4	9.79	12.13	9.64	4	13.62	16.80	13.33	4	17.75	21.79	17.29
3	9.86	12.22	9.71	3	13.69	16.89	13.40	3	17.83	21.89	17.37
2	9.93	12.31	9.77	2	13.77	16.98	13.48	2	17.92	21.99	17.46
1	10.00	12.40	9.84	1	13.85	17.08	13.56	1	18.00	22.09	17.54
0	10.03	12.49	9.92	0	13.92	17.17	13.63	0	18.08	22.18	17.61
0.9830	10.15	12.58	9.99	0.9789	14.00	17.26	13.71	0.9739	18.15	22.27	17.68
8	10.23	12.68	10.06	8	14.09	17.37	13.79	8	18.23	22.36	17.76
7	10.31	12.77	10.13	7	14.18	17.48	13.88	7	18.31	22.46	17.82
6	10.38	12.87	10.20	6	14.27	17.59	13.96	6	18.38	22.55	17.90
5	10.46	12.96	10.28	5	14.36	17.70	14.04	5	18.46	22.64	17.97
4	10.54	13.05	10.36	4	14.45	17.81	14.13	4	18.54	22.73	18.05
3	10.62	13.15	10.44	3	14.55	17.92	14.23	3	18.62	22.82	18.13
2	10.69	13.24	10.51	2	14.64	18.03	14.32	2	18.69	22.92	18.19
1	10.77	13.34	10.59	1	14.73	18.14	14.39	1	18.77	23.01	18.27
0	10.85	13.43	10.67	0	14.82	18.25	14.48	0	18.85	23.10	18.34



## SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(Continued).

Spec. Grav. at 15.6° C.	Absolute Alcohol.			Spec. Grav. at 15.6° C.	Absolute Alcohol.			Spec. Grav. at 15.6° C.	Absolute Alcohol.		
	Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.		Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.		Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.
0.9729	18.92	23.19	18.41	0.9679	22.92	27.95	22.18	0.9629	26.60	32.27	25.61
8	19.00	23.18	18.48	8	23.00	28.04	22.26	8	26.67	32.34	25.67
7	19.08	23.38	18.56	7	23.08	28.13	22.33	7	26.73	32.42	25.73
6	19.17	23.48	18.65	6	23.15	28.22	22.40	6	26.80	32.50	25.79
5	19.25	23.58	18.73	5	23.23	28.31	22.47	5	26.87	32.58	25.85
4	19.33	23.68	18.80	4	23.31	28.41	22.54	4	26.93	32.65	25.91
3	19.42	23.78	18.88	3	23.38	28.50	22.61	3	27.00	32.73	25.98
2	19.50	23.88	18.95	2	23.46	28.59	22.69	2	27.07	32.81	26.04
1	19.58	23.98	19.03	1	23.54	28.68	22.76	1	27.14	32.90	26.10
0	19.67	24.08	19.12	0	23.62	28.77	22.83	0	27.21	32.98	26.17
0.9719	19.75	24.18	19.19	0.9669	23.69	28.86	22.90	0.9619	27.29	33.06	26.25
8	19.83	24.28	19.27	8	23.77	28.95	22.97	8	27.36	33.15	26.31
7	19.92	24.38	19.36	7	23.85	29.04	23.05	7	27.43	33.23	26.37
6	20.00	24.48	19.44	6	23.92	29.13	23.11	6	27.50	33.31	26.43
5	20.08	24.58	19.51	5	24.00	29.22	23.19	5	27.57	33.39	26.51
4	20.17	24.68	19.59	4	24.08	29.31	23.27	4	27.64	33.48	26.57
3	20.25	24.78	19.66	3	24.15	29.40	23.33	3	27.71	33.56	26.64
2	20.33	24.88	19.74	2	24.23	29.49	23.40	2	27.79	33.64	26.71
1	20.42	24.98	19.83	1	24.31	29.58	23.48	1	27.86	33.73	26.78
0	20.50	25.07	19.90	0	24.38	29.67	23.55	0	27.93	33.81	26.84
0.9709	20.58	25.17	19.98	0.9659	24.46	29.76	23.62	0.9609	28.00	33.89	26.90
8	20.67	25.27	20.07	8	24.54	29.86	23.70	8	28.06	33.97	26.96
7	20.75	25.37	20.14	7	24.62	29.95	23.77	7	28.12	34.04	27.01
6	20.83	25.47	20.22	6	24.69	30.04	23.84	6	28.19	34.11	27.07
5	20.92	25.57	20.30	5	24.77	30.13	23.91	5	28.25	34.18	27.13
4	21.00	25.67	20.33	4	24.85	30.22	23.99	4	28.31	34.25	27.18
3	21.08	25.76	20.46	3	24.92	30.31	24.05	3	28.37	34.33	27.24
2	21.15	25.86	20.52	2	25.00	30.40	24.12	2	28.44	34.40	27.31
1	21.23	25.95	20.59	1	25.07	30.48	24.19	1	28.50	34.47	27.36
0	21.31	26.04	20.67	0	25.14	30.57	24.26	0	28.56	34.54	27.42
0.9699	21.38	26.13	20.73	0.9649	25.21	30.65	24.32	0.9599	28.62	34.61	27.47
8	21.46	26.22	20.81	8	25.29	30.73	24.39	8	28.69	34.69	27.53
7	21.54	26.31	20.89	7	25.36	30.82	24.46	7	28.75	34.76	27.59
6	21.62	26.40	20.96	6	25.43	30.90	24.53	6	28.81	34.83	27.64
5	21.69	26.49	21.03	5	25.50	30.98	24.59	5	28.87	34.90	27.70
4	21.77	26.58	21.11	4	25.57	31.07	24.66	4	28.94	34.97	27.76
3	21.85	26.67	21.18	3	25.64	31.13	24.72	3	29.00	35.05	27.82
2	21.92	26.77	21.25	2	25.71	31.23	24.79	2	29.07	35.12	27.89
1	22.00	26.86	21.33	1	25.79	31.32	24.86	1	29.13	35.20	27.95
0	22.08	26.95	21.40	0	25.86	31.40	24.93	0	29.20	35.28	28.00
0.9689	22.15	27.04	21.47	0.9639	25.93	31.48	24.99	0.9589	29.27	35.35	28.07
8	22.23	27.13	21.54	8	26.00	31.57	25.06	8	29.33	35.43	28.12
7	22.31	27.22	21.61	7	26.07	31.65	25.12	7	29.40	35.51	28.18
6	22.38	27.31	21.68	6	26.13	31.72	25.18	6	29.47	35.58	28.24
5	22.46	27.40	21.76	5	26.20	31.80	25.23	5	29.53	35.66	28.30
4	22.54	27.49	21.83	4	26.27	31.88	25.30	4	29.60	35.74	28.36
3	22.62	27.59	21.90	3	26.33	31.96	25.36	3	29.67	35.81	28.43
2	22.69	27.68	21.96	2	26.40	32.03	25.43	2	29.73	35.89	28.48
1	22.77	27.77	22.01	1	26.47	32.11	25.49	1	29.80	35.97	28.54
0	22.85	27.86	22.12	0	26.53	32.19	25.55	0	29.87	36.04	28.61



## SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(Continued).

Spec. Grav. at 15.6° C.	Absolute Alcohol.			Spec. Grav. at 15.6° C.	Absolute Alcohol.			Spec. Grav. at 15.6° C.	Absolute Alcohol.		
	Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.		Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.		Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.
0.9579	29.93	36.12	28.67	0.9529	32.94	39.54	31.38	0.9479	35.55	42.45	33.70
8	30.00	36.20	28.73	8	33.00	39.61	31.43	8	35.60	42.51	33.75
7	30.06	36.26	28.78	7	33.06	39.68	31.48	7	35.65	42.56	33.79
6	30.11	36.32	28.82	6	33.12	39.74	31.53	6	35.70	42.62	33.83
5	30.17	36.39	28.88	5	33.18	39.81	31.59	5	35.75	42.67	33.88
4	30.22	36.45	28.92	4	33.24	39.87	31.63	4	35.80	42.73	33.92
3	30.28	36.51	28.98	3	33.29	39.94	31.69	3	35.85	42.78	33.97
2	30.33	36.57	29.03	2	33.35	40.01	31.74	2	35.90	42.84	34.01
1	30.39	36.64	29.08	1	33.41	40.07	31.80	1	35.95	42.89	34.05
0	30.44	36.70	29.13	0	33.47	40.14	31.86	0	36.00	42.95	34.09
0.9569	30.50	36.76	29.18	0.9519	33.53	40.20	31.91	0.9469	36.06	43.01	34.14
8	30.56	36.83	29.23	8	33.59	40.27	31.96	8	36.11	43.07	34.09
7	30.61	36.89	29.27	7	33.65	40.34	32.01	7	36.17	43.13	34.24
6	30.67	36.95	29.33	6	33.71	40.40	32.07	6	36.22	43.19	34.28
5	30.72	37.02	29.38	5	33.76	40.47	32.12	5	36.28	43.26	34.34
4	30.78	37.08	29.43	4	33.82	40.53	32.17	4	36.33	43.32	34.38
3	30.83	37.14	29.48	3	33.88	40.60	32.22	3	36.39	43.38	34.44
2	30.89	37.20	29.53	2	33.94	40.67	32.27	2	36.44	43.44	34.48
1	30.94	37.27	29.58	1	34.00	40.74	32.32	1	36.50	43.50	34.54
0	31.00	37.34	29.63	0	34.05	40.79	32.37	0	36.56	43.56	34.58
0.9559	31.06	37.41	29.69	0.9509	34.10	40.84	32.41	0.9459	36.61	43.63	34.63
8	31.12	37.48	29.74	8	34.14	40.90	32.45	8	36.67	43.69	34.69
7	31.19	37.55	29.81	7	34.19	40.95	32.49	7	36.72	43.75	34.73
6	31.25	37.62	29.86	6	34.24	41.00	32.54	6	36.78	43.81	34.79
5	31.31	37.69	29.91	5	34.29	41.05	32.59	5	36.83	43.87	34.83
4	31.37	37.76	29.97	4	34.33	41.11	32.63	4	36.89	43.93	34.88
3	31.44	37.83	30.03	3	34.38	41.16	32.67	3	36.94	44.00	34.92
2	31.50	37.90	30.09	2	34.43	41.21	32.71	2	37.00	44.06	34.96
1	31.56	37.97	30.14	1	34.48	41.26	32.75	1	37.06	44.12	35.02
0	31.62	38.04	30.20	0	34.52	41.32	32.79	0	37.11	44.18	35.07
0.9549	31.69	38.11	30.26	0.9499	34.57	41.37	32.84	0.9449	37.17	44.24	35.12
8	31.75	38.18	30.31	8	34.62	41.42	32.88	8	37.22	44.30	35.16
7	31.81	38.25	30.36	7	34.67	41.48	32.92	7	37.28	44.36	35.21
6	31.87	38.33	30.42	6	34.71	41.53	32.96	6	37.33	44.43	35.26
5	31.94	38.40	30.48	5	34.76	41.58	33.00	5	37.39	44.49	35.31
4	32.00	38.47	30.53	4	34.81	41.63	33.04	4	37.44	44.55	35.35
3	32.06	38.53	30.59	3	34.86	41.69	33.09	3	37.50	44.61	35.41
2	32.12	38.60	30.64	2	34.90	41.74	33.13	2	37.56	44.67	35.46
1	32.19	38.68	30.71	1	34.95	41.79	33.17	1	37.61	44.73	35.51
0	32.25	38.75	30.77	0	35.00	41.84	33.21	0	37.67	44.79	35.56
0.9539	32.31	38.82	30.81	0.9489	35.05	41.90	33.26	0.9439	37.73	44.86	35.60
8	32.37	38.89	30.87	8	35.10	41.95	33.30	8	37.78	44.92	35.65
7	32.44	38.96	30.93	7	35.15	42.01	33.34	7	37.83	44.98	35.70
6	32.50	39.04	30.99	6	35.20	42.06	33.39	6	37.89	45.04	35.75
5	32.56	39.11	31.05	5	35.25	42.12	33.43	5	37.94	45.10	35.80
4	32.62	39.18	31.10	4	35.30	42.17	33.48	4	38.00	45.16	35.85
3	32.69	39.25	31.15	3	35.35	42.23	33.53	3	38.06	45.22	35.90
2	32.75	39.32	31.20	2	35.40	42.29	33.57	2	38.11	45.28	35.95
1	32.81	39.40	31.26	1	35.45	42.34	33.61	1	38.17	45.34	36.00
0	32.87	39.47	31.32	0	35.50	42.40	33.65	0	38.22	45.41	36.04



## SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(Continued).

Spec. Grav. at 15.6° C.	Absolute Alcohol.			Spec. Grav. at 15.6° C.	Absolute Alcohol.			Spec. Grav. at 15.6° C.	Absolute Alcohol.		
	Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.		Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.		Per Cent by Weight	Per Cent by Vol- ume.	Grams per 100 cc.
0.9429	38.28	45.47	36.08	0.9379	40.85	48.26	38.31	0.9329	43.29	50.87	40.38
8	38.33	45.53	36.13	8	40.90	48.32	38.35	8	43.33	50.92	40.42
7	38.39	45.59	36.18	7	40.95	48.37	38.39	7	43.39	50.97	40.46
6	38.44	45.65	36.23	6	41.00	48.43	38.44	6	43.43	51.02	40.50
5	38.50	45.71	36.28	5	41.05	48.48	38.48	5	43.48	51.07	40.54
4	38.56	45.77	36.33	4	41.10	48.54	38.52	4	43.52	51.12	40.58
3	38.61	45.83	36.38	3	41.15	48.59	38.58	3	43.57	51.17	40.62
2	38.67	45.89	36.43	2	41.20	48.64	38.62	2	43.62	51.22	40.66
1	38.72	45.95	36.48	1	41.25	48.70	38.66	1	43.67	51.27	40.70
0	38.78	46.02	36.53	0	41.30	48.75	38.70	0	43.71	51.32	40.74
0.9419	38.83	46.08	36.57	0.9369	41.35	48.80	38.74	0.9319	43.76	51.38	40.78
8	38.89	46.14	36.62	8	41.40	48.86	38.78	8	43.81	51.43	40.81
7	38.94	46.20	36.67	7	41.45	48.91	38.82	7	43.86	51.48	40.85
6	39.00	46.26	36.72	6	41.50	48.97	38.87	6	43.90	51.53	40.89
5	39.05	46.32	36.76	5	41.55	49.02	38.91	5	43.95	51.58	40.93
4	39.10	46.37	36.80	4	41.60	49.07	38.95	4	44.00	51.63	40.97
3	39.15	46.42	36.85	3	41.65	49.13	38.99	3	44.05	51.68	41.01
2	39.20	46.48	36.89	2	41.70	49.18	39.04	2	44.09	51.72	41.05
1	39.25	46.53	36.94	1	41.75	49.23	39.08	1	44.14	51.77	41.09
0	39.30	46.59	36.98	0	41.80	49.29	39.13	0	44.18	51.82	41.13
0.9409	39.35	46.64	37.02	0.9359	41.85	49.34	39.17	0.9309	44.23	51.87	41.17
8	39.40	46.70	37.07	8	41.90	49.40	39.21	8	44.27	51.91	41.20
7	39.45	46.75	37.11	7	41.95	49.45	39.25	7	44.32	51.96	41.24
6	39.50	46.80	37.15	6	42.00	49.50	39.30	6	44.36	52.01	41.28
5	39.55	46.86	37.19	5	42.05	49.55	39.34	5	44.41	52.06	41.31
4	39.60	46.91	37.23	4	42.10	49.61	39.38	4	44.46	52.10	41.35
3	39.65	46.97	37.27	3	42.14	49.66	39.42	3	44.50	52.15	41.39
2	39.70	47.02	37.32	2	42.19	49.71	39.46	2	44.55	52.20	41.43
1	39.75	47.08	37.36	1	42.24	49.76	39.50	1	44.59	52.25	41.47
0	39.80	47.13	37.41	0	42.29	49.81	39.54	0	44.64	52.29	41.51
0.9399	39.85	47.18	37.45	0.9349	42.33	49.86	39.58	0.9299	44.68	52.34	41.55
8	39.90	47.24	37.49	8	42.38	49.91	39.62	8	44.73	52.39	41.59
7	39.95	47.29	37.53	7	42.43	49.96	39.66	7	44.77	52.44	41.63
6	40.00	47.35	37.58	6	42.48	50.01	39.70	6	44.82	52.48	41.67
5	40.05	47.40	37.62	5	42.52	50.06	39.74	5	44.86	52.53	41.70
4	40.10	47.45	37.67	4	42.57	50.11	39.78	4	44.91	52.58	41.74
3	40.15	47.51	37.71	3	42.62	50.16	39.82	3	44.96	52.63	41.77
2	40.20	47.56	37.75	2	42.67	50.21	39.86	2	45.00	52.68	41.81
1	40.25	47.62	37.80	1	42.71	50.26	39.90	1	45.05	52.72	41.85
0	40.30	47.67	37.84	0	42.76	50.31	39.94	0	45.09	52.77	41.89
0.9389	40.35	47.72	37.88	0.9339	42.81	50.37	39.98	0.9289	45.14	52.82	41.93
8	40.40	47.78	37.92	8	42.86	50.42	40.02	8	45.18	52.87	41.97
7	40.45	47.83	37.96	7	42.90	50.47	40.06	7	45.23	52.91	42.00
6	40.50	47.89	38.00	6	42.95	50.52	40.10	6	45.27	52.96	42.04
5	40.55	47.94	38.05	5	43.00	50.57	40.14	5	45.32	53.01	42.08
4	40.60	47.99	38.09	4	43.05	50.62	40.18	4	45.36	53.06	42.12
3	40.65	48.05	38.13	3	43.10	50.67	40.22	3	45.41	53.10	42.16
2	40.70	48.10	38.18	2	43.13	50.72	40.26	2	45.46	53.15	42.19
1	40.75	48.16	38.22	1	43.19	50.77	40.30	1	45.50	53.20	42.23
0	40.80	48.21	38.27	0	43.24	50.82	40.34	0	45.55	53.24	42.27



## SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(Continued).

Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.	
	Per Cent by Weight.	Per Cent by Vol- ume.		Per Cent by Weight.	Per Cent by Vol- ume.		Per Cent by Weight.	Per Cent by Vol- ume.
0.9279	45.59	53.29	0.9229	47.86	55.65	0.9179	50.13	57.97
8	45.64	53.34	8	47.91	55.69	8	50.17	58.01
7	45.68	53.39	7	47.96	55.74	7	50.22	58.06
6	45.73	53.43	6	48.00	55.79	6	50.26	58.10
5	45.77	53.48	5	48.05	55.83	5	50.30	58.14
4	45.82	53.53	4	48.09	55.88	4	50.35	58.19
3	45.86	53.58	3	48.14	55.93	3	50.39	58.23
2	45.91	53.62	2	48.18	55.97	2	50.43	58.28
1	45.96	53.67	1	48.23	56.02	1	50.48	58.32
0	46.00	53.72	0	48.27	56.07	0	50.52	58.36
0.9269	46.05	53.77	0.9219	48.32	56.11	0.9169	50.57	58.41
8	46.09	53.81	8	48.36	56.16	8	50.61	58.45
7	46.14	53.86	7	48.41	56.21	7	50.65	58.50
6	46.18	53.91	6	48.46	56.25	6	50.70	58.54
5	46.23	53.95	5	48.50	56.30	5	50.74	58.58
4	46.27	54.00	4	48.55	56.35	4	50.78	58.63
3	46.32	54.05	3	48.59	56.40	3	50.83	58.67
2	46.36	54.10	2	48.64	56.44	2	50.87	58.72
1	46.41	54.14	1	48.68	56.49	1	50.91	58.76
0	46.46	54.19	0	48.73	56.54	0	50.96	58.80
0.9259	46.50	54.24	0.9209	48.77	56.58	0.9159	51.00	58.85
8	46.55	54.29	8	48.82	56.63	8	51.04	58.89
7	46.59	54.33	7	48.86	56.68	7	51.08	58.93
6	46.64	54.38	6	48.91	56.72	6	51.13	58.97
5	46.68	54.43	5	48.96	56.77	5	51.17	59.01
4	46.73	54.47	4	49.00	56.82	4	51.21	59.05
3	46.77	54.52	3	49.04	56.86	3	51.25	59.09
2	46.82	54.57	2	49.08	56.90	2	51.29	59.14
1	46.86	54.62	1	49.12	56.94	1	51.33	59.18
0	46.91	54.66	0	49.16	56.98	0	51.38	59.22
0.9249	46.96	54.71	0.9199	49.20	57.02	0.9149	51.42	59.26
8	47.00	54.76	Proof 8	49.24	57.06	8	51.46	59.30
7	47.05	54.80	7	49.29	57.10	7	51.50	59.34
6	47.09	54.85	6	49.34	57.15	6	51.54	59.39
5	47.14	54.90	5	49.39	57.20	5	51.58	59.43
4	47.18	54.95	4	49.44	57.25	4	51.63	59.47
3	47.23	54.99	3	49.49	57.30	3	51.67	59.51
2	47.27	55.04	2	49.54	57.35	2	51.71	59.55
1	47.32	55.09	1	49.59	57.40	1	51.75	59.59
0	47.36	55.13	0	49.64	57.45	0	51.79	59.63
0.9239	47.41	55.18	0.9189	49.68	57.49	0.9139	51.83	59.68
8	47.46	55.23	8	49.73	57.54	8	51.88	59.72
7	47.50	55.27	7	49.77	57.59	7	51.92	59.76
6	47.55	55.32	6	49.82	57.64	6	51.96	59.80
5	47.59	55.37	5	49.86	57.69	5	52.00	59.84
4	47.64	55.41	4	49.91	57.74	4	52.05	59.89
3	47.68	55.46	3	49.95	57.79	3	52.09	59.93
2	47.73	55.51	2	50.00	57.84	2	52.14	59.98
1	47.77	55.55	1	50.04	57.88	1	52.18	60.02
0	47.82	55.60	0	50.09	58.92	0	52.23	60.07



## SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(Continued).

Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.	
	Per Cent by Weight.	Per Cent by Vol- ume.		Per Cent by Weight.	Per Cent by Vol- ume.		Per Cent by Weight.	Per Cent by Vol- ume.
0.9129	52.27	60.12	0.9079	54.52	62.36	0.9029	56.82	64.63
8	52.32	60.16	8	54.57	62.41	8	56.86	64.67
7	52.36	60.21	7	54.62	62.45	7	56.91	64.71
6	52.41	60.25	6	54.67	62.50	6	56.95	64.76
5	52.45	60.30	5	54.71	62.55	5	57.00	64.80
4	52.50	60.34	4	54.76	62.60	4	57.04	64.85
3	52.55	60.39	3	54.81	62.65	3	57.08	64.89
2	52.59	60.44	2	54.86	62.69	2	57.13	64.93
1	52.64	60.47	1	54.90	62.74	1	57.17	64.97
0	52.68	60.52	0	54.95	62.79	0	57.21	65.01
0.9119	52.73	60.56	0.9069	55.00	62.84	0.9019	57.25	65.05
8	52.77	60.61	8	55.05	62.88	8	57.29	65.09
7	52.82	60.65	7	55.09	62.93	7	57.33	65.13
6	52.86	60.70	6	55.14	62.97	6	57.38	65.17
5	52.91	60.74	5	55.18	63.02	5	57.42	65.21
4	52.95	60.79	4	55.23	63.06	4	57.46	65.25
3	53.00	60.85	3	55.27	63.11	3	57.50	65.29
2	53.04	60.89	2	55.32	63.15	2	57.54	65.33
1	53.09	60.93	1	55.36	63.20	1	57.58	65.37
0	53.13	60.97	0	55.41	63.24	0	57.63	65.41
0.9109	53.17	61.02	0.9059	55.45	63.28	0.9009	57.67	65.45
8	53.22	61.06	8	55.50	63.33	8	57.71	65.49
7	53.26	61.10	7	55.55	63.37	7	57.75	65.53
6	53.30	61.15	6	55.59	63.42	6	57.79	65.57
5	53.35	61.19	5	55.64	63.46	5	57.83	65.61
4	53.39	61.23	4	55.68	63.51	4	57.88	65.65
3	53.43	61.28	3	55.73	63.55	3	57.92	65.69
2	53.48	61.32	2	55.77	63.60	2	57.96	65.73
1	53.52	61.36	1	55.82	63.64	1	58.00	65.77
0	53.57	61.40	0	55.86	63.69	0	58.05	65.81
0.9099	53.61	61.45	0.9049	55.91	63.73	0.8999	58.09	65.85
8	53.65	61.49	8	55.95	63.78	8	58.14	65.90
7	53.70	61.53	7	56.00	63.82	7	58.18	65.94
6	53.74	61.58	6	56.05	63.87	6	58.23	65.99
5	53.78	61.62	5	56.09	63.91	5	58.27	66.03
4	53.83	61.66	4	56.14	63.96	4	58.32	66.07
3	53.87	61.71	3	56.18	64.00	3	58.36	66.12
2	53.91	61.75	2	56.23	64.05	2	58.41	66.16
1	53.96	61.79	1	56.27	64.09	1	58.45	66.21
0	54.00	61.84	0	56.32	64.14	0	58.50	66.25
0.9089	54.05	61.88	0.9039	56.36	64.18	0.8989	58.55	66.29
8	54.10	61.93	8	56.41	64.22	8	58.59	66.34
7	54.14	61.98	7	56.45	64.27	7	58.64	66.38
6	54.19	62.03	6	56.50	64.31	6	58.68	66.43
5	54.24	62.07	5	56.55	64.36	5	58.73	66.47
4	54.29	62.12	4	56.59	64.40	4	58.77	66.51
3	54.33	62.17	3	56.64	64.45	3	58.82	66.56
2	54.38	62.22	2	56.68	64.49	2	58.86	66.60
1	54.43	62.26	1	56.73	64.54	1	58.91	66.65
0	54.48	62.31	0	56.77	64.58	0	58.95	66.69



## SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(Continued).

Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.	
	Per Cent by Weight.	Per Cent by Vol- ume.		Per Cent by Weight.	Per Cent by Vol- ume.		Per Cent by Weight.	Per Cent by Vol- ume.
0.8979	59.00	66.74	0.8929	61.13	68.76	0.8879	63.30	70.81
8	59.04	66.78	8	61.17	68.80	8	63.35	70.85
7	59.09	66.82	7	61.21	68.83	7	63.39	70.89
6	59.13	66.86	6	61.25	68.87	6	63.43	70.93
5	59.17	66.90	5	61.29	68.91	5	63.48	70.97
4	59.22	66.94	4	61.33	68.95	4	63.52	71.01
3	59.26	66.99	3	61.38	68.99	3	63.57	71.05
2	59.30	67.03	2	61.42	69.03	2	63.61	71.09
1	59.35	67.07	1	61.46	69.07	1	63.65	71.13
0	59.39	67.11	0	61.50	69.11	0	63.70	71.17
0.8969	59.43	67.15	0.8919	61.54	69.15	0.8869	63.74	71.22
8	59.48	67.19	8	61.58	69.19	8	63.78	71.26
7	59.52	67.24	7	61.63	69.22	7	63.83	71.30
6	59.57	67.28	6	61.67	69.26	6	63.87	71.34
5	59.61	67.32	5	61.71	69.30	5	63.91	71.38
4	59.65	67.36	4	61.75	69.34	4	63.96	71.42
3	59.70	67.40	3	61.79	69.38	3	64.00	71.46
2	59.74	67.44	2	61.83	69.42	2	64.04	71.50
1	59.78	67.49	1	61.88	69.46	1	64.09	71.54
0	59.83	67.53	0	61.92	69.50	0	64.13	71.58
0.8959	59.87	67.57	0.8909	61.96	69.54	0.8859	64.17	71.62
8	59.91	67.61	8	62.00	69.58	8	64.22	71.66
7	59.96	67.65	7	62.05	69.62	7	64.26	71.70
6	60.00	67.69	6	62.09	69.66	6	64.30	71.74
5	60.04	67.73	5	62.14	69.71	5	64.35	71.78
4	60.08	67.77	4	62.18	69.75	4	64.39	71.82
3	60.13	67.81	3	62.23	69.79	3	64.43	71.86
2	60.17	67.85	2	62.27	69.84	2	64.48	71.90
1	60.21	67.89	1	62.32	69.88	1	64.52	71.94
0	60.26	67.93	0	62.36	69.92	0	64.57	71.98
0.8949	60.29	67.97	0.8899	62.41	69.96	0.8849	64.61	72.02
8	60.33	68.01	8	62.45	70.01	8	64.65	72.06
7	60.38	68.05	7	62.50	70.05	7	64.70	72.10
6	60.42	68.09	6	62.55	70.09	6	64.74	72.14
5	60.46	68.13	5	62.59	70.14	5	64.78	72.18
4	60.50	68.17	4	62.64	70.18	4	64.83	72.22
3	60.54	68.21	3	62.68	70.22	3	64.87	72.26
2	60.58	68.25	2	62.73	70.27	2	64.91	72.30
1	60.63	68.29	1	62.77	70.31	1	64.96	72.34
0	60.67	68.33	0	62.82	70.35	0	65.00	72.38
0.8939	60.71	68.36	0.8889	62.86	70.40	0.8839	65.04	72.42
8	60.76	68.40	8	62.91	70.44	8	65.08	72.46
7	60.79	68.44	7	62.95	70.48	7	65.13	72.50
6	60.83	68.48	6	63.00	70.52	6	65.17	72.54
5	60.88	68.52	5	63.04	70.57	5	65.21	72.58
4	60.92	68.56	4	63.09	70.61	4	65.25	72.61
3	60.96	68.60	3	63.13	70.65	3	65.29	72.65
2	61.00	68.64	2	63.17	70.69	2	65.33	72.69
1	61.04	68.68	1	63.22	70.73	1	65.38	72.73
0	61.08	68.72	0	63.26	70.77	0	65.42	72.77



## SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(Continued).

Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.	
	Per Cent by Weight.	Per Cent by Vol- ume.		Per Cent by Weight.	Per Cent by Vol- ume.		Per Cent by Weight.	Per Cent by Vol- ume.
0.8829	65.46	72.80	0.8779	67.58	74.74	0.8729	69.67	76.61
8	65.50	72.84	8	67.63	74.78	8	69.71	76.65
7	65.54	72.88	7	67.67	74.82	7	69.75	76.68
6	65.58	72.92	6	67.71	74.86	6	69.79	76.72
5	65.63	72.96	5	67.75	74.89	5	69.83	76.76
4	65.67	72.99	4	67.79	74.93	4	69.88	76.80
3	65.71	73.03	3	67.83	74.97	3	69.92	76.83
2	65.75	73.07	2	67.88	75.01	2	69.96	76.87
1	65.79	73.11	1	67.92	75.04	1	70.00	76.91
0	65.83	73.15	0	67.96	75.08	0	70.04	76.94
0.8819	65.88	73.19	0.8769	68.00	75.12	0.8719	70.08	76.98
8	65.92	73.22	8	68.04	75.16	8	70.12	77.01
7	65.96	73.26	7	68.08	75.19	7	70.16	77.05
6	66.00	73.30	6	68.13	75.23	6	70.20	77.08
5	66.04	73.34	5	68.17	75.27	5	70.24	77.12
4	66.09	73.38	4	68.21	75.30	4	70.28	77.15
3	66.13	73.42	3	68.25	75.34	3	70.32	77.19
2	66.17	73.46	2	68.29	75.38	2	70.36	77.22
1	66.22	73.50	1	68.33	75.42	1	70.40	77.25
0	66.26	73.54	0	68.38	75.45	0	70.44	77.29
0.8809	66.30	73.57	0.8759	68.42	75.49	0.8709	70.48	77.32
8	66.35	73.61	8	68.46	75.53	8	70.52	77.36
7	66.39	73.65	7	68.50	75.57	7	70.56	77.39
6	66.43	73.69	6	68.54	75.60	6	70.60	77.43
5	66.48	73.73	5	68.58	75.64	5	70.64	77.46
4	66.52	73.77	4	68.63	75.68	4	70.68	77.50
3	66.57	73.81	3	68.67	75.72	3	70.72	77.53
2	66.61	73.85	2	68.71	75.75	2	70.76	77.57
1	66.65	73.89	1	68.75	75.79	1	70.80	77.60
0	66.70	73.93	0	68.79	75.83	0	70.84	77.64
0.8799	66.74	73.97	0.8749	68.83	75.87	0.8699	70.88	77.67
8	66.78	74.01	8	68.88	75.90	8	70.92	77.71
7	66.83	74.05	7	68.92	75.94	7	70.96	77.74
6	66.87	74.09	6	68.96	75.98	6	71.00	77.78
5	66.91	74.13	5	69.00	76.01	5	71.04	77.82
4	66.96	74.17	4	69.04	76.05	4	71.08	77.85
3	67.00	74.22	3	69.08	76.09	3	71.13	77.89
2	67.04	74.25	2	69.13	76.13	2	71.17	77.93
1	67.08	74.29	1	69.17	76.16	1	71.21	77.96
0	67.13	74.33	0	69.21	76.20	0	71.25	78.00
0.8789	67.17	74.37	0.8739	69.25	76.24	0.8689	71.29	78.04
8	67.21	74.40	8	69.29	76.27	8	71.33	78.07
7	67.25	74.44	7	69.33	76.31	7	71.38	78.11
6	67.29	74.48	6	69.38	76.35	6	71.42	78.14
5	67.33	74.52	5	69.42	76.39	5	71.46	78.18
4	67.38	74.55	4	69.46	76.42	4	71.50	78.22
3	67.42	74.59	3	69.50	76.46	3	71.54	78.25
2	67.46	74.63	2	69.54	76.50	2	71.58	78.29
1	67.50	74.67	1	69.58	76.53	1	71.63	78.33
0	67.54	74.70	0	69.63	76.57	0	71.67	78.36



## SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(Continued).

Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.	
	Per Cent by Weight.	Per Cent by Vol- ume.		Per Cent by Weight.	Per Cent by Vol- ume.		Per Cent by Weight.	Per Cent by Vol- ume.
0.8679	71.71	78.40	0.8629	73.83	80.26	0.8579	76.08	82.23
8	71.75	78.44	8	73.88	80.30	8	76.13	82.26
7	71.79	78.47	7	73.92	80.33	7	76.17	82.30
6	71.83	78.51	6	73.96	80.37	6	76.21	82.33
5	71.88	78.55	5	74.00	80.40	5	76.25	82.37
4	71.92	78.58	4	74.05	80.44	4	76.29	82.40
3	71.96	78.62	3	74.09	80.48	3	76.33	82.44
2	72.00	78.66	2	74.14	80.52	2	76.38	82.47
1	72.04	78.70	1	74.18	80.56	1	76.42	82.51
0	72.09	78.73	0	74.23	80.60	0	76.46	82.54
0.8669	72.13	78.77	0.8619	74.27	80.64	0.8569	76.50	82.58
8	72.17	78.81	8	74.32	80.68	8	76.54	82.61
7	72.22	78.85	7	74.36	80.72	7	76.58	82.65
6	72.26	78.89	6	74.41	80.76	6	76.63	82.69
5	72.30	78.93	5	74.45	80.80	5	76.67	82.72
4	72.35	78.96	4	74.50	80.84	4	76.71	82.76
3	72.39	79.00	3	74.55	80.88	3	76.75	82.79
2	72.43	79.04	2	74.59	80.92	2	76.79	82.83
1	72.48	79.08	1	74.64	80.96	1	76.83	82.86
0	72.52	79.12	0	74.68	81.00	0	76.88	82.90
0.8659	72.57	79.16	0.8609	74.73	81.04	0.8559	76.92	82.93
8	72.61	79.19	8	74.77	81.08	8	76.96	82.97
7	72.65	79.23	7	74.82	81.12	7	77.00	83.00
6	72.70	79.27	6	74.86	81.16	6	77.04	83.04
5	72.74	79.31	5	74.91	81.20	5	77.08	83.07
4	72.78	79.35	4	74.95	81.24	4	77.13	83.11
3	72.83	79.39	3	75.00	81.28	3	77.17	83.14
2	72.87	79.42	2	75.05	81.32	2	77.21	83.18
1	72.91	79.46	1	75.09	81.36	1	77.25	83.21
0	72.96	79.50	0	75.14	81.40	0	77.29	83.25
0.8649	73.00	79.54	0.8599	75.18	81.44	0.8549	77.33	83.28
8	73.04	79.57	8	75.23	81.48	8	77.38	83.32
7	73.08	79.61	7	75.27	81.52	7	77.42	83.36
6	73.13	79.65	6	75.33	81.56	6	77.46	83.39
5	73.17	79.68	5	75.36	81.60	5	77.50	83.43
4	73.21	79.72	4	75.41	81.64	4	77.54	83.46
3	73.25	79.75	3	75.45	81.68	3	77.58	83.50
2	73.29	79.79	2	75.50	81.72	2	77.63	83.53
1	73.33	79.83	1	75.55	81.76	1	77.67	83.57
0	73.38	79.86	0	75.59	81.80	0	77.71	83.60
0.8639	73.42	79.90	0.8589	75.64	81.84	0.8539	77.75	83.64
8	73.46	79.94	8	75.68	81.88	8	77.79	83.67
7	73.50	79.97	7	75.73	81.92	7	77.83	83.71
6	73.54	80.01	6	75.77	81.96	6	77.88	83.74
5	73.58	80.04	5	75.82	82.00	5	77.92	83.78
4	73.63	80.08	4	75.86	82.04	4	77.96	83.81
3	73.67	80.12	3	75.91	82.08	3	78.00	83.85
2	73.71	80.15	2	75.95	82.12	2	78.04	83.88
1	73.75	80.19	1	76.00	82.16	1	78.08	83.91
0	73.79	80.22	0	76.04	82.19	0	78.12	83.94



## SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(Continued).

Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.	
	Per Cent by Weight.	Per Cent. by Vol- ume.		Per Cent by Weight.	Per Cent by Vol- ume.		Per Cent by Weight.	Per Cent by Vol- ume.
0.8529	78.16	83.98	0.8479	80.17	85.63	0.8429	82.19	87.27
8	78.20	84.01	8	80.21	85.66	8	82.23	87.30
7	78.24	84.04	7	80.25	85.70	7	82.27	87.34
6	78.28	84.08	6	80.29	85.73	6	82.31	87.37
5	78.32	84.11	5	80.33	85.77	5	82.35	87.40
4	78.36	84.14	4	80.38	85.80	4	82.38	87.43
3	78.40	84.18	3	80.42	85.84	3	82.42	87.46
2	78.44	84.21	2	80.46	85.87	2	82.46	87.49
1	78.48	84.24	1	80.50	85.90	1	82.50	87.52
0	78.52	84.27	0	80.54	85.94	0	82.54	87.55
0.8519	78.56	84.31	0.8469	80.58	85.97	0.8419	82.58	87.58
8	78.60	84.34	8	80.63	86.01	8	82.62	87.61
7	78.64	84.37	7	80.67	86.04	7	82.65	87.64
6	78.68	84.41	6	80.71	86.08	6	82.69	87.67
5	78.72	84.44	5	80.75	86.11	5	82.73	87.70
4	78.76	84.47	4	80.79	86.15	4	82.77	87.73
3	78.80	84.51	3	80.83	86.18	3	82.81	87.76
2	78.84	84.54	2	80.88	86.22	2	82.85	87.79
1	78.88	84.57	1	80.92	86.25	1	82.88	87.82
0	78.92	84.60	0	80.96	86.28	0	82.92	87.85
0.8509	78.96	84.64	0.8459	81.00	86.32	0.8409	82.96	87.88
8	79.00	84.67	8	81.04	86.35	8	83.00	87.91
7	79.04	84.70	7	81.08	86.38	7	83.04	87.94
6	79.08	84.74	6	81.12	86.42	6	83.08	87.97
5	79.12	84.77	5	81.16	86.45	5	83.12	88.00
4	79.16	84.80	4	81.20	86.48	4	83.15	88.03
3	79.20	84.83	3	81.24	86.51	3	83.19	88.06
2	79.24	84.87	2	81.28	86.54	2	83.23	88.09
1	79.28	84.90	1	81.32	86.58	1	83.27	88.13
0	79.32	84.93	0	81.36	86.61	0	83.31	88.16
0.8499	79.36	84.97	0.8449	81.40	86.64	0.8399	83.35	88.19
8	79.40	85.00	8	81.44	86.67	8	83.38	88.22
7	79.44	85.03	7	81.48	86.71	7	83.42	88.25
6	79.48	85.06	6	81.52	86.74	6	83.46	88.28
5	79.52	85.10	5	81.56	86.77	5	83.50	88.31
4	79.56	85.13	4	81.60	86.80	4	83.54	88.34
3	79.60	85.16	3	81.64	86.83	3	83.58	88.37
2	79.64	85.19	2	81.68	86.87	2	83.62	88.40
1	79.68	85.23	1	81.72	86.90	1	83.65	88.43
0	79.72	85.26	0	81.76	86.93	0	83.69	88.46
0.8489	79.76	85.29	0.8439	81.80	86.96	0.8389	83.73	88.49
8	79.80	85.33	8	81.84	86.99	8	83.77	88.52
7	79.84	85.36	7	81.88	87.03	7	83.81	88.55
6	79.88	85.39	6	81.92	87.06	6	83.85	88.58
5	79.92	85.42	5	81.96	87.09	5	83.88	88.61
4	79.96	85.46	4	82.00	87.12	4	83.92	88.64
3	80.00	85.49	3	82.04	87.15	3	83.96	88.67
2	80.04	85.53	2	82.08	87.18	2	84.00	88.70
1	80.08	85.56	1	82.12	87.21	1	84.04	88.73
0	80.13	85.59	0	82.15	87.24	0	84.08	88.76



## SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(Continued).

Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.	
	Per Cent by Weight.	Per Cent by Vol- ume.		Per Cent by Weight.	Per Cent by Vol- ume.		Per Cent by Weight.	Per Cent. by Vol- ume.
0.8379	84.12	88.79	0.8329	86.08	90.32	0.8279	88.00	91.78
8	84.16	88.83	8	86.12	90.35	8	88.04	91.81
7	84.20	88.86	7	86.15	90.38	7	88.08	91.84
6	84.24	88.89	6	86.19	90.40	6	88.12	91.87
5	84.28	88.92	5	86.23	90.43	5	88.16	91.90
4	84.32	88.95	4	86.27	90.46	4	88.20	91.93
3	84.36	88.98	3	86.31	90.49	3	88.24	91.96
2	84.40	89.01	2	86.35	90.52	2	88.28	91.99
1	84.44	89.05	1	86.38	90.55	1	88.32	92.02
0	84.48	89.08	0	86.42	90.58	0	88.36	92.05
0.8369	84.52	89.11	0.8319	86.46	90.61	0.8269	88.40	92.08
8	84.56	89.14	8	86.50	90.64	8	88.44	92.12
7	84.60	89.17	7	86.54	90.67	7	88.48	92.15
6	84.64	89.20	6	86.58	90.70	6	88.52	92.18
5	84.68	89.24	5	86.62	90.73	5	88.56	92.21
4	84.72	89.27	4	86.65	90.76	4	88.60	92.24
3	84.76	89.30	3	86.69	90.79	3	88.64	92.27
2	84.80	89.33	2	86.73	90.82	2	88.68	92.30
1	84.84	89.36	1	86.77	90.85	1	88.72	92.33
0	84.88	89.39	0	86.81	90.88	0	88.76	92.36
0.8359	84.92	89.42	0.8309	86.85	90.90	0.8259	88.80	92.39
8	84.96	89.46	8	86.88	90.93	8	88.84	92.42
7	85.00	89.49	7	86.92	90.96	7	88.88	92.45
6	85.04	89.52	6	86.96	90.99	6	88.92	92.48
5	85.08	89.55	5	87.00	91.02	5	88.96	92.51
4	85.12	89.58	4	87.04	91.05	4	89.00	92.54
3	85.15	89.61	3	87.08	91.08	3	89.04	92.57
2	85.19	89.64	2	87.12	91.11	2	89.08	92.60
1	85.23	89.67	1	87.15	91.14	1	89.12	92.63
0	85.27	89.70	0	87.19	91.17	0	89.16	92.66
0.8349	85.31	89.72	0.8299	87.23	91.20	0.8249	89.19	92.68
8	85.35	89.75	8	87.27	91.23	8	89.23	92.71
7	85.38	89.78	7	87.31	91.25	7	89.27	92.74
6	85.42	89.81	6	87.35	91.28	6	89.31	92.77
5	85.46	89.84	5	87.38	91.31	5	89.35	92.80
4	85.50	89.87	4	87.42	91.34	4	89.38	92.83
3	85.54	89.90	3	87.46	91.37	3	89.42	92.86
2	85.58	89.93	2	87.50	91.40	2	89.46	92.89
1	85.62	89.96	1	87.54	91.43	1	89.50	92.91
0	85.65	89.99	0	87.58	91.46	0	89.54	92.94
0.8339	85.69	90.02	0.8289	87.62	91.49	0.8239	89.58	92.97
8	85.73	90.05	8	87.65	91.52	8	89.62	93.00
7	85.77	90.08	7	87.69	91.55	7	89.65	93.03
6	85.81	90.11	6	87.73	91.57	6	89.69	93.06
5	85.85	90.14	5	87.77	91.60	5	89.73	93.09
4	85.88	90.17	4	87.81	91.63	4	89.77	93.11
3	85.92	90.20	3	87.85	91.66	3	89.81	93.14
2	85.96	90.23	2	87.88	91.69	2	89.85	93.17
1	86.00	90.26	1	87.92	91.72	1	89.88	93.20
0	86.04	90.29	0	87.96	91.75	0	89.92	93.23



## SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(Continued).

Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.	
	Per Cent by Weight.	Per Cent by Vol- ume.		Per Cent by Weight.	Per Cent by Vol- ume.		Per Cent by Weight.	Per Cent by Vol- ume.
0.8229	89.96	93.26	0.8179	91.75	94.53	0.8129	93.59	95.84
8	90.00	93.29	8	91.79	94.56	8	93.63	95.87
7	90.04	93.31	7	91.82	94.59	7	93.67	95.90
6	90.07	93.34	6	91.86	94.61	6	93.70	95.92
5	90.11	93.36	5	91.89	94.64	5	93.74	95.95
4	90.14	93.39	4	91.93	94.66	4	93.78	95.97
3	90.18	93.41	3	91.96	94.69	3	93.81	96.00
2	90.21	93.44	2	92.00	94.71	2	93.85	96.03
1	90.25	93.47	1	92.04	94.74	1	93.89	96.05
0	90.29	93.49	0	92.07	94.76	0	93.92	96.08
0.8219	90.32	93.52	0.8169	92.11	94.79	0.8119	93.96	96.11
8	90.36	93.74	8	92.15	94.82	8	94.00	96.13
7	90.39	93.57	7	92.18	94.84	7	94.03	96.16
6	90.43	93.59	6	92.22	94.87	6	94.07	96.18
5	90.46	93.62	5	92.26	94.90	5	94.10	96.20
4	90.50	93.64	4	92.30	94.92	4	94.14	96.22
3	90.54	93.67	3	92.33	94.95	3	94.17	96.25
2	90.57	93.70	2	92.37	94.98	2	94.21	96.27
1	90.61	93.72	1	92.41	95.00	1	94.24	96.29
0	90.64	93.75	0	92.44	95.03	0	94.28	96.32
0.8209	90.68	93.77	0.8159	92.48	95.06	0.8109	94.31	96.34
8	90.71	93.80	8	92.52	95.08	8	94.34	96.36
7	90.75	93.82	7	92.55	95.11	7	94.38	96.39
6	90.79	93.85	6	92.59	95.13	6	94.41	96.41
5	90.82	93.87	5	92.63	95.16	5	94.45	96.43
4	90.86	93.90	4	92.67	95.19	4	94.48	96.46
3	90.89	93.93	3	92.70	95.21	3	94.52	96.48
2	90.93	93.95	2	92.74	95.24	2	94.55	96.50
1	90.96	93.98	1	92.78	95.27	1	94.59	96.53
0	91.00	94.00	0	92.81	95.29	0	94.62	96.55
0.8199	91.04	94.03	0.8149	92.85	95.32	0.8099	94.65	96.57
8	91.07	94.05	8	92.89	95.35	8	94.69	96.60
7	91.11	94.08	7	92.92	95.37	7	94.73	96.62
6	91.14	94.10	6	92.96	95.40	6	94.76	96.64
5	91.18	94.13	5	93.00	95.42	5	94.80	96.67
4	91.21	94.15	4	93.04	95.45	4	94.83	96.69
3	91.25	94.18	3	93.07	95.48	3	94.86	96.71
2	91.29	94.21	2	93.11	95.50	2	94.90	96.74
1	91.32	94.23	1	93.15	95.53	1	94.93	96.76
0	91.36	94.26	0	93.18	95.55	0	94.97	96.78
0.8189	91.39	94.28	0.8139	93.22	95.58	0.8089	95.00	96.80
8	91.43	94.31	8	93.26	95.61	8	95.04	96.83
7	91.46	94.33	7	93.30	95.63	7	95.07	96.85
6	91.50	94.36	6	93.33	95.66	6	95.11	96.88
5	91.54	94.38	5	93.37	95.69	5	95.14	96.90
4	91.57	94.41	4	93.41	95.71	4	95.18	96.93
3	91.61	94.43	3	93.44	95.74	3	95.21	96.95
2	91.64	94.46	2	93.48	95.76	2	95.25	96.98
1	91.68	94.48	1	93.52	95.79	1	95.29	97.00
0	91.71	94.51	0	93.55	95.82	0	95.32	97.02



## SPECIFIC GRAVITY AND PERCENTAGE OF ALCOHOL—(Continued).

Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.		Spec. Grav. at 15.6° C.	Absolute Alcohol.	
	Per Cent by Weight.	Per Cent by Vol- ume.		Per Cent by Weight.	Per Cent by Vol- ume.		Per Cent by Weight.	Per Cent by Vol- ume.
0.8079	95.36	97.05	0.8029	97.07	98.18	0.7979	98.69	99.18
2	95.39	97.07	8	97.10	98.20	8	98.72	99.20
7	95.43	97.10	7	97.13	98.22	7	98.75	99.22
6	95.46	97.12	6	97.16	98.24	6	98.78	99.24
5	95.50	97.15	5	97.20	98.27	5	98.81	99.26
4	95.54	97.17	4	97.23	98.29	4	98.84	99.27
3	95.57	97.20	3	97.26	98.31	3	98.87	99.29
2	95.61	97.22	2	97.30	98.33	2	98.91	99.31
1	95.64	97.24	1	97.33	98.35	1	98.94	99.33
0	95.68	97.27	0	97.37	98.37	0	98.97	99.35
0.8069	95.71	97.29	0.8019	97.40	98.39	0.7969	99.00	99.37
8	95.75	97.32	8	97.43	98.42	8	99.03	99.39
7	95.79	97.34	7	97.46	98.44	7	99.06	99.41
6	95.82	97.37	6	97.50	98.46	6	99.10	99.43
5	95.86	97.39	5	97.53	98.48	5	99.13	99.45
4	95.89	97.41	4	97.57	98.50	4	99.16	99.47
3	95.93	97.44	3	97.60	98.52	3	99.19	99.49
2	95.96	97.46	2	97.63	98.54	2	99.23	99.51
1	96.00	97.49	1	97.66	98.56	1	99.26	99.53
0	96.03	97.51	0	97.70	98.59	0	99.29	99.55
0.8059	96.07	97.53	0.8009	97.73	98.61	0.7959	99.32	99.57
8	96.10	97.55	8	97.76	98.63	8	99.36	99.59
7	96.13	97.57	7	97.80	98.65	7	99.39	99.61
6	96.16	97.60	6	97.83	98.67	6	99.42	99.63
5	96.20	97.62	5	97.87	98.69	5	99.45	99.65
4	96.23	97.64	4	97.90	98.71	4	99.48	99.67
3	96.26	97.66	3	97.93	98.74	3	99.52	99.69
2	96.30	97.68	2	97.96	98.76	2	99.55	99.71
1	96.33	97.70	1	98.00	98.78	1	99.58	99.73
0	96.37	97.73	0	98.03	98.80	0	99.61	99.75
0.8049	96.40	97.75	0.7999	98.06	98.82	0.7949	99.65	99.77
8	96.43	97.77	8	98.09	98.83	8	99.68	99.80
7	96.46	97.79	7	98.12	98.85	7	99.71	99.82
6	96.50	97.81	6	98.16	98.87	6	99.74	99.84
5	96.53	97.83	5	98.19	98.89	5	99.78	99.86
4	96.57	97.86	4	98.22	98.91	4	99.81	99.88
3	96.60	97.88	3	98.25	98.93	3	99.84	99.90
2	96.63	97.90	2	98.28	98.94	2	99.87	99.92
1	96.66	97.92	1	98.31	98.96	1	99.90	99.94
0	96.70	97.94	0	98.34	98.98	0	99.94	99.96
0.8039	96.73	97.96	0.7989	98.37	99.00	0.7939	99.97	99.98
8	96.76	97.98	8	98.41	99.02		Abs.	Alc.
7	96.80	98.01	7	98.44	99.04	0.7938	100.00	100.00
6	96.83	98.03	6	98.47	99.05			
5	96.87	98.05	5	98.50	99.07			
4	96.90	98.07	4	98.53	99.09			
3	96.93	98.09	3	98.56	99.11			
2	96.96	98.11	2	98.59	99.13			
1	97.00	98.14	1	98.62	99.15			
0	97.03	98.16	0	98.66	99.16			



(4) *Determination of Alcohol by the Ebullioscope or Vaporimeter* is based on the variation in boiling-point of mixtures of alcohol and water, in accordance with the amount of alcohol present. There are various forms of this instrument, one of the simplest and most convenient being that of Salleron, Fig. 113, the apparatus being known in France as an

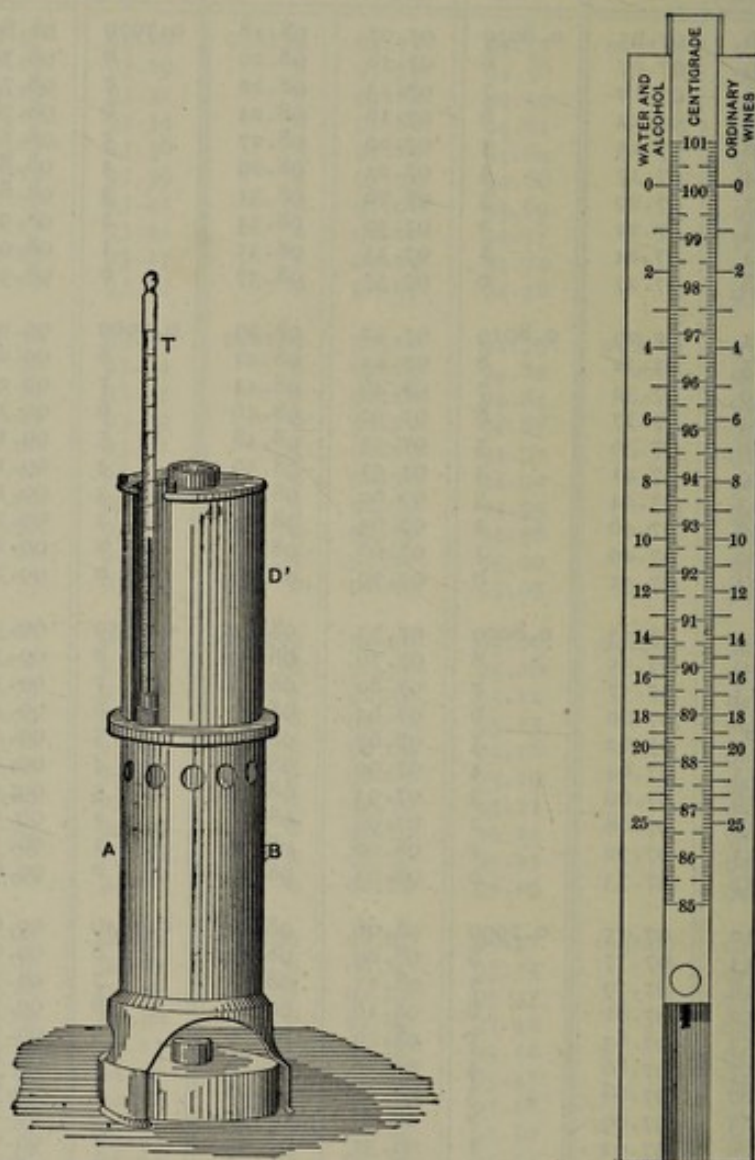


FIG. 113.—Salleron's Ebullioscope and Scale for Calculation of Results.

ebulliometer. This consists of a jacketed metallic reservoir, heated by a lamp placed beneath, and fitted with a return-flow condenser at the top and with a delicate thermometer graduated in tenths of a degree.

As the boiling-point of water varies with the atmospheric pressure, it is necessary to determine the actual boiling-point corresponding with the barometric conditions each time a series of determinations are made.



This is done by boiling a measured portion of distilled water in the reservoir, and carefully noting the temperature when it becomes constant.

The reservoir is then rinsed out with a little of the liquor to be tested, after which a measured amount of this liquor is boiled in the reservoir and the temperature again noted. A sliding scale (Fig. 113) accompanies the instrument, having three graduated parts as shown. The central movable portion is graduated in degrees and tenths of a degree centigrade, the part at the left has the per cent of alcohol corresponding to the temperature in the case of simple mixtures of alcohol and water, while the part at the right is used for reading the per cent in the case of wine, cider, beer, etc., which have a considerable residue. The movable scale bearing the degrees of temperature is first set with the actual temperature of boiling water (as ascertained) opposite the 0 mark on the stationary scale. Suppose the temperature of boiling water has been found to be  $100.1^{\circ}$ . The scale is in this case set as shown in Fig. 113. Suppose also the temperature of boiling of the wine to be tested is found to be  $89.3^{\circ}$ . From the right-hand scale the corresponding per cent of alcohol is found to be 17.2.

When the liquor to be tested contains more than 25% of alcohol, it is necessary to dilute with a measured amount of distilled water and calculate the per cent from the dilution.

When once the boiling-point of water has been determined for a given barometric pressure, it is unnecessary to change the position of the sliding scale during a series of alcohol determinations unless that pressure changes.

*Expression of Results.*—Some confusion is caused by the three ways of expressing results of the alcohol determination, whether as per cent by weight, per cent by volume, or grams per 100 cc. The particular mode adopted should depend upon the nature of the case and upon the prevailing custom. In laboratory analyses, unless otherwise qualified, the simple expression of "per cent" usually implies per cent by weight, and for the reason that this conforms with other determinations, the adoption of the weight-percentage plan is perhaps most natural to the chemist on the grounds of uniformity.

In enforcing the laws regulating the liquor traffic, the custom leans to volume percentage, and many of the laws are based on the "volume of alcohol at  $60^{\circ}$  F." (see page 685).

In recent years many European analysts have adopted the custom of expressing results of analyses of wines and other liquors in grams per



100 cc. and, in order to have a common basis of comparison between the composition of American and of European wines, this manner of expression has to some extent been adopted in the United States.

*Proof-spirit in the United States* is an alcoholic liquor containing 50% of absolute alcohol by volume at 15.6° C. A common method of expressing alcohol is in "degree proof" or simply "proof," which in the United States is twice the per cent of alcohol by volume. Thus, 91.3 proof or degree proof is the same as 45.65% alcohol by volume.

*English Proof-spirit* differs from that in the United States in that it contains 49.28% by weight, or 57.10% by volume of absolute alcohol at 15.6° C. Strength is expressed in degrees over or under proof. Thus liquor 20° under proof has 80 parts by volume of proof-spirit and 20 parts of water at 15.6° C., while 20° over-proof means that 100 volumes of the liquor have to be diluted to 120 volumes with water to yield proof-spirit. To calculate the per cent by volume of English proof-spirit from the per cent of alcohol by volume, divide the latter by 0.5703, or multiply it by 1.7535.

**Direct Determination of Extract.**—In liquors having a high sugar content, the extract or total solids cannot be determined accurately by evaporation at the temperature of boiling water, owing to the dehydration of the reducing sugars at temperatures exceeding 75°. When extreme accuracy is required, such liquors should be dried *in vacuo* at 75°, or in a McGill oven (page 609).

In the case of dry wines having an extract content of less than 3% evaporate 50 cc. in a flat-bottom platinum dish 85 mm. in diameter to a syrup on the water bath and dry for two and one-half hours at the temperature of boiling water. Sweet wines with an extract of from 3 to 6% are treated in the same manner, using, however, only 25 cc. With sweet wines containing over 6% of extract calculate from the specific gravity of the dealcoholized liquor (page 726).

With distilled liquors having low residues accurate results are obtainable by direct evaporation at 100° (page 777).

**Determination of Ash.**—The residue from the determination of the extract is incinerated to a white ash in the original dish at a low red heat, either over a Bunsen flame or in a muffle. The dish is finally cooled in a desiccator and weighed.

**Preservatives and Artificial Sweeteners** in liquors are identified as described in Chapters XVIII and XIX.



## FERMENTED LIQUORS.

The fermented juices of many varieties of fruits and berries furnish beverages more or less popular in various localities, especially for home consumption, though, with the exception of the products of the apple and the grape, few of them are found on the market. The following table shows the average percentage of sugar and free acid in the expressed juice or must of fruits, according to Fresenius, arranged in the order of their sugar content:

	Per Cent Sugar.	Per Cent Free Acid as Malic.
Peaches. ....	1.99	0.85
Apricots. ....	2.13	1.29
Plums. ....	2.80	1.72
Green gages. ....	4.18	0.67
Raspberries. ....	4.84	1.80
Blackberries. ....	5.32	1.42
Strawberries. ....	6.89	1.57
Currants. ....	7.30	2.43
German prunes. ....	7.56	1.08
Gooseberries. ....	8.00	1.63
Pears. ....	8.43	0.09
Apples. ....	9.14	0.82
Mulberries. ....	10.00	2.02
Sour cherries. ....	10.44	1.52
Sweet cherries. ....	15.30	0.88
Grapes. ....	16.15	0.80

## CIDER.

Cider is the expressed juice of the apple. When fresh and before fermentation has set in, it is known as sweet cider, but it does not long remain in this condition, developing after a good fermentation from 3 to 6 per cent of alcohol by volume.

The predominating yeast under the influence of which the fermentation of cider takes place is *Saccharomyces apiculatus*, found in considerable quantity on the outside of the apples as well as in the soil in which the trees grow.

**Process of Manufacture.**—The best cider is made from ripe fruit, taking care to avoid the green and the rotten apples, both of which impair the quality of the product. After gathering, the apples are best allowed to stand in piles until perfectly ripe, being kept under cover. If exposed to the weather, certain of the yeast organisms found on the skins of the apples that are useful in promoting subsequent fermentation would be



washed off. As a rule the apples commonly used by farmers for cider-making are those that are unsalable or unfit for other purposes, being chiefly windfalls or bruised and imperfect fruit. The apples are usually first crushed in a mill to a coarse pulp, which is afterward subjected to pressure in a suitable press and the juice thus extracted.

In this country but little attention is paid to the after processes, the juice being usually transferred directly to barrels, which are not always particularly clean, and allowed to ferment spontaneously in a convenient place, subject to changes in temperature. There is little wonder that cider so made will keep but a short time and quickly goes over into vinegar, unless salicylic acid or other antiseptic is added.

In France more care is taken to regulate the temperature of fermentation, to insure absolute cleanness of all receptacles, and to separate out contaminating impurities. A preliminary fermentation is usually given to the juice in open vats, during which the yeast spores are developed, while impurities separate out both by rising to the surface and by settling to the bottom, care being taken to avoid the development of acetic fermentation. At the proper time the juice is "racked off" or drawn from the clear portion between the top and bottom, transferred to scrupulously clean barrels, and allowed to undergo a second fermentation at a lower temperature than before.

Sometimes the "racking off" is repeated, and the juice is further clarified by "fining" or treating with isinglass, which carries down certain albuminous substances.

Cider thus made is capable of keeping a very long time.

In England cider is sometimes "fined" by treatment with milk, one quart of the latter being added to eighteen gallons of cider.

The apple pomace, left as a residue, is generally steeped in water and repressed. The juice from the second pressing is occasionally added to the first for cider manufacture, but more often is concentrated and made into apple jelly, or used as a fortifier for vinegar to make up deficiency in solids.

**Composition of Cider.**—The following tables, due to Browne,\* show the chemical composition of the freshly expressed juice of several American varieties of apple, as well as that of a few fermented samples of cider of known purity.

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\* Penn. Dept. of Agric., Bul. 58.



## APPLE JUICES.

	Specific Gravity.	Solids.	Invert Sugar.	Sucrose.	Total Sugar.	Total Sugar after Inversion.	Free Malic Acid.	Ash.	Undetermined, Pectin, etc.	Rotation, 400-mm. Tube, Ventzke Scale, Degrees to the Left.
Red astrachan .....	1.05317	11.78	6.87	3.63	10.50	10.60	1.14	0.37	0.77	23.72
Early harvest. ....	1.05522	13.29	7.49	3.97	11.46	11.67	0.90	0.28	0.65	24.32
Yellow transparent. .	1.05020	11.71	8.03	2.10	10.14	10.24	0.86	0.27	0.44	
Early strawberry. ..	1.04949	11.81	5.47	4.21	9.68	9.90	0.78	0.24	1.11	19.24
Sweet bough. ....	1.04979	11.87	7.61	3.08	10.69	10.85	0.10	.....	.....	39.40
Baldwin, green. ....	1.04882	11.36	6.96	1.63	8.59	8.68	1.24	0.31	1.22	36.16
"    ripe. ....	1.07362	16.82	7.97	7.05	15.02	15.39	0.67	0.26	0.87	
Ben Davis. ....	1.05389	12.77	7.11	3.85	10.96	11.16	0.46	0.28	1.07	49.00
Bellflower. ....	1.06270	14.90	9.06	4.32	13.38	13.61	0.58	0.28	0.66	39.20
Tulpahocken. ....	1.05727	13.94	9.68	3.11	12.79	12.95	0.26	0.24	0.49	48.20
Unknown. ....	1.05901	13.75	10.52	2.31	12.83	12.95	0.44	0.26	0.22	44.18

## FERMENTED CIDER (MIXED APPLES).

	Specific Gravity.	Solids.	Invert Sugar.	Malic Acid.	Acetic Acid.	Alcohol.	Pectin.	Ash.	Rotation, 400-mm. Tube, Ventzke Scale, Degrees to the Left.
A...	1.99805	1.94	0.19	0.21	0.24	6.85	0.03	0.25	2.30
B...	1.00122	2.71	0.19	0.24	0.42	5.13	0.03	0.32	2.49
C...	1.00525	3.26	0.89	0.30	0.48	4.67	0.05	0.29	5.28
D. .	1.00071	1.93	0.34	0.27	0.21	4.95	0.05	0.23	2.00
E...	1.00512	2.71	0.24	0.29	1.96	4.26	0.06	0.36	1.76

The following are summaries of the results of a large number of analyses of European apple juices made by Truelle, the quantities being expressed in grams per liter:

	Mean.	Minimum.	Maximum.
Specific gravity. ....	1.0760	1.0573	1.1100
Invert sugar. ....	135.85	108.38	181.81
Sucrose. ....	25.01	5.58	71.7
Total fermentable sugars (as dextrose) ...	162.18	119.22	231.57
Tannin. ....	2.90	0.26	8.07
Pectin and albuminous substances. ....	12	0	23
Acidity (sulphuric acid). ....	2.14	0.69	7.41



In the municipal laboratory of Paris, Sanglé Ferrière has analyzed eleven samples of known-purity cider with the following results:

	Density.	Per Cent Alcohol by Volume.	Extract per Liter.	Sugar per Liter.		Polarization, Laurent.	Ash per Liter.	Alkalinity of Ash, as $K_2CO_3$ per Liter.	Acidity as $H_2SO_4$ .	
				Before Inversion.	After Inversion.				Total.	Fixed.
Mean.....	1.0159	3.9	52.67	21.31	21.62	-4°.26	3.26	2.56	5.27	2.55
Maximum...	1.0410	6.2	114.00	59.40	60.80	-11°.20	4.32	3.68	6.59	2.94
Minimum. . .	1.0012	1.1	22.62	Trace	Trace	0	2.48	2.04	4.20	1.47

Six samples of bottled "sweet" cider purchased in Massachusetts were analyzed in the Food and Drug Laboratory of the Board of Health with the following results:

	Per Cent Alcohol by Weight.	Per Cent Acid as Malic.	Per Cent Extract.
Maximum.....	8.00	0.72	7.82
Minimum.....	3.55	0.48	2.42
Average.....	5.71	0.58	4.19

Browne gives the following as the composition of the mixed ash of several varieties of apple:

Ingredient.	Per-cent-age.		Ingredient.	Per-cent-age.
Potash ( $K_2O$ ).....	55.94	Equivalent to	Potassium carbonate ( $K_2CO_3$ )...	6.85
Soda ( $Na_2O$ ).....	0.31		Potassium phosphate ( $K_3PO_4$ )...	14.55
Lime ( $CaO$ ).....	4.43		Sodium chloride ( $NaCl$ ).....	0.60
Magnesia ( $MgO$ ).....	3.78		Calcium sulphate ( $CaSO_4$ ).....	4.52
Oxide of iron ( $Fe_2O_3$ ).....	0.95		Calcium oxide ( $CaO$ ).....	2.57
Oxide of aluminum ( $Al_2O_3$ ).....	0.80		Magnesium phosphate ( $Mg_3P_2O_8$ ).....	6.97
Chlorine ( $Cl$ ).....	0.39		Magnesium oxide ( $MgO$ ).....	0.59
Silica ( $SiO_2$ ).....	0.40		Ferric oxide ( $Fe_2O_3$ ).....	0.95
Sulphuric acid ( $SO_3$ ).....	2.66		Aluminum oxide ( $Al_2O_3$ ).....	0.80
Phosphoric acid ( $P_2O_5$ ).....	8.64		Silica ( $SiO_2$ ).....	0.40
Carbonic acid ( $CO_2$ ).....	21.60			
	99.90			
Deduct oxygen equivalent to $Cl$ ..	.09			
Total.....	99.81		Total.....	99.80



Burcker \* gives the following composition of the ash of cider:

	Per Cent.
Silica.....	0.94
Phosphoric acid.....	12.68
Lime.....	2.77
Magnesia.....	2.05
Oxides of iron and manganese.....	0.94
Potash.....	53.74
Soda.....	1.10
Carbonic acid.....	25.78
	<hr/>
	100.00

**Adulteration of Cider.**—The Committee on Standards of the A. O. A. C. have submitted for adoption the following standards for cider: Alcohol not more than 8%, extract not less than 1.8% determined by evaporation in an open vessel at ordinary atmospheric pressure and at the temperature of boiling water; ash not less than 0.2%.

Entirely factitious cider made from other than apple stock is rarely found, though the product as sold is frequently of inferior quality and adulterated. The chief adulterants are water and sugar, and the use of antiseptics is common, especially of salicylic and sulphurous acids, sodium benzoate, and occasionally beta-naphthol.

Sodium carbonate is sometimes added to cider to neutralize the acid and thus prevent acetic fermentation. An abnormally high ash (say in excess of 0.35%) would point toward the presence of added alkali.

Watering is apparent when the content of alcohol, solids, and ash of the suspected sample are found to be considerably below the corresponding constants of pure cider. According to Sanglé Ferrière, the following are the minimum figures for these constants in a pure cider, so that a sample may safely be pronounced as watered if they all run distinctly below:

Alcohol.....	3% by volume
Extract.....	1.8%
Ash.....	0.17%

Besides these determinations, it is useful also to determine the fixed and volatile acids.

Caramel is to be looked for, especially in watered samples. Other

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\* Les Falsifications des Substances Alimentaires, p. 176.



adulterants alleged to be of frequent occurrence in French cider, but not commonly found in this country are commercial glucose, tartaric acid (to increase the acidity of a watered product), and coal-tar colors.

Absence or deficiency of malates is conclusive evidence of fraud, indicating the admixture of notable quantities of the juice of the second pressing of pomace.

Sugar is rendered apparent by the right-handed polarization of the sample, pure cider always polarizing well to the left. If after inversion of a dextro-rotary cider the polarization is still to the right, commercial glucose is indicated; if the reading after inversion is to the left, cane sugar has undoubtedly been added.

Frequently the analyst has only to determine the alcohol, especially in cases of seizure, to ascertain whether or not there has been violation of the liquor laws.

#### PERRY OR PEAR CIDER.

This is a common French product, but is rarely if ever found on sale in this country, though sometimes made for home consumption. In composition and in method of manufacture it much resembles apple cider. It is also subject to the same forms of adulteration.

The following table summarizes a number of analyses made by Truelle on pear juice, or must, amounts being expressed in parts per thousand:

	Mean.	Maximum.	Minimum.
Specific gravity. ....	1.0845	1.0675	1.0980
Invert sugar. ....	145.64	108.10	200
Sucrose. ....	36.74	16.69	61.41
Total fermentable sugars (as dextrose)....	184.14	143.78	220
Tannin. ....	1.78	1.01	3.20
Pectin and albuminous substances. ....	13.08	3	18
Acidity (as sulphuric acid) ....	1.47	0.76	2.40

The following analysis of champagne perry is taken from the *Lancet* of October 1, 1892:

Alcohol by weight. ....	1.45
Alcohol by volume. ....	1.80
Solids. ....	11.00
Ash. ....	0.35



## WINE.

Wine in its broadest sense is the fermented expressed juice of any fruit, though the term, unless otherwise restricted, is generally understood to apply to the juice of the grape.

The organism present in grape juice that plays the chief part in its alcoholic fermentation is the *Saccharomyces ellipsoideus*, a yeast which exists on the skins of the grape.

**Process of Manufacture.**—The grapes, which should be fully ripe, are picked and sometimes sorted, according to the care that is taken in grading the product. They are also sometimes freed from the stems, which contain considerable tannic acid, and which when crushed with the grapes impart a certain astringency to the final product. The grapes are crushed either by machinery or by the bare feet, and the juice is pressed out from the pulp in various ways, by screw or hydraulic press, or by the centrifugal process.

A certain amount of juice runs off from the preliminary crushing known as the first run, and makes the choicest wine. The product from the pressure constitutes the second run, after which the pomace, by steeping in water and repressing, is made to yield an inferior juice used in making piquette.

Red wines are made from dark grapes by fermenting the pulp, before pressing, with the skins, which by this treatment yield up their rich color (*ænocyanin*) to the juice. Besides the color, the skins contain also tannin. White wine is made from the pressed pulp, freed from the skins at once, or from the pulp of white grapes. The unfermented must constitutes from 60 to 80 per cent of the weight of the grape.

Fermentation progresses most rapidly at a temperature between 25° and 30° C., but wine having a much finer bouquet is produced by slower fermentation, hence the must is allowed to ferment in open vats or tubs in cool cellars, at a temperature of from 5° to 20° till it settles out comparatively clear, special care being taken to avoid development of acetic fermentation. At the end of the first or active fermentation, the wine is drawn off and allowed to undergo a second or slow fermentation in casks, during which most of the lees or crude argols, composed of potassium bitartrate, settle out, being insoluble in alcohol, and the characteristic bouquet or flavor of the wine is developed. Occasionally during this process the wine is racked or drawn off.

Undesirable fermentations and vegetable fungus growth, which are liable to occur at this time, are avoided by using especially clean casks.



which are frequently "sulphured" (or burnt out with sulphur) before being used. The wine is often clarified, by treatment with gelatin, which mechanically removes many impurities by precipitation, or is subjected to pasteurization before finally being bottled or stored in casks.

**Classification of Wines.**—*Natural wines* are those which are exclusively the product of the simple juice, fermented under the best conditions, either till the sugar has been used up, or till the protein is exhausted, or until the yeast growth has been checked by the strength of the alcohol developed. When the alcohol reaches 18% by volume (in extreme cases 20%) fermentation due to yeast ceases. Examples of natural wines are hock and claret. *Fortified wines* are those to which alcohol has been added. As the addition is commonly made before the fermentation is complete, such wines are usually sweet. Examples of fortified wines are madeira, sherry and port.

*Still wines* are those in which there is but little carbon dioxide remaining, so that they do not effervesce. *Sparkling wines* are more or less heavily charged with carbon dioxide, either naturally, as in the case of champagne, wherein the gas is formed by after fermentation of added sugar in the corked bottle, or artificially, by carbonating.

Wines are also classified according to color. *Red wines* include clarets, chianti and red burgundies, while *white wines* are those of a yellowish color such as the Rhenish and Moselle wines and the sauternes.

"*Dry*" wines are those in which the sugar has been exhausted by fermentation, while *sweet wines* possess a considerable amount of unfermented sugar which remains after the yeast ceases to grow because of the exhaustion of the protein or else the formation or addition of an excess of alcohol. Sweet wines are often reinforced by the addition of sugar.

*Dealcoholized wine* is prepared by distilling off the esters at a low temperature and then the alcohol at a higher temperature, returning the esters to the residue and charging the whole with carbon dioxide and adding a little sugar and in some cases tartaric acid. The product contains a negligible amount of alcohol.

**Varieties of Wine.**—*Champagne* according to French law is the sparkling wine made in the old province of Champagne, although similar wines of other provinces and countries are often incorrectly designated by that name. It is prepared from selected white wine clarified with gelatine, bottled with the addition of cane-sugar and tightly corked. The bottles are placed on their sides and fermentation is allowed to proceed, thus charging the wine with carbon dioxide. The bottles are then



gradually inverted until the sediment gathers above the cork, which by careful manipulation is quickly removed so as to throw out the sediment. A small amount of a liqueur prepared from sugar, wine and brandy is then added, after which the cork is replaced and secured. Champagne contains from 8 to 10% of alcohol and varying amounts of sugar as indicated by the designation *sec* (dry), *extra sec* and *brut* (natural).

*Sauternes* are sweetish white French wines containing 8 to 14% of alcohol by volume and varying amounts of sugar up to 2.5%. Château Yquem is a well-known sauterne.

*Rhine Wines* (Hocks) and *Moselle Wines* are prized because of their delicate mildly tart flavor. They contain little sugar and from 7 to 13% of alcohol. Johannisberger, Steinberger, Hochheimer, Liebfraumilch, Niersteiner and Rüdesheimer are well-known hocks, and Zeltinger and Berncasteler Doctor are examples of moselles.

*Claret* is the common designation for French red wines produced in the neighborhood of Bordeaux. It is somewhat acid and astringent, contains 7 to 13% by volume of alcohol and very little sugar. St. Julien, Pontet Canet, Lafitte, and St. Estèphe are well-known examples.

*Burgundies* are somewhat heavier wines than the clarets and are both red and white, still and sparkling. The best are produced in the Côte d'Or of the old province of Burgundy.

*Chianti* is an Italian wine similar in flavor to the burgundies. It is commonly sold in wicker-covered flasks.

Red and white natural wines are also produced in other European countries as well as in the United States.

*Sherry* is a deep-amber-colored sweet Spanish wine high in alcohol (18-24% by vol.) and consequently fortified. It is commonly plastered.

*Port* is a sweet Portuguese wine, fortified with brandy, containing from 15 to 24% of alcohol by volume. It may be either red or white. Its name is a corruption of Oporto.

*Madeira* is a rich wine, much improved by age, containing from 18 to 20% by vol. of alcohol and a marked quantity of sugar. It is named from the island producing it.

*Tokay Wines* are choice medicinal Hungarian wines high in alcohol and very sweet.

**The Constituents of Wine** may be classified as volatile organic, non-volatile organic, and mineral. The volatile organic constituents, aside from *ethyl alcohol*, consist of higher alcohols, notably *amyl alcohol*, which



go to form the fusel oil of brandy, traces of *methyl alcohol*, volatile acids, chiefly *acetic acid* which is present to some extent in carefully prepared wines and in large amount in wines which have undergone acetic fermentation, also minute quantities of *propionic*, *butyric*, and *higher acids* of the series, as well as very small quantities of *various ethers*, *acetaldehyde*, *furfural*, *acetal*, and other substances influencing the bouquet.

The principal non-volatile organic substances are sugars and related substances, organic acids, *glycerol*, *nitrogenous substances* including traces of *nitrates*, and coloring matter (*ænocyanin* of red wines and *quercetin* of red wine and white and red pomace wines).

The principal saccharine substance is *invert sugar*. Pentoses (chiefly *arabinose*), methylpentoses (chiefly *rhamnose*), *inosite*, and *mannite* occur in small amounts, the last named found only in unsound wines.

The fixed acids derived from the fruit are (1) *tartaric acid*, the most abundant, existing as the free acid and as acid salts, (2) *malic acid*, next to tartaric in abundance, exists largely as the free acid which in the fresh juice exceeds the free tartaric acid but is largely destroyed during fermentation, and (3) *citric acid*, occurring only in small amounts. Traces of *salicylic acid* and probably of *oxalic acid* are also present in grape juice and wine. *Tannic acid* (tannin), the astringent principle of wines, notably the red varieties, may also be grouped with the acids derived from the fruit. The fixed acids formed during fermentation are *lactic* and *succinic*. The acidity as determined by titration does not represent accurately the sourness to the taste. This is shown by the hydrogen ion concentration.

The ash constituents of chief diagnostic importance are *phosphoric acid* (reduced by dilution), *potassium sulphate* (increased by plastering), and *sodium chlorate* (increased by use of salt as a clarifier and preservative). *Manganese* is a normal constituent of wines. Traces of *boric acid* occur in normal wines. *Arsenic* and *copper*, derived from spraying solutions, are present in only infinitesimal quantities.

**Composition of Wines.**—Averages of analyses of typical European wines as compiled by König are given in the table on page 717. A summary of analyses of California wines compiled by Bigelow appear in page 718.

**Standards.**—The U. S. Standards follow: *Wine* is the product made by the normal alcoholic fermentation of the juice of sound, ripe grapes, and the usual cellar treatment, and contains not less than 7 nor more than 16 per cent of alcohol, by volume, and, in 100 cc. (20° C.); not more than 0.1 gram of sodium chloride nor more than 0.2 gram of potassium



## AVERAGE COMPOSITION OF EUROPEAN WINES

	Number Anal- yzed.	Specific Grav- ity.	Grams per 100 cc.										Carbon di- oxide (CO <sub>2</sub> )			
			Alcohol.	Extract.	Total Acids as Tartaric.	Volatile Acids as Acetic.	Tartaric Acid.	Sugar.	Glycerin.	Ash.	Potassium bitar- trate.	Free Tar- taric Acid.		Nitrogen.	Phosphor- ic Acid (P <sub>2</sub> O <sub>5</sub> )	Sulphuric Acid (SO <sub>2</sub> )
RED DRY WINES.																
Bordeaux (Claret).....	44	0.9958	8.16	2.42	0.58	0.10	.....	0.23	0.73	0.25	.....	.....	.....	0.029	0.034	
North Italy.....	70	0.9958	9.25	2.57	0.76	0.11	.....	0.16	0.70	0.25	0.257	0.050	0.015	0.030		
Central Italy (Chianti, etc.)...	35	0.9946	10.29	2.91	0.72	0.12	.....	0.12	0.84	0.26	0.159	0.043	0.021	0.030		
Hungary.....	47	0.9952	9.15	2.62	0.68	.....	.....	.....	0.85	0.22	.....	.....	0.033	0.036	0.026	
Tyrol.....	88	0.9934	8.91	2.16	0.58	0.07	.....	0.15	0.69	0.23	.....	.....	0.035	0.028	0.023	
WHITE DRY WINES.																
Moselle and Saar.....	187	0.9963	7.36	2.31	0.77	0.05	0.34	0.20	0.66	0.16	0.171	0.071	0.061	0.033	0.017	
Rhine and Main.....	68	0.9977	8.12	2.91	0.77	0.05	0.18	0.23	0.85	0.20	0.130	0.017	.....	0.045	0.014	
Lower Rhine.....	17	0.9959	7.99	2.39	0.75	0.04	.....	0.15	0.72	0.15	.....	.....	.....	0.028	0.017	
Rhine-Hesse.....	116	0.9960	7.42	2.15	0.58	0.04	0.19	0.08	0.63	0.22	.....	.....	.....	0.025	0.008	
Pfalz.....	129	0.9946	8.54	2.26	0.64	0.05	0.19	0.13	0.71	0.21	.....	.....	.....	0.032	0.022	
Franconia.....	319	0.9972	7.01	2.17	0.69	0.05	0.21	0.07	0.64	0.19	.....	0.015	.....	0.032	0.022	
Alsace.....	242	0.9961	6.44	1.92	0.64	.....	0.23	0.09	0.53	0.22	0.134	0.026	.....	0.026		
France (Sauterne).....	5	0.9963	9.48	3.03	0.66	0.09	.....	0.84	0.97	0.25	.....	.....	.....	0.032	0.036	
Hungary.....	86	0.9950	8.97	2.45	0.55	.....	.....	0.25	0.94	0.20	.....	.....	0.016	0.048	0.028	
SWEET WINES.																
Spain (Sherry).....	25	0.9932	16.09	4.06	0.4	.....	.....	2.40	0.51	0.46	0.071	.....	.....	0.028	0.186	
Portugal (Port).....	15	1.0088	16.18	8.25	0.42	0.09	.....	6.04	0.34	0.22	.....	.....	.....	0.035	0.023	
Madeira.....	13	0.9996	14.47	5.23	0.49	.....	.....	2.95	0.67	0.25	.....	.....	.....	0.05	0.067	
Tokay, Real.....	51	1.0354	11.19	12.72	0.60	0.10	.....	9.01	1.11	0.27	0.079	.....	.....	0.070	0.015	
Tokay, Commercial.....	57	1.0767	9.93	23.76	0.65	0.16	.....	19.80	0.69	0.35	.....	.....	.....	0.058	0.044	
SPARKLING WINES (CHAMPAGNE, ETC.)																
France and Germany (Dry)...	27	0.9925	10.42	2.36	0.61	.....	0.25	0.53	0.71	0.14	.....	.....	.....	.....	.....	0.857
France and Germany (Sweet) .	32	1.0347	9.50	12.88	0.63	.....	.....	10.92	0.70	0.15	.....	.....	.....	0.022	0.026	0.628



## CALIFORNIA WINES.

	Number of Samples.	Specific Gravity.	Per Cent Alcohol by Volume.	Grams per 100 cc.		Glycerin-Ratio.	Grams per 100 cc.		Polarization.	Grams per 100 cc.					
				Alcohol.	Glycerin.		Total Acids.	Extract.		Reducing Sugar.	Potassium Sulphate.	Proteids.	Tannin and Coloring Matter.	Ash.	
RED WINES.															
Bordeaux, or claret type.....	81														
Maximum.....		1.0020	15.09	11.97	.852	8.7:100	.888	3.81	-2.1	.628	.1570	.5544	.358	.429	
Minimum.....		.9900	8.92	7.09	.330	3.4:100	.368	2.09	-0.5	.040	.0470	.1865	.064	.209	
Rhine type.....	6														
Maximum.....		.9970	13.82	10.96			.718	3.34					.349		
Minimum.....		.9940	11.00	8.73			.358	2.69					.211		
Red Burgundy type.....	25														
Maximum.....		.9962	15.48	12.29	.656	7:100	.762	3.46	-1.7	.418	.2515	.4482	.328	.416	
Minimum.....		.9911	8.00	6.35	.461	4.7:100	.408	2.10	-0.5	.030	.0455	.1864	.033	.188	
Southern French type.....	92														
Maximum.....		1.0050	10.28*	15.30			.834	6.88*					.344	.430	
Minimum.....		.9900	8.07	6.40			.201	1.91					.050	.202	
WHITE WINES.															
Rhine-wine type.....	87														
Maximum.....		1.0020	14.57	11.57	.971	10.2:100	.788	4.38	-3.5	.626	.1771	.5164		.447	
Minimum.....		.9883	5.00	3.98	.474	4.6:100	.327	1.51	-0.2	.060	.0633	.0989		.140	
Sauterne type.....	55														
Maximum.....		1.0157	15.20	12.07	.904	7.7:100	.766	6.78	+0.6	3.569	.1648	.3809	.087	.368	
Minimum.....		.9892	8.23	6.53	.178	1.8:100	.377	1.69	-18.6	.069	.0453	.0867	.015	.050	
Southern French type.....	63														
Maximum.....		.9988	22.18	17.60	.918	14:100	.656	4.56	-3.4	.936		.3162	.045	.290	
Minimum.....		.9882	8.07	6.40	.318	3.4:100	.219	1.09	-0.1	.069		.0988	.034	.148	
Port type.....	50														
Maximum.....		1.0429	22.19	17.61	.707	4.5:100	.700	17.22	-27.1	13.559	.1861	.9379	1.066	.394	
Minimum.....		.9875	10.38	8.24	.163	1.1:100	.181	2.43	-14.4	.228	.0594	.1893	.059	.222	
Sherry and Madeira type.....	66														
Maximum.....		1.0560	21.85	17.34	.936	6.8:100	.798	19.66	-23.1	17.210	.1196	.4751	.350	.436	
Minimum.....		.9866	8.22	6.52	.324	2:100	.253	1.31	-0.5	.115	.0504	.0859	.021	.156	

\* Portified.



sulphate; and for red wine not more than 0.14 gram, and for white wine not more than 0.12 gram of volatile acids produced by fermentation and calculated as acetic acid. *Red wine* is wine containing the red coloring matter of the skins of grape. *White wine* is wine made from white grapes or the expressed fresh juice of other grapes.

*Dry wine* is wine in which the fermentation of the sugars is practically complete, and which contains, in 100 cc. (20° C.), less than 1 gram of sugars, and for dry red wine not less than 0.16 gram of grape ash and not less than 1.6 grams of sugar-free grape solids, and for dry white wine not less than 0.13 gram of grape ash and not less than 1.4 grams of sugar-free grape solids.

*Fortified dry wine* is dry wine to which brandy has been added, but which conforms in all other particulars to the standard of dry wine.

*Sweet wine* is wine in which the alcoholic fermentation has been arrested, and which contains, in 100 cc. (20° C.), not less than 1 gram of sugars, and for sweet red wine not less than 0.16 gram of grape ash, and for sweet white wine not less than 0.13 gram of grape ash.

*Fortified sweet wine* is sweet wine to which wine spirits have been added. By act of Congress, "sweet wine" used for making fortified sweet wine and "wine spirits" used for such fortification are defined as follows (sec. 43, Act. of October 1, 1890, 26 Stat. 567, as amended by section 68, Act of August 27, 1894, 28 Stat. 509, and further amended by Act of Congress, approved June 7, 1906): "That the wine spirits mentioned in section 42 of this act is the product resulting from the distillation of fermented grape juice to which water may have been added, prior to, during, or after fermentation, for the sole purpose of facilitating the fermentation, and economical distillation thereof, and shall be held to include the products from grapes or their residues, commonly known as grape brandy; and the pure sweet wine, which may be fortified free of tax, as provided in said section, is fermented grape juice only, and shall contain no other substance whatever introduced before, at the time of, or after fermentation, except as herein expressly provided; and such sweet wine shall contain not less than 4 per cent of saccharine matter, which saccharine strength may be determined by testing with Balling's saccharometer or must scale, such sweet wine, after the evaporation of the spirits contained therein, and restoring the sample tested to original volume by addition of water: *Provided*, That the addition of pure boiled or condensed grape must, or pure crystallized



cane or beet sugar, or pure anhydrous sugar to the pure grape juice aforesaid, or the fermented product of such grape juice prior to the fortification provided by this act, for the sole purpose of perfecting sweet wine according to commercial standard, or the addition of water in such quantities only as may be necessary in the mechanical operation of grape conveyors, crushers, and pipes leading to fermenting tanks, shall not be excluded by the definition of pure sweet wine aforesaid: *Provided, however,* That the cane or beet sugar, or pure anhydrous sugar, or water, so used shall not in either case be in excess of 10% of the weight of the wine to be fortified under this act: *And provided further,* That the addition of water herein authorized shall be under such regulations and limitations as the Commissioner of Internal Revenue, with the approval of the Secretary of the Treasury, may from time to time prescribe; but in no case shall such wines to which water has been added be eligible for fortification under the provisions of this act where the same, after fermentation and before fortification, have an alcoholic strength of less than 5% of their volume."

*Sparkling wine* is wine in which the after part of the fermentation is completed in the bottle, the sediment being disgorged and its place supplied by wine or sugar liquor, and which contains in 100 cc. (20° C.), not less than 0.12 gram of grape ash.

*Modified wine, ameliorated wine, corrected wine*, is the product made by the alcoholic fermentation, with the usual cellar treatment, of a mixture of the juice of sound, ripe grapes with sugar (sucrose), or a syrup containing not less than 65% of sugar (sucrose), and in quantity not more than enough to raise the alcoholic strength after fermentation, to 11% by volume.

*Raisin wine* is the product made by the alcoholic fermentation of an infusion of dried or evaporated grapes, or of a mixture of such infusion, or of raisins with grape juice.

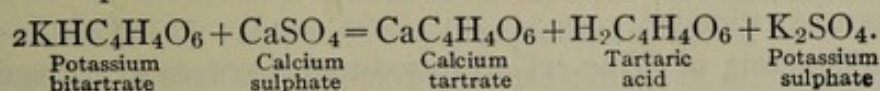
**Adulteration of Wine.**—Beverages purporting to be wine are sometimes found on sale that are entirely spurious, in that they contain little if any fermented grape juice. Brannt gives recipes for the manufacture of such artificial products employing the following ingredients: apple juice, sugar syrup, rectified spirits, crushed raisins, cream of tartar, bilberry, elderberry, and black currant juice, acetic ether, elderberry flowers, and oil of bitter almonds. After fermentation the imitation wines are clarified with isinglass.



If the imitation is largely cider the tartaric acid will be low and the ash will give a potash instead of sodium flame.

Wines are most frequently adulterated by "plastering," by the addition of excessive amounts of sugar or glucose, by watering, by fraudulent fortification with alcohol, by the admixture of raisin wine or imitation wine made from pomace, by the addition of glycerin, by flavoring with ethers, saccharin, etc., by artificial coloring, by the addition of preservatives, and by the addition of citric or tartaric acid.

**Plastering.**—By this term is understood the addition of gypsum or plaster of Paris to the must before fermentation, a practice in vogue in parts of France, Italy, and Spain. The reaction which takes place with the potassium bitartrate present in the wine is, according to Chancel, as follows:



Various advantages are said to result from this practice. The wine is clarified by the precipitation of the calcium tartrate, which mechanically carries down with it many impurities, the color of the wine is improved, since the solubility of the coloring principle present in the skins is increased, the fermentation is rendered more rapid and complete, and the keeping qualities are enhanced. Plastering of dry wines is permitted both by German and French law, provided not more than 2 grams of potassium sulphate per liter remains in the wine. Larger amounts are allowed in sweet wines (sherry, etc.). According to Blarez the limit for new natural wine is 0.6 gram and for old, 1.0 gram.

Deplastering by means of chloride or tartrate of barium or strontium is also practiced.

**Addition of Cane Sugar.**—During some seasons the must is deficient in sugar but contains an excess of acid. To bring the yield of alcohol up to normal the addition of sucrose is permitted in France and of sucrose, invert sugar and commercially pure dextrose in Germany. This practice is known as "chaptalizing." The addition of pure calcium carbonate to correct the acidity is also permitted.

The use of commercial glucose in wine is not regarded with favor, since it contains more or less unfermentable matter, and introduces dextrin and various mineral salts into the wine.

Invert sugar is the only sugar that should be present in natural wine. In normal fermentation the dextrose is more quickly destroyed



than the levulose, hence the polarization of pure wine is always left-handed unless all the sugar has been fermented, in which case the reading should be zero.

Bigelow found in his investigation of California wines that seventy-five samples of red types polarized from  $-0.5$  to  $-2.1$ , upward of eighty of white types from  $-0.1$  to  $-3.5$  (excepting four, evidently abnormal, showing 0 to  $+1$ ) and thirteen of the port type from  $-14.7$  to  $-27.1$ .

A sharp, right-handed polarization would indicate the presence of either commercial glucose, dextrose or cane sugar. After inversion, if the reading is still right-handed, glucose or dextrose is apparent, while if inversion changes the reading from right to left, cane sugar has undoubtedly been added. By application of Clerget's formula the amount of cane sugar can be estimated.

**The Watering of Wine.**—Gall introduced a system of correcting must for an excess of acid as well as a deficiency of sweetness by adding water together with sugar. The German wine law of 1901 permitted "gallizing" if not more than 25% of water was added and the wine did not contain less of the other ingredients than the average of natural wines of the same class. The following minimum limits in grams per 100 cc. were adopted for gallized white and red wines: total extract, white 1.6, red 1.7; total extract minus fixed acids, white 1.1, red 1.3; total extract less total acids, white 1.0, red 1.2; ash, white 0.13, red 0.16.

The law of 1909 permits only 20% of added water and requires that the modified must conform to the natural product of grapes of the same kind and region during good years.

In France watering in any degree is not permitted. Gautier in a large number of analyses of unwatered wines found that the sum of the per cent by volume of alcohol and the total acidity expressed in grams of sulphuric acid per liter varied within very narrow limits, rarely being below 13 or above 17. In applying this rule to plastered or soured wines the following preliminary corrections should be made: (1) if the potassium sulphate exceeds 1 gram per liter (plastered wine) multiply the excess by 0.2 and deduct from the total acids; (2) if the volatile acids exceed 1 (soured wine) multiply the excess by 0.1 and add the product to the alcohol, also add 1 to the fixed acids to obtain the total acids.

This rule according to Pratolongo\* applies not only to all natural Italian wines but also to other wines.

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\* Staz. sper. agr. ital., 50, 1917, p. 315.



Halphen \* adds 0.70 to the fixed acid (expressed in grams of sulphuric acid per liter) and divides by the percentage of alcohol by volume. This ratio is highest in wines containing the lowest percentages of alcohol, as shown by the curves in Halphen's chart (Plate XLI). If the percentage of alcohol corresponding to the ratio found in a given sample is considerably greater than that obtained in the actual analysis, watering is indicated.

Issoglio † concluded that this ratio is applicable to Italian wines, but Scurti and Rolando ‡ found that 25% of water would escape detection, while Pratolongo, on the other hand, found that it would exclude one-fifth of the natural wines.

Roos § lays stress on the ratio of the sum of the fixed acid and percentage of alcohol by volume ( $C$ ) to the quotient obtained by dividing the percentage of alcohol by volume by the percentage of extract ( $B$ ). This ratio  $\left(\frac{C}{B}\right)$  should not be less than 3.2 (or in extreme cases 3.0) for red wines or less than 2.4 for white wines.

Blarez || employs a more complicated scheme of distinguishing natural from watered wines.

Ash ¶ regards California red and white wines as suspicious when they contain less than 2.3 and 1.6% of sugar-free solids and at the same time less than 16 and 15% of alcohol plus acids respectively. The same author concludes that a large amount of free tartaric acid in proportion to potassium bitartrate coupled with low sugar-free solids indicates acidification with citric or tartaric acid as well as dilution.

The presence of nitrates in wines has been regarded as evidence of watering, but Tillman,\*\* and also Paris and Marsaglia,†† find this test valueless, as nitrates occur in natural wines.

**The Fortification of Wine.**—The addition of alcohol to sweet wine such as sherry and port is recognized as an essential step in the process of manufacture, and in Germany the use of 1% by vol. in dry wines is also

\* Ann. chim. anal., 12, 1907, pp. 129, 196.

† Ind. chim., 14, p. 23.

‡ Ann. chim. appl., 8, 1917, p. 47.

§ Ann. fals. 4, 1911, p. 361.

|| Vin et Spiritueux, etc., Paris, 1908.

¶ 8th Int. Cong. App. Chem., 18, 1912, p. 17.

\*\* Zeits. Unters. Nahr. Genussm., 22, 1911, p. 201.

†† Staz. sperim. agrar. ital., 11, 1908, p. 123.



allowed. When, however, alcohol is added to imitation or stretched wines such an addition is distinctly an adulteration.

A committee appointed in France to devise means of detecting added alcohol established the rule that the grams of alcohol per 100 cc. divided by the grams of extract should not exceed 4.5 for red wines or 6.5 for white wines. In the case of plastered wines, or wines having added sugar, it is necessary to deduct from the total extract the weight of the reducing sugar and of the potassium sulphate (less 1 gram for each of these substances), the reduced extract thus obtained being used in calculating the ratio.

In Germany the glycerol-alcohol ratio expressed in grams per 100 cc. is also used in detecting added alcohol, the accepted limits being 7 : 100 and 14 : 100. Both limits, however, should be extended, as German wines often have a ratio as high as 6 : 100 and American wines still higher, and 19 : 100 is on record for genuine European wines.

**"Pomace Wine."**—This term is applied to imitation wines prepared from the marc of grapes with the addition of sugar, water, and often tartaric or citric acid. It is not strictly a wine, and that term according to U. S. rulings is regarded as a misbranding even if modified by the word pomace. The product although not lacking in alcohol is naturally deficient in other characteristic constituents of true wine except, perchance, these are reinforced by skillful sophistication.

Eoff\* has made exhaustive analyses of pomace wines from which he concludes that its presence in wine is usually indicated if the results per 100 cc. are below the following: Nitrogen 10 mg. (below 5 mg. almost certain proof), total tartaric acid 20 cg. (unfortified wine 10 cg.), fixed tartaric acid 50 cg., non-sugar extract 1.5 grams for white and 2.0 grams for red wines, and pentosans 50 mg. for white and 100 mg. for red wines. Pomace wine is also indicated if the ash exceeds 20 cg. (white dry wines), if the alkalinity of the water soluble ash is below 8 cc. N/10 hydrochloric acid per 100 cc., or if the amount of phosphoric acid in the ash is below 10%. Natural wines seldom contain over 5 mg. of chlorine in 100 cc.; if it exceeds 10 mg. the presence of ammonium chloride or corn sugar solution may be suspected.

**Piquette** is prepared in France from second pressings obtained after soaking the marc in water. It is in a sense a diluted wine.

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\* Jour. Ind. Eng. Chem., 8, 1916, p. 723.



**Raisin Wine** is defined on page 720. Its detection by chemical analysis is often more difficult than by organoleptic test.

**The Addition of Glycerol** to increase the extract, known as scheelizing, is indicated by a high glycerol-alcohol ratio. The German commission on wine statistics decided that the presence of over 0.5 gram of glycerol per 100 cc. is proof of addition of this substance provided: (1) the extract minus fixed acids is more than two-thirds glycerol or (2) with a glycerol-alcohol ratio of more than 10 : 100 the total extract is less than 1.8 grams per 100 cc. or the total extract minus the glycerol is less than 1 gram.

**The Coloring of Wine** involves the use of both vegetable and coal-tar dyes and is considered on pages 736-737.

**The Addition of Preservatives** other than sulphur compounds is prohibited in most countries and the amount of sulphur dioxide is limited. Sauternes are commonly sulphured.

**Hexamethylenetetraamine** (urotropin) is used for concealing the presence of sulphur dioxide used as a preservative. On distillation with acid the wine thus treated yields a distillate containing formaldehyde.

**Alum** is sometimes employed to clarify and to improve the color and keeping qualities of wine. French and German laws prohibit its use.

**Common Salt** also serves as a clarifier and preservative. While normal wines contain only traces of chlorine, under certain conditions the quantity is considerable, hence the following rather generous limits expressed in grams per 100 cc.: Germany 0.05, France 0.10, Spain 0.20.

**Fruit Wines other than Grape.**—Wines mostly of domestic manufacture are sometimes made from small fruits, such as raspberries, strawberries, blackberries, gooseberries, elderberries, and currants, as well as from cherries, plums, and apricots. Wines made from most of these fruits readily undergo acetic fermentation unless antiseptics are added, or unless extreme care is taken in their manufacture and keeping. Most of the sour fruits require a liberal admixture of sugar to produce an acceptable wine.

The following analysis of currant wine is due to Fresenius:

Alcohol.....	10.01%
Free acid.....	0.79%
Sugar .....	11.94%
Water. ....	77.26%



## METHODS OF ANALYSIS OF WINE AND CIDER.

For determination of specific gravity, alcohol, extract (by direct method), and ash, see pages 686-706.

**Calculation of the Extract in Wine.**—Attention has already been called to the difficulty in accurately determining the extract of sweet wines gravimetrically by evaporation. An approximate determination of the extract may be obtained by calculation from the specific gravity of the dealcoholized liquor, or one may use for this purpose the tables compiled by Windisch, and based on experiments made on drying wine *in vacuo* at 75° C. In wines high in sugar, with more than 6% of extract, this method is far more accurate than drying at 100°, and is to be recommended.

Evaporate a measured portion of the wine on the water-bath to one-fourth its volume, and dilute with water to exactly the volume measured. Determine the specific gravity of this dealcoholized liquid at 15.6°, and from the table on pages 727-729 ascertain the extract corresponding.

**Determination of Total Acidity.**—Carbonated beverages are first freed from carbon dioxide by agitation as described on page 687, after which 25 cc. of the sample are heated just to the boiling-point and titrated with tenth-normal sodium hydroxide, using in the case of white wine neutral litmus solution as an indicator. With red wine delicate litmus paper should be used. Total acidity is usually expressed, in the case of cider as malic, and of wine as tartaric acid. Each cubic centimeter of tenth-normal alkali corresponds to 0.0067 gram malic, or 0.0075 gram tartaric acid. Some chemists express total acidity in terms of sulphuric acid, each cubic centimeter of tenth-normal alkali being equivalent to 0.0049 gram of sulphuric acid.

**Volatile Acids** in all liquors are usually expressed as acetic, although traces of propionic and other volatile acids may be present. 50 cc. of the cider or wine and a little tannic acid are transferred to a distilling-flask, Fig. 115, the stopper of which is provided with two tubes, one of which connects with the condenser, while the other, arranged to reach nearly to the bottom of the distilling-flask, communicates with a second flask which contains about 300 cc. of water. The contents of both flasks are brought to boiling, after which the flame under the distilling flask is lowered, and steam from the water-flask is passed through the wine till about 200 cc. of distillate have collected in the receiving-flask. Titrate this with tenth-normal sodium hydroxide, using phenolphthalein as an indicator. Each cubic centimeter of tenth-normal alkali is equivalent to 0.006 gram acetic acid.



EXTRACT IN WINE.  
[ACCORDING TO WINDISCH.]

Specific Gravity.	Ex-tract.	Specific Gravity.	Ex-tract.	Specific Gravity.	Ex-tract.	Specific Gravity.	Ex-tract.	Specific Gravity.	Ex-tract.	Specific Gravity.	Ex-tract.
1.0000	0.00	1.0065	1.68	1.0130	3.36	1.0195	5.04	1.0260	6.72	1.0325	8.40
1.0001	0.03	1.0066	1.70	1.0131	3.38	1.0196	5.06	1.0261	6.75	1.0326	8.43
1.0002	0.05	1.0067	1.73	1.0132	3.41	1.0197	5.09	1.0262	6.77	1.0327	8.46
1.0003	0.08	1.0068	1.76	1.0133	3.43	1.0198	5.11	1.0263	6.80	1.0328	8.48
1.0004	0.10	1.0069	1.78	1.0134	3.46	1.0199	5.14	1.0264	6.82	1.0329	8.51
1.0005	0.13	1.0070	1.81	1.0135	3.49	1.0200	5.17	1.0265	6.85	1.0330	8.53
1.0006	0.15	1.0071	1.83	1.0136	3.51	1.0201	5.19	1.0266	6.88	1.0331	8.56
1.0007	0.18	1.0072	1.86	1.0137	3.54	1.0202	5.22	1.0267	6.90	1.0332	8.59
1.0008	0.20	1.0073	1.88	1.0138	3.56	1.0203	5.25	1.0268	6.93	1.0333	8.61
1.0009	0.23	1.0074	1.91	1.0139	3.59	1.0204	5.27	1.0269	6.95	1.0334	8.64
1.0010	0.26	1.0075	1.94	1.0140	3.62	1.0205	5.30	1.0270	6.98	1.0335	8.66
1.0011	0.28	1.0076	1.96	1.0141	3.64	1.0206	5.32	1.0271	7.01	1.0336	8.69
1.0012	0.31	1.0077	1.99	1.0142	3.67	1.0207	5.35	1.0272	7.03	1.0337	8.72
1.0013	0.34	1.0078	2.01	1.0143	3.69	1.0208	5.38	1.0273	7.06	1.0338	8.74
1.0014	0.36	1.0079	2.04	1.0144	3.72	1.0209	5.40	1.0274	7.08	1.0339	8.77
1.0015	0.39	1.0080	2.07	1.0145	3.75	1.0210	5.43	1.0275	7.11	1.0340	8.79
1.0016	0.41	1.0081	2.09	1.0146	3.77	1.0211	5.45	1.0276	7.13	1.0341	8.82
1.0017	0.44	1.0082	2.12	1.0147	3.80	1.0212	5.48	1.0277	7.16	1.0342	8.85
1.0018	0.46	1.0083	2.14	1.0148	3.82	1.0213	5.51	1.0278	7.19	1.0343	8.87
1.0019	0.49	1.0084	2.17	1.0149	3.85	1.0214	5.53	1.0279	7.21	1.0344	8.90
1.0020	0.52	1.0085	2.19	1.0150	3.87	1.0215	5.56	1.0280	7.24	1.0345	8.92
1.0021	0.54	1.0086	2.22	1.0151	3.90	1.0216	5.58	1.0281	7.26	1.0346	8.95
1.0022	0.57	1.0087	2.25	1.0152	3.93	1.0217	5.61	1.0282	7.29	1.0347	8.97
1.0023	0.59	1.0088	2.27	1.0153	3.95	1.0218	5.64	1.0283	7.32	1.0348	9.00
1.0024	0.62	1.0089	2.30	1.0154	3.98	1.0219	5.66	1.0284	7.34	1.0349	9.03
1.0025	0.64	1.0090	2.32	1.0155	4.00	1.0220	5.69	1.0285	7.37	1.0350	9.05
1.0026	0.67	1.0091	2.35	1.0156	4.03	1.0221	5.71	1.0286	7.39	1.0351	9.08
1.0027	0.69	1.0092	2.38	1.0157	4.06	1.0222	5.74	1.0287	7.42	1.0352	9.10
1.0028	0.72	1.0093	2.40	1.0158	4.08	1.0223	5.77	1.0288	7.45	1.0353	9.13
1.0029	0.75	1.0094	2.43	1.0159	4.11	1.0224	5.79	1.0289	7.47	1.0354	9.16
1.0030	0.77	1.0095	2.45	1.0160	4.13	1.0225	5.82	1.0290	7.50	1.0355	9.18
1.0031	0.80	1.0096	2.48	1.0161	4.16	1.0226	5.84	1.0291	7.52	1.0356	9.21
1.0032	0.82	1.0097	2.50	1.0162	4.19	1.0227	5.87	1.0292	7.55	1.0357	9.23
1.0033	0.85	1.0098	2.53	1.0163	4.21	1.0228	5.89	1.0293	7.58	1.0358	9.26
1.0034	0.87	1.0099	2.56	1.0164	4.24	1.0229	5.92	1.0294	7.60	1.0359	9.29
1.0035	0.90	1.0100	2.58	1.0165	4.26	1.0230	5.94	1.0295	7.63	1.0360	9.31
1.0036	0.93	1.0101	2.61	1.0166	4.29	1.0231	5.97	1.0296	7.65	1.0361	9.34
1.0037	0.95	1.0102	2.63	1.0167	4.31	1.0232	6.00	1.0297	7.68	1.0362	9.36
1.0038	0.98	1.0103	2.66	1.0168	4.34	1.0233	6.02	1.0298	7.70	1.0363	9.39
1.0039	1.00	1.0104	2.69	1.0169	4.37	1.0234	6.05	1.0299	7.73	1.0364	9.42
1.0040	1.03	1.0105	2.71	1.0170	4.39	1.0235	6.07	1.0300	7.76	1.0365	9.44
1.0041	1.05	1.0106	2.74	1.0171	4.42	1.0236	6.10	1.0301	7.78	1.0366	9.47
1.0042	1.08	1.0107	2.76	1.0172	4.44	1.0237	6.12	1.0302	7.81	1.0367	9.49
1.0043	1.11	1.0108	2.79	1.0173	4.47	1.0238	6.15	1.0303	7.83	1.0368	9.52
1.0044	1.13	1.0109	2.82	1.0174	4.50	1.0239	6.18	1.0304	7.86	1.0369	9.55
1.0045	1.16	1.0110	2.84	1.0175	4.52	1.0240	6.20	1.0305	7.89	1.0370	9.57
1.0046	1.18	1.0111	2.87	1.0176	4.55	1.0241	6.23	1.0306	7.91	1.0371	9.60
1.0047	1.21	1.0112	2.89	1.0177	4.57	1.0242	6.25	1.0307	7.94	1.0372	9.62
1.0048	1.24	1.0113	2.92	1.0178	4.60	1.0243	6.28	1.0308	7.97	1.0373	9.65
1.0049	1.26	1.0114	2.94	1.0179	4.63	1.0244	6.31	1.0309	7.99	1.0374	9.68
1.0050	1.29	1.0115	2.97	1.0180	4.65	1.0245	6.33	1.0310	8.02	1.0375	9.70
1.0051	1.32	1.0116	3.00	1.0181	4.68	1.0246	6.36	1.0311	8.04	1.0376	9.73
1.0052	1.34	1.0117	3.02	1.0182	4.70	1.0247	6.38	1.0312	8.07	1.0377	9.75
1.0053	1.37	1.0118	3.05	1.0183	4.73	1.0248	6.41	1.0313	8.09	1.0378	9.78
1.0054	1.39	1.0119	3.07	1.0184	4.75	1.0249	6.44	1.0314	8.12	1.0379	9.80
1.0055	1.42	1.0120	3.10	1.0185	4.78	1.0250	6.46	1.0315	8.14	1.0380	9.83
1.0056	1.45	1.0121	3.12	1.0186	4.81	1.0251	6.49	1.0316	8.17	1.0381	9.86
1.0057	1.47	1.0122	3.15	1.0187	4.83	1.0252	6.51	1.0317	8.20	1.0382	9.88
1.0058	1.50	1.0123	3.18	1.0188	4.86	1.0253	6.54	1.0318	8.22	1.0383	9.91
1.0059	1.52	1.0124	3.20	1.0189	4.88	1.0254	6.56	1.0319	8.25	1.0384	9.93
1.0060	1.55	1.0125	3.23	1.0190	4.91	1.0255	6.59	1.0320	8.27	1.0385	9.96
1.0061	1.57	1.0126	3.26	1.0191	4.94	1.0256	6.62	1.0321	8.30	1.0386	9.99
1.0062	1.60	1.0127	3.28	1.0192	4.96	1.0257	6.64	1.0322	8.33	1.0387	10.01
1.0063	1.63	1.0128	3.31	1.0193	4.99	1.0258	6.67	1.0323	8.35	1.0388	10.04
1.0064	1.65	1.0129	3.33	1.0194	5.01	1.0259	6.70	1.0324	8.38	1.0389	10.06



## EXTRACT IN WINE—(Continued).

Specific Gravity.	Ex-tract.	Specific Gravity.	Ex-tract.	Specific Gravity.	Ex-tract.	Specific Gravity.	Ex-tract.	Specific Gravity.	Ex-tract.	Specific Gravity.	Ex-tract.
1.0390	10.09	1.0455	11.78	1.0520	13.47	1.0585	15.16	1.0650	16.86	1.0715	18.56
1.0391	10.11	1.0456	11.81	1.0521	13.49	1.0586	15.19	1.0651	16.88	1.0716	18.58
1.0392	10.14	1.0457	11.83	1.0522	13.52	1.0587	15.22	1.0652	16.91	1.0717	18.61
1.0393	10.17	1.0458	11.86	1.0523	13.55	1.0588	15.24	1.0653	16.94	1.0718	18.63
1.0394	10.19	1.0459	11.88	1.0524	13.57	1.0589	15.27	1.0654	16.96	1.0719	18.66
1.0395	10.22	1.0460	11.91	1.0525	13.60	1.0590	15.29	1.0655	16.99	1.0720	18.69
1.0396	10.25	1.0461	11.94	1.0526	13.62	1.0591	15.32	1.0656	17.01	1.0721	18.71
1.0397	10.27	1.0462	11.96	1.0527	13.65	1.0592	15.35	1.0657	17.04	1.0722	18.74
1.0398	10.30	1.0463	11.99	1.0528	13.68	1.0593	15.37	1.0658	17.07	1.0723	18.76
1.0399	10.32	1.0464	12.01	1.0529	13.70	1.0594	15.40	1.0659	17.09	1.0724	18.79
1.0400	10.35	1.0465	12.04	1.0530	13.73	1.0595	15.42	1.0660	17.12	1.0725	18.82
1.0401	10.37	1.0466	12.06	1.0531	13.75	1.0596	15.45	1.0661	17.14	1.0726	18.84
1.0402	10.40	1.0467	12.09	1.0532	13.78	1.0597	15.48	1.0662	17.17	1.0727	18.87
1.0403	10.43	1.0468	12.12	1.0533	13.81	1.0598	15.50	1.0663	17.20	1.0728	18.90
1.0404	10.45	1.0469	12.14	1.0534	13.83	1.0599	15.53	1.0664	17.22	1.0729	18.92
1.0405	10.48	1.0470	12.17	1.0535	13.86	1.0600	15.55	1.0665	17.25	1.0730	18.95
1.0406	10.51	1.0471	12.19	1.0536	13.89	1.0601	15.58	1.0666	17.27	1.0731	18.97
1.0407	10.53	1.0472	12.22	1.0537	13.91	1.0602	15.61	1.0667	17.30	1.0732	19.00
1.0408	10.56	1.0473	12.25	1.0538	13.94	1.0603	15.63	1.0668	17.33	1.0733	19.03
1.0409	10.58	1.0474	12.27	1.0539	13.96	1.0604	15.66	1.0669	17.35	1.0734	19.05
1.0410	10.61	1.0475	12.30	1.0540	13.99	1.0605	15.68	1.0670	17.38	1.0735	19.08
1.0411	10.63	1.0476	12.32	1.0541	14.01	1.0606	15.71	1.0671	17.41	1.0736	19.10
1.0412	10.66	1.0477	12.35	1.0542	14.04	1.0607	15.74	1.0672	17.43	1.0737	19.13
1.0413	10.69	1.0478	12.38	1.0543	14.07	1.0608	15.76	1.0673	17.46	1.0738	19.16
1.0414	10.71	1.0479	12.40	1.0544	14.09	1.0609	15.79	1.0674	17.48	1.0739	19.18
1.0415	10.74	1.0480	12.43	1.0545	14.12	1.0610	15.81	1.0675	17.51	1.0740	19.21
1.0416	10.76	1.0481	12.45	1.0546	14.14	1.0611	15.84	1.0676	17.54	1.0741	19.23
1.0417	10.79	1.0482	12.48	1.0547	14.17	1.0612	15.87	1.0677	17.56	1.0742	19.26
1.0418	10.82	1.0483	12.51	1.0548	14.20	1.0613	15.89	1.0678	17.59	1.0743	19.29
1.0419	10.84	1.0484	12.53	1.0549	14.22	1.0614	15.92	1.0679	17.62	1.0744	19.31
1.0420	10.87	1.0485	12.56	1.0550	14.25	1.0615	15.94	1.0680	17.64	1.0745	19.34
1.0421	10.90	1.0486	12.58	1.0551	14.28	1.0616	15.97	1.0681	17.67	1.0746	19.37
1.0422	10.92	1.0487	12.61	1.0552	14.30	1.0617	16.00	1.0682	17.69	1.0747	19.39
1.0423	10.95	1.0488	12.64	1.0553	14.33	1.0618	16.02	1.0683	17.72	1.0748	19.42
1.0424	10.97	1.0489	12.66	1.0554	14.35	1.0619	16.05	1.0684	17.75	1.0749	19.44
1.0425	11.00	1.0490	12.69	1.0555	14.38	1.0620	16.07	1.0685	17.77	1.0750	19.47
1.0426	11.03	1.0491	12.71	1.0556	14.41	1.0621	16.10	1.0686	17.80	1.0751	19.50
1.0427	11.05	1.0492	12.74	1.0557	14.43	1.0622	16.13	1.0687	17.83	1.0752	19.52
1.0428	11.08	1.0493	12.77	1.0558	14.46	1.0623	16.15	1.0688	17.85	1.0753	19.55
1.0429	11.10	1.0494	12.79	1.0559	14.48	1.0624	16.18	1.0689	17.88	1.0754	19.58
1.0430	11.13	1.0495	12.82	1.0560	14.51	1.0625	16.21	1.0690	17.90	1.0755	19.60
1.0431	11.15	1.0496	12.84	1.0561	14.54	1.0626	16.23	1.0691	17.93	1.0756	19.63
1.0432	11.18	1.0497	12.87	1.0562	14.56	1.0627	16.26	1.0692	17.95	1.0757	19.65
1.0433	11.21	1.0498	12.90	1.0563	14.59	1.0628	16.28	1.0693	17.98	1.0758	19.68
1.0434	11.23	1.0499	12.92	1.0564	14.61	1.0629	16.31	1.0694	18.01	1.0759	19.71
1.0435	11.26	1.0500	12.95	1.0565	14.64	1.0630	16.33	1.0695	18.03	1.0760	19.73
1.0436	11.28	1.0501	12.97	1.0566	14.67	1.0631	16.36	1.0696	18.06	1.0761	19.76
1.0437	11.31	1.0502	13.00	1.0567	14.69	1.0632	16.39	1.0697	18.08	1.0762	19.79
1.0438	11.34	1.0503	13.03	1.0568	14.72	1.0633	16.41	1.0698	18.11	1.0763	19.81
1.0439	11.37	1.0504	13.05	1.0569	14.74	1.0634	16.44	1.0699	18.14	1.0764	19.84
1.0440	11.39	1.0505	13.08	1.0570	14.77	1.0635	16.47	1.0700	18.16	1.0765	19.86
1.0441	11.42	1.0506	13.10	1.0571	14.80	1.0636	16.49	1.0701	18.19	1.0766	19.89
1.0442	11.44	1.0507	13.13	1.0572	14.82	1.0637	16.52	1.0702	18.22	1.0767	19.92
1.0443	11.47	1.0508	13.16	1.0573	14.85	1.0638	16.54	1.0703	18.24	1.0768	19.94
1.0444	11.49	1.0509	13.18	1.0574	14.87	1.0639	16.57	1.0704	18.27	1.0769	19.97
1.0445	11.52	1.0510	13.21	1.0575	14.90	1.0640	16.60	1.0705	18.30	1.0770	20.00
1.0446	11.55	1.0511	13.23	1.0576	14.93	1.0641	16.62	1.0706	18.32	1.0771	20.02
1.0447	11.57	1.0512	13.26	1.0577	14.95	1.0642	16.65	1.0707	18.35	1.0772	20.05
1.0448	11.60	1.0513	13.29	1.0578	14.98	1.0643	16.68	1.0708	18.37	1.0773	20.07
1.0449	11.62	1.0514	13.31	1.0579	15.00	1.0644	16.70	1.0709	18.40	1.0774	20.10
1.0450	11.65	1.0515	13.34	1.0580	15.03	1.0645	16.73	1.0710	18.43	1.0775	20.12
1.0451	11.68	1.0516	13.36	1.0581	15.06	1.0646	16.75	1.0711	18.45	1.0776	20.15
1.0452	11.70	1.0517	13.39	1.0582	15.08	1.0647	16.78	1.0712	18.48	1.0777	20.18
1.0453	11.73	1.0518	13.42	1.0583	15.11	1.0648	16.80	1.0713	18.50	1.0778	20.20
1.0454	11.75	1.0519	13.44	1.0584	15.14	1.0649	16.83	1.0714	18.53	1.0779	20.23



## EXTRACT IN WINE—(Continued).

Specific Gravity.	Ex-tract.	Specific Gravity.	Ex-tract.	Specific Gravity.	Ex-tract.	Specific Gravity.	Ex-tract.	Specific Gravity.	Ex-tract.	Specific Gravity.	Ex-tract.
1.0780	20.26	1.0845	21.96	1.0910	23.67	1.0975	25.38	1.1040	27.09	1.1105	28.81
1.0781	20.28	1.0846	21.99	1.0911	23.70	1.0976	25.41	1.1041	27.12	1.1106	28.83
1.0782	20.31	1.0847	22.02	1.0912	23.72	1.0977	25.43	1.1042	27.15	1.1107	28.86
1.0783	20.34	1.0848	22.04	1.0913	23.75	1.0978	25.46	1.1043	27.17	1.1108	28.88
1.0784	20.36	1.0849	22.07	1.0914	23.77	1.0979	25.49	1.1044	27.20	1.1109	28.91
1.0785	20.39	1.0850	22.09	1.0915	23.80	1.0980	25.51	1.1045	27.22	1.1110	28.94
1.0786	20.41	1.0851	22.12	1.0916	23.83	1.0981	25.54	1.1046	27.25	1.1111	28.96
1.0787	20.44	1.0852	22.15	1.0917	23.85	1.0982	25.56	1.1047	27.27	1.1112	28.99
1.0788	20.47	1.0853	22.17	1.0918	23.88	1.0983	25.59	1.1048	27.30	1.1113	29.02
1.0789	20.49	1.0854	22.20	1.0919	23.91	1.0984	25.62	1.1049	27.33	1.1114	29.04
1.0790	20.52	1.0855	22.22	1.0920	23.93	1.0985	25.64	1.1050	27.35	1.1115	29.07
1.0791	20.55	1.0856	22.25	1.0921	23.96	1.0986	25.67	1.1051	27.38	1.1116	29.09
1.0792	20.57	1.0857	22.28	1.0922	23.99	1.0987	25.70	1.1052	27.41	1.1117	29.12
1.0793	20.60	1.0858	22.30	1.0923	24.01	1.0988	25.72	1.1053	27.43	1.1118	29.15
1.0794	20.62	1.0859	22.33	1.0924	24.04	1.0989	25.75	1.1054	27.46	1.1119	29.17
1.0795	20.65	1.0860	22.36	1.0925	24.07	1.0990	25.78	1.1055	27.49	1.1120	29.20
1.0796	20.68	1.0861	22.38	1.0926	24.09	1.0991	25.80	1.1056	27.51	1.1121	29.23
1.0797	20.70	1.0862	22.41	1.0927	24.12	1.0992	25.83	1.1057	27.54	1.1122	29.25
1.0798	20.73	1.0863	22.43	1.0928	24.14	1.0993	25.85	1.1058	27.57	1.1123	29.28
1.0799	20.75	1.0864	22.46	1.0929	24.17	1.0994	25.88	1.1059	27.59	1.1124	29.31
1.0800	20.78	1.0865	22.49	1.0930	24.20	1.0995	25.91	1.1060	27.62	1.1125	29.33
1.0801	20.81	1.0866	22.51	1.0931	24.22	1.0996	25.93	1.1061	27.65	1.1126	29.36
1.0802	20.83	1.0867	22.54	1.0932	24.25	1.0997	25.96	1.1062	27.67	1.1127	29.39
1.0803	20.86	1.0868	22.57	1.0933	24.27	1.0998	25.99	1.1063	27.70	1.1128	29.41
1.0804	20.89	1.0869	22.59	1.0934	24.30	1.0999	26.01	1.1064	27.72	1.1129	29.44
1.0805	20.91	1.0870	22.62	1.0935	24.33	1.1000	26.04	1.1065	27.75	1.1130	29.47
1.0806	20.94	1.0871	22.65	1.0936	24.35	1.1001	26.06	1.1066	27.78	1.1131	29.49
1.0807	20.96	1.0872	22.67	1.0937	24.38	1.1002	26.09	1.1067	27.80	1.1132	29.52
1.0808	20.99	1.0873	22.70	1.0938	24.41	1.1003	26.12	1.1068	27.83	1.1133	29.54
1.0809	21.02	1.0874	22.72	1.0939	24.43	1.1004	26.14	1.1069	27.86	1.1134	29.57
1.0810	21.04	1.0875	22.75	1.0940	24.46	1.1005	26.17	1.1070	27.88	1.1135	29.60
1.0811	21.07	1.0876	22.78	1.0941	24.49	1.1006	26.20	1.1071	27.90	1.1136	29.62
1.0812	21.10	1.0877	22.80	1.0942	24.51	1.1007	26.22	1.1072	27.93	1.1137	29.65
1.0813	21.12	1.0878	22.83	1.0943	24.54	1.1008	26.25	1.1073	27.96	1.1138	29.68
1.0814	21.15	1.0879	22.86	1.0944	24.57	1.1009	26.27	1.1074	27.99	1.1139	29.70
1.0815	21.17	1.0880	22.88	1.0945	24.59	1.1010	26.30	1.1075	28.01	1.1140	29.73
1.0816	21.20	1.0881	22.91	1.0946	24.62	1.1011	26.33	1.1076	28.04	1.1141	29.76
1.0817	21.23	1.0882	22.93	1.0947	24.64	1.1012	26.35	1.1077	28.07	1.1142	29.78
1.0818	21.25	1.0883	22.96	1.0948	24.67	1.1013	26.38	1.1078	28.09	1.1143	29.81
1.0819	21.28	1.0884	22.99	1.0949	24.70	1.1014	26.41	1.1079	28.12	1.1144	29.83
1.0820	21.31	1.0885	23.01	1.0950	24.72	1.1015	26.43	1.1080	28.15	1.1145	29.86
1.0821	21.33	1.0886	23.04	1.0951	24.75	1.1016	26.46	1.1081	28.17	1.1146	29.89
1.0822	21.36	1.0887	23.07	1.0952	24.78	1.1017	26.49	1.1082	28.20	1.1147	29.91
1.0823	21.38	1.0888	23.09	1.0953	24.80	1.1018	26.51	1.1083	28.22	1.1148	29.94
1.0824	21.41	1.0889	23.12	1.0954	24.83	1.1019	26.54	1.1084	28.25	1.1149	29.96
1.0825	21.44	1.0890	23.14	1.0955	24.85	1.1020	26.56	1.1085	28.28	1.1150	29.99
1.0826	21.46	1.0891	23.17	1.0956	24.88	1.1021	26.59	1.1086	28.30	1.1151	30.02
1.0827	21.49	1.0892	23.20	1.0957	24.91	1.1022	26.62	1.1087	28.33	1.1152	30.04
1.0828	21.52	1.0893	23.22	1.0958	24.93	1.1023	26.64	1.1088	28.36	1.1153	30.07
1.0829	21.54	1.0894	23.25	1.0959	24.96	1.1024	26.67	1.1089	28.38	1.1154	30.10
1.0830	21.57	1.0895	23.28	1.0960	24.99	1.1025	26.70	1.1090	28.41	1.1155	30.13
1.0831	21.59	1.0896	23.30	1.0961	25.01	1.1026	26.72	1.1091	28.43	1.1156	30.15
1.0832	21.62	1.0897	23.33	1.0962	25.04	1.1027	26.75	1.1092	28.46	1.1157	30.18
1.0833	21.65	1.0898	23.35	1.0963	25.07	1.1028	26.78	1.1093	28.49	1.1158	30.21
1.0834	21.67	1.0899	23.38	1.0964	25.09	1.1029	26.80	1.1094	28.51	1.1159	30.23
1.0835	21.70	1.0900	23.41	1.0965	25.12	1.1030	26.83	1.1095	28.54		
1.0836	21.73	1.0901	23.43	1.0966	25.14	1.1031	26.85	1.1096	28.57		
1.0837	21.75	1.0902	23.46	1.0967	25.17	1.1032	26.88	1.1097	28.59		
1.0838	21.78	1.0903	23.49	1.0968	25.20	1.1033	26.91	1.1098	28.62		
1.0839	21.80	1.0904	23.51	1.0969	25.22	1.1034	26.93	1.1099	28.65		
1.0840	21.83	1.0905	23.54	1.0970	25.25	1.1035	26.96	1.1100	28.67		
1.0841	21.86	1.0906	23.57	1.0971	25.28	1.1036	26.99	1.1101	28.70		
1.0842	21.88	1.0907	23.59	1.0972	25.30	1.1037	27.01	1.1102	28.73		
1.0843	21.91	1.0908	23.62	1.0973	25.33	1.1038	27.04	1.1103	28.75		
1.0844	21.94	1.0909	23.65	1.0974	25.36	1.1039	27.07	1.1104	28.78		



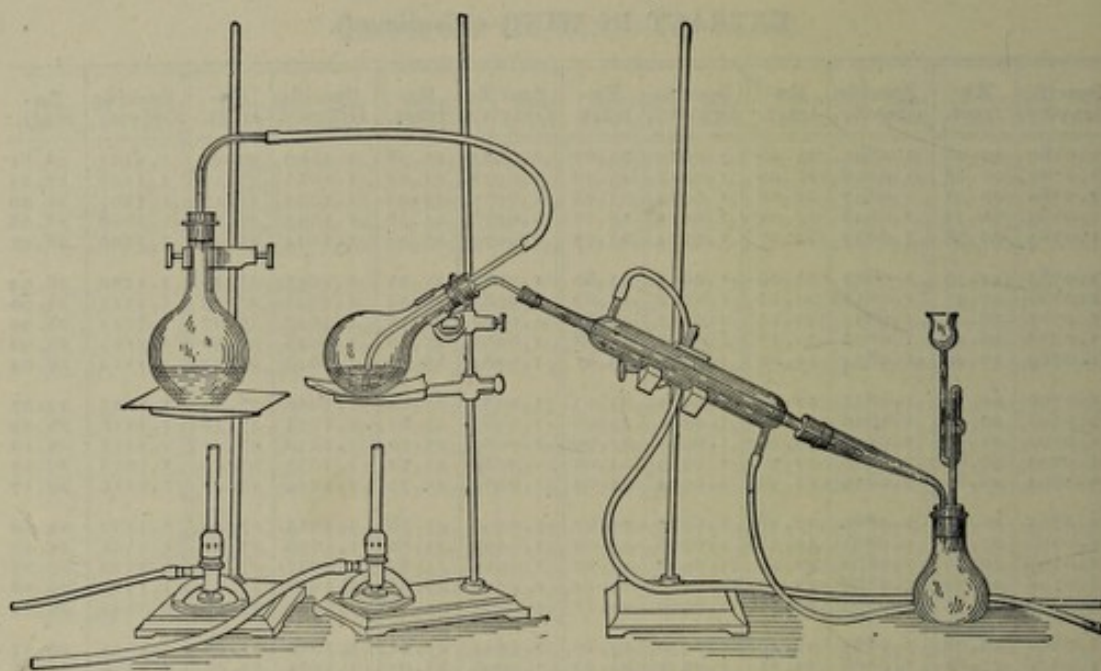


FIG. 114.—Apparatus for Determining Volatile Acids in Wine.

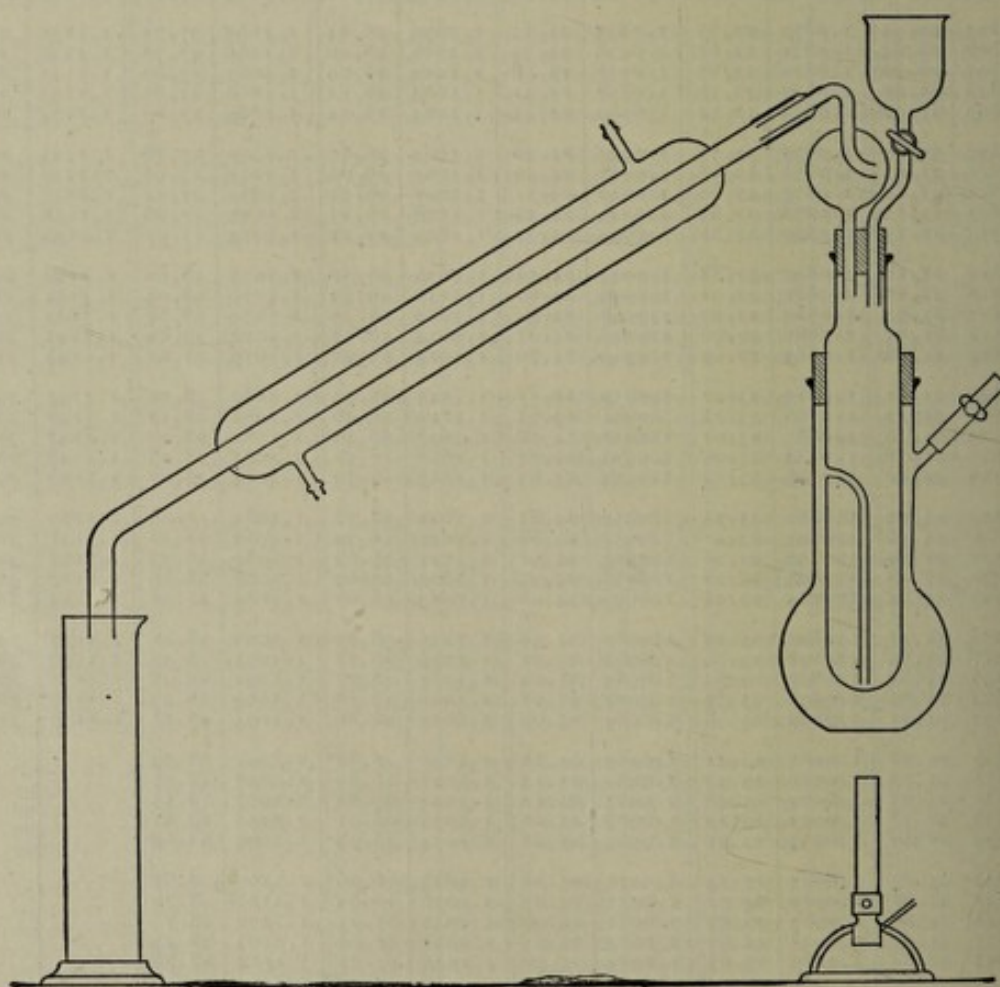


FIG. 115.—Hortvet's Apparatus for Determining the Volatile Acids in Wine.



**Hortvet Method.\***—The apparatus (Fig. 115) consists of a 300-cc. flask into the neck of which is fitted a 200-cc. cylindrical flask, with a steam tube, a bulb-trap leading to a condenser, and a stop-cock funnel. The procedure is as follows: Pour 150 cc. of recently boiled water into the larger flask, attach the smaller flask by means of a section of rubber tubing, run in 10 cc. of wine (previously freed from carbonic acid), close the stop-cock and boil. In extreme cases add to the wine a small piece of paraffin to prevent foaming. When the water has boiled a moment, close the tube at the side of the larger flask and distil until 70 cc. of distillate have passed over. Transfer to a beaker, without discontinuing the distillation, and titrate, using phenolphthalein as indicator. Continue the distillation until the last 10 cc. portion requires not more than one drop of tenth-normal alkali for neutralization. Usually 80 or 90 cc. of distillate includes practically all of the volatile acids. Cool the apparatus, thus allowing the wine residue to be drawn back into the lower flask, rinse with boiled water, and reserve the total liquid for determination of non-volatile acids.

**Cruess and Bettoli Method.†**—Shake 75 cc. of the wine with bone black free from carbonates, filter, and titrate 20 cc. of the decolorized filtrate with  $N/10$  alkali using phenolphthalein as indicator. Place another aliquot of 20 cc. of the decolorized wine in a 200-cc. Erlenmeyer flask, add 2 grams of sodium chloride, and evaporate rapidly until sodium chloride separates copiously and the liquid begins to spatter. Dilute with 20 cc. of water and repeat the operation. Dilute a second time and titrate with  $N/10$  alkali as before. The difference between the two titrations multiplied by 0.03 gives the amount of volatile acids in grams per 100 cc.

**Detection of Free Tartaric Acid.—Nessler's Method.**—Some powdered cream of tartar is added to a portion of the wine in a corked flask, which is shaken from time to time, and the liquid finally filtered. To the filtrate is added a little 20% potassium acetate solution. If free tartaric acid is present, on stirring and especially after standing for some time, there will be a precipitate of cream of tartar.

**Determination of Total Tartaric Acid.—Hartmann and Eoff Method.‡**  
—Neutralize 100 cc. of the wine with  $N$  sodium hydroxide to counteract the influence of free mineral acids, especially phosphoric; if more than 10 cc.

\* Jour. Ind. Eng. Chem., 1, 1909, p. 31.

† Ses. Int. Cong. Vit. Off. Rep., 1915, 263.

‡ U. S. Dept. of Agric., Bur. of Chem., Bul. 162, 1913, p. 72; Jour. Assn. Off. Agric. Chem., 2, II, 1917, p. 182.



are required evaporate to about 100 cc. Dissolve in the solution for each cc. of alkali added 0.075 gram accurately weighed, powdered c.p. tartaric acid, dried for 2 hours at 100° C. Add 2 cc. of glacial acetic acid and 15 grams of potassium chloride, stir until dissolved, then add 15 cc. of 95% alcohol, and stir vigorously until cream of tartar begins to precipitate. Allow to stand for at least 15 hours in an ice box, decant onto a Gooch crucible or Büchner funnel, and carefully wash the precipitate and filter three times with a solution of 15 grams of potassium chloride in 20 cc. of 95% alcohol and 100 cc. of water, using a total of not more than 20 cc. Return contents of the crucible or funnel to the beaker, rinsing with 50 cc. of hot water, heat to boiling, and titrate the solution while hot with N/10 sodium hydroxide using phenolphthalein as indicator. Add to the number of cc. required 1.5 to correct for solubility of cream of tartar and multiply by 0.015 to obtain the total weight of tartaric acid present in the solution. Subtract from the product the weight of tartaric acid added, thus obtaining the total tartaric acid present in 100 cc. of wine.

**Determination of Cream of Tartar.**—Exhaust the ash of 50 cc. of wine with hot water on a filter, add 25 cc. of N/10 hydrochloric acid, heat to incipient boiling, and titrate with N/10 alkali solution, using litmus as indicator. Deduct from 25 cc. the number of cc. of N/10 alkali employed, and multiply the remainder by 0.0188 to obtain potassium bitartrate in grams.

**Determination of Free Tartaric Acid.**—Add 25 cc. of N/10 hydrochloric acid to the ash of 50 cc. of wine, heat to incipient boiling, and titrate with N/10 sodium hydroxide, using litmus as indicator. Deduct the number of cc. of alkali employed from 25, and multiply the remainder by 0.0075 to obtain the amount of tartaric acid necessary to combine with all the ash (considering it to consist entirely of potash). Deduct the figure so obtained from the total tartaric acid. The difference is the amount of free tartaric acid.

**Determination of Lactic Acid.**—*Möslinger Method* \* *Modified by Baragiola and Schuppli*.†—Distil in steam 25 cc. of the sample and 25 cc. of water in von der Heide's fractionating apparatus until 200 cc. have been collected. Transfer the residue to a small dish, add 5 cc. of 10% barium chloride solution and hot saturated barium hydroxide solution to neutral

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\* Zeits. Unters. Nahr. Genussm., 4, 1901, p. 1123.

† Ibid., 27, 1914, p. 841.



reaction, then 2 to 4 cc. in excess, and heat for ten minutes on a water-bath. Add hydrochloric acid until neutral to azolitmin paper, evaporate to 10 to 15 cc., taking care that the reaction remains neutral, transfer to a graduated cylinder, rinsing with water, and make up to 25 cc. Add 95% alcohol gradually with shaking up to 100 cc. and after standing for several hours again adjust the volume to 100 cc. and filter. To 75 cc. of the filtrate add 25 cc. of 5% sodium sulphate solution, shake, filter after standing fifteen minutes, evaporate 75 cc. of the filtrate in platinum, and ignite to whiteness. Take up the ash in water, add an excess of N/10 hydrochloric acid, heat for five minutes on a water-bath, and titrate back with standard alkali using azolitmin paper, methyl orange, or phenolphthalein as indicator.

To obtain the lactic acid in grams per liter multiply the corrected number of N/10 acid by 0.64.

*Möslinger Method Modified by Roettgen.\**—Distil 50 cc. of the wine in a fractionating apparatus, provided with a column containing glass beads, until 200 cc. have passed over. To the residue add 5 cc. of 20% sulphuric acid, extract for twenty-four hours with ether in a continuous flow apparatus, add 30 cc. of water to the extract, and remove the ether by cautious distillation. To the residue add barium hydroxide solution to slightly alkaline reaction, heat for fifteen minutes on a water-bath, taking care that the solution remains slightly alkaline, then neutralize with N/4 hydrochloric acid, evaporate to 10 cc., and transfer to a 100-cc. graduated flask, rinsing with 5 cc. of hot water and 95% alcohol. Make up to the mark with 95% alcohol, cool to 15° C. with shaking, and after thirty minutes at that temperature again make up to the mark, and allow to stand two hours longer at 15° C. Filter into a graduated cylinder, cool to 15°, read the volume of the filtrate, then evaporate in a platinum dish, ignite, cool, and titrate with N/4 hydrochloric acid.

Calculate as in the Baragiola and Schuppli modification, taking account of the amount of wine used, the aliquot, and the strength of the standard solution.

Determine free lactic acid in the same manner except that no sulphuric acid is added after distillation.

Results obtained by Roettgen indicate that all or practically all of the lactic acid present in wine exists in the form of free lactic acid.

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\* Zeits. Unters. Nahr. Genussm., 24, 1912, p. 113; 26, 1913, p. 437; 30, 1915, p. 294; 34, 1917, p. 198.



**Polarization.**—Treat a measured amount of wine or cider with one-tenth of its volume of lead subacetate, filter and polarize the filtrate in the 200 mm. tube. The reading is increased by 10% for the true direct polarization.

If the reducing sugars are also to be determined, the same solutions may be used for both the polarization and the reducing sugars as follows:

Exactly neutralize with sodium hydroxide solution 200 cc. of the wine, using litmus paper as an indicator, and evaporate on the water-bath to about one-fourth its original volume. Wash with water into a 200 cc. flask, add enough normal lead acetate solution to clarify, and make up with water to the mark. Filter and to the filtrate add powdered sodium sulphate or carbonate sufficient to precipitate the lead, again filter and polarize before and after inversion (page 610).

**Determination of Reducing Sugars.**—Determine reducing sugars in portions of the wine treated as described in the preceding section, after dilution so as not to contain above 0.5% of sugar for the Defren and the Munson and Walker methods or above 1% of sugar for the Allihn method. One may assume 2% as the sugar-free extract of wine, the number of volumes of water to be added to the filtrate being determined by the difference between 2 and the total extract as determined.

**Determination of Glycerol.**—*In Dry Wines.*—Evaporate 100 cc. of the wine in a porcelain dish on the water-bath to about 10 cc., add about 5 grams of fine sand and from 3 to 4 cc. of milk of lime (containing about 15% of calcium oxide) for each gram of extract present and evaporate nearly to dryness. Treat the moist residue with 50 cc. of 95% (by vol.) alcohol, remove the substance adhering to the sides of the dish with a spatula, and rub the whole mass to a paste. Heat on a water-bath, with constant stirring, to incipient boiling and decant through a filter into a small flask. Wash by decantation with 10 cc. portions of hot 95% alcohol until the filtrate amounts to about 150 cc. Evaporate the filtrate to a sirup on a hot, but not boiling, water-bath, transfer to a small glass-stoppered graduated cylinder with 20 cc. of absolute alcohol, and add 3 portions of 10 cc. each of absolute ether, shaking thoroughly after each addition. Let stand until clear, then pour off through a filter and wash the cylinder with a mixture of absolute alcohol and absolute ether (1:1.5), pouring the wash liquor also through the filter. Evaporate the filtrate to a sirup, dry for one hour in a boiling-water oven, weigh, ignite, and weigh again. The loss on ignition gives the weight of glycerol.



A more accurate method is that proposed by Ross and described under vinegar, page 801.

*In Sweet Wines.*—If the extract exceeds 5% heat 100 cc. to boiling in a flask and treat with successive small portions of milk of lime until the color becomes at first darker and then lighter. When cool add 200 cc. of 95% alcohol, allow the precipitate to subside, filter, and wash with 95% alcohol. With the filtrate thus obtained proceed as directed for dry wines.

**Determination of Potassium Sulphate.**—Acidify 100 cc. of the sample with hydrochloric acid, heat to boiling, and add an excess of barium chloride solution. Filter, wash, dry, ignite, and weigh as barium sulphate, calculating the equivalent of potassium sulphate.

**Determination of Sodium Chloride.**—To 50 cc. of the wine add sodium carbonate solution until alkaline, evaporate, burn at low redness and determine chlorine gravimetrically as silver chloride.

**Detection of Nitrates.**—*Egger Method.\**—Treat a few drops of the wine in a porcelain dish with 2 or 3 cc. of concentrated sulphuric acid which contains about 0.1 gram of diphenylamin per 100 cc. The deep blue color formed in the presence of nitrates appears so quickly that it is not obscured, even in sweet wine, by the blackening produced by the action of sulphuric acid on the sugar. In the case of red wines clarify with lead subacetate, removing the excess with sodium sulphate.

**Determination of Tannin.**—*Neubauer-Löwenthal Method.\**—The reagents are those given on pages 429 and 430, also finely pulverized bone black extracted with hydrochloric acid and washed with distilled water until neutral. It should be kept covered with water.

Dealcoholize 100 cc., dilute with water to the original volume, transfer 10 cc. to a porcelain dish of about 2 liters capacity, add about a liter of water and exactly 20 cc. of indigo solution. Add tenth-normal potassium permanganate solution, one cc. at a time, until the blue color changes to green, then a few drops at a time until the color becomes golden yellow. Designate the number of cubic centimeters of permanganate solution employed as "a."

Treat 10 cc. of the dealcoholized wine, prepared as above, with bone-black for fifteen minutes; filter and wash thoroughly with water. Add a liter of water and 20 cc. of indigo solution and titrate with permanganate

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 107 (rev.) p. 88.



as above. Designate the number of cubic centimeters of permanganate employed as "*b*."

Then  $a - b = c$ , the number of cubic centimeters of permanganate solution required for the oxidation of the tannin and coloring matter in 10 cc. of wine. 1 cc. = 0.004157 gram of tannin.

**Detection and Determination of Preservatives.**—See Chapter XVIII.

**Detection of Colors.**—*Dupré Method*. \*—Dissolve 1 part of pure gelatin in 10 parts boiling water, pour upon a plate to harden and cut into 2 cm. cubes. Immerse one of the cubes in the suspected sample, allow to remain for twenty-four hours, wash slightly in cold water, and cut through with a knife. If the color is a natural one, it will lightly tinge the outer surface of the cube, but will not permeate far below the surface, so that the inner portion of the cross-section will be largely free from color. Nearly all foreign coloring matters used in wine, such as most coal-tar dyes, cochineal, Brazil wood, logwood, etc., will be found to deeply permeate the jelly cube often to the center. Information as to the nature of the color may sometimes be gained by immersing the dyed jelly cube in weak ammonia. If the color be rosanilin, the cube is decolorized; if cochineal, a purple coloration will result, and if logwood, a brown tinge.

*Cazeneuve Method*.—While by no means complete and not of recent origin the scheme of Cazeneuve (page 737) as condensed and arranged by Gautier (*La Sophistication des Vins*) will often be found helpful. If other colors than these are evidently present, tests should be made as indicated in Chapter XVII. Cazeneuve employs the following reagents:

- (1) Yellow oxide of mercury, finely pulverized.
- (2) Lead hydrate, freshly precipitated, well washed, suspended in about twice its volume of water; to be kept in a stoppered bottle; should be renewed after several days' use.
- (3) Gelatinous ferric hydrate, well washed from ammonia, suspended in about twice its volume of water.
- (4) Manganese dioxide, pulverized.
- (5) Concentrated, chemically pure sulphuric acid.
- (6) White wool.
- (7) Stannous hydrate, freshly precipitated, well washed, suspended in water, and kept from exposure to light and air.
- (8) Collodion silk, the artificial silk produced from nitro-cellulose. This fiber has a special affinity for basic dyes.

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\* Jour. Chem. Soc., 37, p. 572.



To 10 cc. of the wine are added 0.2 gram finely powdered yellow oxide of mercury.  
Boil and pour upon a double filter.

Filtrate colored either before or after acidifying.		Filtrate colorless after acidifying.
<p>Filtrate colored yellow. 10 cc. of the wine are warmed with 2 grams lead hydrate. Filter.</p> <p>Filtrate colored yellow. A large excess of lead hydrate is added and the liquid is boiled.</p> <p>Filtrate is colored red. Boil up with wool fibers, which after washing and drying are treated with pure concentrated <math>H_2SO_4</math>. Color produced is</p> <p>Solution colorless. Wool fibers are dyed, washed, dried, and treated with pure concentrated <math>H_2SO_4</math>. Color produced is</p> <p>Solution colored yellow, but only faintly. Wool fibers are dyed, washed, dried, and treated with pure concentrated <math>H_2SO_4</math>. Color produced is</p> <p>Filtrate colored blue. The wine is boiled with collodion silk. Fiber is colored blue</p>		<p>Ten cc. of wine are treated with 10 grams of iron hydrate and brought to boiling.</p> <p>Filtrate colorless ....</p> <p>Filtrate colored ....</p> <p>To a portion of the wine is added its weight of manganese dioxide. Acidify and filter.</p> <p>Ten cc. wine are treated with 20 grams stannous hydrate and brought to boiling.</p> <p>A fluorescent rose.....</p> <p>A non-fluorescent rose.....</p> <p>Colorless. ....</p> <p>Colored. ....</p> <p>Pure wine</p> <p>Wine-colored with vegetable pigments</p> <p>Cochineal</p> <p>Eosin</p> <p>Erythrosin</p> <p>Fuchsin</p> <p>Acid fuchsin</p> <p>Filtrate yellowish.</p> <p>Filtrate colorless or yellowish.</p> <p>Filtrate yellow-red or red.</p> <p>Purple-violet.</p> <p>Blue-violet</p> <p>Blue.....</p> <p>Crimson.....</p> <p>Deep green.....</p> <p>Indigo-blue.....</p> <p>Violet.....</p> <p>Roccellin</p> <p>Purple-red</p> <p>Bordeaux red</p> <p>Ponceaux</p> <p>Biebrich scarlet</p> <p>Croceine 3B</p> <p>Croceine 7B</p> <p>Sulphur</p> <p>Tropaeolin 000, 1 and 2</p> <p>Orange 1 and 2</p> <p>Tropaeolin 0</p> <p>Tropaeolin y.</p> <p>Tropaeolin oo (Orange IV)</p> <p>Helianthin, Orange III</p> <p>Chrysoin</p> <p>Vesuvium</p> <p>Brown.....</p> <p>Yellow, becoming salmon-red on diluting with water.....</p> <p>Blue-green.....</p> <p>Brown-yellow.....</p> <p>Golden yellow.....</p> <p>Ammonia is added to the wine freed from its natural color by yellow oxide of mercury. Causes a blue to yellow precipitate.</p> <p>Methylene blue</p> <p>Martius yellow</p> <p>Yellow NS</p> <p>Solid yellow</p> <p>Yellow N</p>



## MALT LIQUORS. BEER.

In its widest sense beer may be defined as the product of fermentation of an infusion of almost any farinaceous grain with various bitter extractives, but unless otherwise qualified it should be strictly applied to the beverage resulting from the fermentation of malted barley and hops. In the manufacture of beer two distinct processes are employed, viz., malting or sprouting the grain, and brewing. Many brewers do nothing but the latter, buying their malt already prepared.

**Malting.**—For the preparation of malt, the barley is steeped in water for several days, after which the water is drained off and the moist grain is "couched," or piled in heaps, on a cement floor, where it undergoes a spontaneous heating process, during which it germinates, forming the ferment diastase. When the maximum amount of diastase has been produced, indicated by the length of growth of the sprout, or "acrosipre" within the grain, the germination is checked by spreading the grain in layers over a perforated iron floor, and finally subjecting it to artificial heat. The character of the malt and of the beer produced from it depends largely on the heat at which the "green" malt is kiln dried. If dried between 32° and 37° C. it forms pale malt, which produces the lightest grades of beer. Most beer is made from malt dried at higher temperatures, say from 38° to 50°, the depth of color of the liquor varying with the heat to which the malt has been subjected, while the color of the malt varies from the "pale" through the "amber" to "brown," or even black. The darkest grades are sometimes dried at temperatures over 100° C., even to the point where the starch becomes caramelized.

A more modern method consists in the so-called pneumatic malting, wherein the whole operation is conducted in a large rotating drum, which holds the grain, and in which the temperature and moisture at different stages is carefully controlled by the admission to the interior of the drum of moisture-laden or dry air, heated to the required degree.

The chief object of malting is the production of diastase, which by its subsequent action on the starch converts it into the fermentable sugars maltose and dextrin. Malt contains much more diastase than is necessary to convert the starch simply contained therein to maltose, and is capable of acting on the starch of a considerable quantity of raw grain, such as corn or rice, when mixed with it. This practice of using other grains than malt is prohibited in some localities, such as Bavaria.



**Brewing.**—The malt, or mixture of malt and raw grain, is first crushed and “mashed” by stirring with water in tubs at 50° to 60° C., finally heating to 70°. During this process the conversion of the starch to maltose and dextrin takes place. The resulting liquor is known as “wort,” containing, besides maltose and dextrin, peptones and amides. The clear wort is then drawn off from the residue, and boiled to concentrate the product and to sterilize it, after which hops (the female flower of the *Humulus lupulus*) are added and the boiling continued. Hops contain resins, bitter principles, tannic acid, and a peculiar essential oil, all of which are to some extent imparted to the wort. After cooling and settling, the clear wort is run into fermenting-vats, where selected yeast, usually *saccharomyces cerevisiæ*, is added, and the alcoholic fermentation allowed to proceed. The temperature greatly affects the character of the fermentation. If kept between 5° and 8° C., a slow fermentation proceeds, known as bottom fermentation, during which the yeast settles out at the bottom. This is much more easily controlled than the quick or top fermentation, which takes place at from 15° to 18°, much of the yeast in the latter case being carried to the surface, from which it is finally removed by skimming. In either case the yeast feeds upon the albuminous matter present. At the proper stage the beer is drawn off from the larger portion of the yeast, and run into casks, or tuns, in which an after-fermentation proceeds. The beer is finally clarified by treatment with gelatin or beech shavings or chips, to which the floating yeast-cells and other impurities attach themselves. It is finally stored in barrels coated with brewers’ pitch, or pasteurized at 60° C. and bottled.

**Varieties of Beer.**—Formerly the division of beers into “lager,” “schenk,” and “bock” was made by reason of the fact that beer had to be brewed under certain climatic conditions and at certain seasons only. Now, with improved means for artificial refrigeration, and with better methods controlling all stages of the process, these distinctions are less marked.

*Lager Beer* (from *lager*, a storehouse) is a term originally applied to Bavarian beer, but is now given to any beer that has been stored several months. Formerly lager beer was made early in the winter, and stored in cool cellars till the following spring or summer, during nearly all of which time a slow after-fermentation took place. The best lager beers contain a low proportion of hops, and are high in extract and alcohol.

*Schenk Beer* is a quickly fermented beer made in winter for immedi-



ate use. It is brewed in from four to six weeks and will not keep long without souring.

*Bock Beer*, according to older systems of nomenclature, occupied a middle place between lager and schenk, being an extra strong beer brewed for spring use and made in limited quantities, not being intended for storage.

*Berlin Weiss Bier* is prepared by the quick or top fermentation of a wort consisting of a mixture of malted barley and wheat with hops. It is high in carbon dioxide, being usually bottled before the second fermentation has ended.

*Ale* is virtually the English name for beer. It is usually lighter colored than lager beer, being made from pale malt by quick or top fermentation, and containing rather more hops than beer. It has a high content of sugar, due to checking fermentation at an earlier stage than in ordinary beer.

*Porter* is a dark ale, the deep color of which should be due to the use of brown malt dried at a high temperature, but which is sometimes colored by the admixture of caramel. It has a large extract, chiefly sugar.

*Stout* is an extra-strong porter, being high both in alcohol and extract.

**Composition of Beer.**—Beer is a somewhat complex liquor. Besides water, alcohol, and sugar, it contains carbon dioxide, succinic acid, dextrin, glycerin, tannic acid, the resinous bitter principles of hops, nitrogenous bodies (chiefly peptones and amides), alkaline and lime salts (chiefly phosphates), fat (traces), acetic acid and lactic acid. The latter acid constitutes the chief fixed acid of beer.

The following analyses of different varieties of beer are due to König:

Variety.	Number of Analyses.	Specific Gravity.	Water.	Carbonic Acid.	Alcohol by Weight.	Extract.	Nitrogenous Substances.	Sugar as Maltose.	Gum and Dextrin.	Acid as Lactic.	Glycerin.	Ash.	Phosphoric Acid.
Schenk. ....	205	1.0114	91.11	0.197	3.36	5.34	0.74	0.95	3.11	0.156	0.12	0.204	0.055
Lager. ....	258	1.0162	90.08	0.196	3.93	5.79	0.71	0.88	3.73	0.151	0.165	0.228	0.077
Export beer.	109	1.0176	89.01	0.209	4.40	6.38	0.74	1.20	3.47	0.161	0.154	0.247	0.074
Bock.....	84	1.0213	87.87	0.234	4.69	7.21	0.73	1.81	3.97	0.165	0.176	0.263	0.089
Weiss beer..	26	1.0137	91.63	0.297	2.73	5.34	0.58	1.62	2.42	0.392	0.092	0.149	0.034
Porter.....	40	1.0191	88.49	0.215	4.70	6.59	0.65	2.62	3.08	0.281	.....	0.363	0.093
Ale. ....	38	1.0141	89.42	0.201	4.75	5.65	0.61	1.07	1.81	0.278	.....	0.31	0.086

Fifteen samples of lager beer and seven samples of pale ale, bought in Massachusetts bar-rooms, representing as nearly as possible the quality



of liquor sold every day to patrons by the bottle or glass, were analyzed by the Board of Health with the following results:

		Per Cent of Original Wort Extract.	Per Cent of Alcohol by Weight.	Per Cent of Extract.
Beer—	Maximum.....	18.91	7.07	7.76
	Minimum.....	7.33	1.10	3.67
	Mean.....	15.04	4.45	5.92
Pale ale—	Maximum.....	15.99	5.37	5.47
	Minimum.....	10.95	3.53	3.38
	Mean.....	13.56	4.49	4.54

Five out of the 15 beer samples and 3 out of the 7 ale samples contained salicylic acid.

The percentage composition of the ash of German beer is thus given by König as the mean of 19 analyses:

Ash in 100 Parts Beer.	Potash.	Soda.	Lime.	Magnesia.	Iron Oxide.	Phos- phoric Acid.	Sul- phuric Acid.	Silica.	Chlorine.
0.306	33.67	8.94	2.78	6.24	0.48	31.35	3.47	9.29	2.93

**Malt and Hop Substitutes.**—By reason of the fluctuation in market price of these two chief constituents of beer, it sometimes becomes a question of economy to employ cheaper substitutes wholly or in part for one or the other. There are two classes of malt substitutes, (1) those which, like corn grits, rice, and wheat, are mixed directly with the malt before "mashing," and, like the malt, have to undergo a saccharous fermentation before being acted on by yeast, and (2) such substances as cane sugar, invert sugar, brewers' sugar, and dextrin, which are added to the wort at a later stage in the brewing, just before the addition of the yeast, being in condition to be readily acted on by the latter.

Brewers' sugar is the common substitute of the second class because of the fact that its sugars much resemble those of malt, and are in readily fermentable form. Diastase forms from the malt dextrin and maltose, while brewers' sugar contains dextrin, maltose, and dextrose.

When the price of malt is abnormally high, the addition of brewers' sugar may be economical, but when ordinary conditions prevail, the cost of the two, figured with reference to their yield in alcohol and extract, is about the same. Aside from the question of economy, however, there are advantages in the use of malt substitutes, such as diminishing the nitrogenous content of the wort without lessening the alcohol or extract yielded.



The nitrogenous matter left after fermentation is one of the chief causes of cloudiness or turbidity in the finished product, and is sometimes difficult to remove. By the use of brewers' sugar, especially in clear bottled ales and sparkling pale beers, the appearance of the product is much enhanced. The temptation at times to add more than is necessary to accomplish this is great. A high-grade malt may have as much as 40% of brewers' sugar added to its wort and still produce an acceptable beer. With a low-grade malt, brewers' sugar yields a very poor quality of beer. Hence its use may or may not be desirable, though it can hardly be considered unqualifiedly as an adulterant.

As to the employment of hop substitutes, the question of relative price again enters in. Only when the price of hops is high is there any special inducement to use substitutes. Quassia wood, chiretta, gentian, and calumba, all of which yield bitter principles, have been used in beer, and cannot be considered detrimental to health. Allen and Chattaway have found the first two in beer examined by them.\* Such poisonous substances as cocculus indicus, picric acid, and strychnine are alleged to have been used as hop substitutes, but there is no authentic record of any of them having been found in recent years, if at all.

**Adulteration of Malt Liquors and Standards of Purity.**—The Joint Committee on Standards adopted in 1908 the following standards:

*Malt Liquor* is a beverage made by the alcoholic fermentation of an infusion, in potable water, of barley malt and hops, with or without unmalted grains or decorticated and degerminated grains.

*Beer* is a malt liquor produced by bottom fermentation, and contains in 100 cc., at 20° C., not less than 5 grams of extractive matter and 0.16 gram of ash, chiefly potassium phosphate, and not less than 2.25 grams of alcohol.

*Lager Beer, Stored Beer*, is beer which has been stored in casks for a period of at least three months, and contains, in 100 cc., at 20° C., not less than 5 grams of extractive matters, and 0.16 gram of ash, chiefly potassium phosphate, and not less than 2.50 grams of alcohol.

*Malted Beer* is beer made of an infusion, in potable water, of barley, malt, and hops, and contains, in 100 cc., at 20° C., not less than 5 grams of extractive matter, nor less than 0.2 gram of ash, chiefly potassium phosphate, not less than 2.25 grams of alcohol, not less than 0.4 gram of crude protein (nitrogen  $\times 6.25$ ).

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\* Analyst, 12, 112.



*Ale* is a malt liquor produced by top fermentation, and contains, in 100 cc., at 20° C., not less than 2.75 grams of alcohol, nor less than 5 grams of extract, and not less than 0.16 gram of ash, chiefly potassium phosphate.

*Porter* and *Stout* are varieties of malt liquors made in part from highly roasted malt.

Non-injurious bitter principles are no doubt employed in place of hops, and unless the liquor is sold for a pure malt beer, they cannot be regarded as adulterants.

The tendency to shorten the time of storage of beer, or to sell it without storing at all, lessens or does away with the after-fermentation, resulting in a lack of "life" or effervescence in the product. This is sometimes made up by the addition of sodium bicarbonate.

**Distinction between Malted and Non-malted Liquors.**—In some states where strict prohibitory liquor laws are in force, it is illegal to sell "malt liquors," so that when convictions are obtained, it is necessary for the analyst to distinguish between liquors brewed wholly or in part from malt and those in which no malt has been used, but which were brewed entirely from malt substitutes. This distinction is not always easy to make with precision. In the absence of malt, brewers' sugar is usually the source of alcohol in these beverages. Parsons\* has shown that the most striking points of difference between malted and non-malted liquors are in their per cent of phosphoric acid and proteins, and that malted beer or ale should contain not less than 0.04%  $P_2O_5$ , and 0.25% protein ( $N \times 6.25$ ). A low ash and high content of sulphates in the ash are also indicative of brewers' sugar. The following analyses made by Parsons clearly show these distinctions:

COMPOSITION OF SEVENTY-SIX SAMPLES OF AMERICAN MALT LIQUORS.

	Specific Gravity.	Alcohol by Volume.	Extract.	Protein ( $N \times 6.25$ )	Phosphoric Acid.	Ash.	Sulphates in Ash.	Free Acid.
Average.....	1.0100	5.61	4.61	0.470	0.061	0.209	6.34	0.26
Maximum.....	1.0210	7.85	7.64	0.614	0.095	0.296	12.67	0.87
Minimum.....	1.0047	0.35	3.15	0.290	0.045	0.147	2.44	0.10

\* Jour. Am. Chem. Soc., 1902, 24, 1170.



## TYPICAL ANALYSES OF BEERS APPARENTLY NOT BREWED FROM MALT.

Number.	Specific Gravity.	Alcohol by Volume.	Extract.	Protein (N × 6.25)	Phosphoric Acid.	Ash.	Sulphates.	Free Acid.
1.....	1.0074	1.68	2.52	0.114	0.010	0.19	21.22	Normal
2.....	1.0098	2.63	3.40	0.215	0.023	0.180	.....	"
3.....	1.0062	2.27	2.25	0.150	0.015	0.124	11.30	"
4.....	1.0112	2.11	3.53	0.133	0.015	0.140	10.81	"
5.....	1.0041	1.85	1.73	0.031	0.010	0.088	12.50	"

The ash of the fifth sample is thus compared with that of the average beer as given by Blyth:

	Malt Beer (Blyth).	"No-malt" Beer (Parsons).
K <sub>2</sub> O.....	37.22	12.93
Na <sub>2</sub> O.....	8.04	19.61
CaO.....	1.93	Undetermined
MgO.....	5.51	"
Fe <sub>2</sub> O <sub>3</sub> .....	Trace	"
SO <sub>3</sub> .....	1.44	10.81
P <sub>2</sub> O <sub>5</sub> .....	32.09	10.71
Cl.....	2.91	21.76
SiO <sub>2</sub> .....	10.82	7.50

**Distinction between all Malt and Malt Substitute Liquors.**—In countries other than Bavaria a considerable, if not the larger part, of the malt liquors is brewed from part malt and part malt substitutes such as rice, corn grits, cerealine, etc. While the use of the malt substitutes produces a product of a somewhat different character from all malt beer and ale this is not necessarily inferior, in fact it is preferred by many consumers. There exists, nevertheless, a strong prejudice in favor of the all-malt product to meet which that brewed in part from substitutes has often been misbranded so as to convey the impression that it was brewed solely from malt and hops.

Tolman and Riley,\* who have investigated the production of both types of malt liquors, find that the all-malt products contain higher percentages of ash, proteins, and phosphoric acid when calculated to a uniform wort basis. These distinctions are clearly brought out by their results calculated to the basis of wort containing 15% of solids given in the table on page 745. In making the calculation the percentage of solids in the

\* U. S. Dept. of Agric., Bul. 493, 1917.



original wort was first obtained by multiplying the per cent by weight of alcohol by 2 and adding the per cent by weight of extract.

ASH, PROTEIN, AND PHOSPHORIC ACID IN MALT LIQUORS OF KNOWN ORIGIN.

Raw Materials.	Product.	Ash.	Protein (N $\times$ 6.25).	Phosphoric Acid (P <sub>2</sub> O <sub>5</sub> ).
Malt.....	Beer (21 samples)			
	Maximum	0.336	1.079	0.143
	Minimum	0.230	0.701	0.087
	Average	0.275	0.870	0.109
Malt 80%, rice 20%.....	Beer	0.202	0.517	0.073
" 66 " 34 .....	"	0.198	0.555	0.084
" 62 " 38 .....	"	0.205	0.488	0.061
" 55 " 45 .....	"	0.148	0.380	0.077
" 50 " 50 .....	"	0.167	0.351	0.056
	Maximum	0.205	0.555	0.084
Malt 70%, corn 30%.....	Beer	0.199	0.343	0.057
" 70 " 30 .....	"	0.188	0.367	0.065
" 68 " 32 .....	"	0.150	0.461	0.057
" 68 " 32 .....	"	0.181	0.466	0.062
" 68 " 32 .....	"	0.164	0.459	0.056
" 60 " 40 .....	"	0.215	0.563	0.074
" 60 " 40 .....	"	0.188	0.593	0.076
" 60 " 40 .....	"	0.223	0.597	0.074
" 45 " 55 .....	"	0.145	0.347	0.057
	Maximum	0.223	0.597	0.076
Malt 65%, cereal 35%.....	Beer	0.192	0.483	0.057
" 80 " 20 .....	Ale	0.215	0.480	0.051
" 78 " 22 .....	"	0.176	0.455	0.050
" 78 " 22 .....	"	0.169	0.476	0.045
" 78 " 22 .....	"	0.181	0.502	0.040
" 75 " 25 .....	"	0.204	0.499	0.044
" 75 " 25 .....	"	0.196	0.509	0.044
" 75 " 25 .....	"	0.191	0.502	0.043
" 65 " 28 brewer's sugar 7%.....	"	0.185	0.409	0.037
" 65 " 28 " " 7 .....	"	0.175	0.443	0.040
" 65 " 28 " " 7 .....	"	0.166	0.427	0.041
	Maximum	0.213	0.509	0.051



**Preservatives in Beer.**—Antiseptics are frequently added to malt liquors, salicylic acid being most commonly used. Fluorides of ammonium and sodium have been found in American beer. Other preservatives to be looked for are benzoic acid and sulphites. Beer casks are frequently "sulphured" or fumed with a solution of calcium bisulphite, so that the beer may derive its content of sulphites from this source.

In case of police seizure of beer sold in bulk or in opened bottles for the purpose of ascertaining whether or not their alcoholic content exceeds certain limits fixed by law, a little formalin had best be added as soon as possible after the seizure to prevent further fermentation. This is especially desirable in cases where there is likely to be some delay in making the analysis, so as to forestall any claim on the part of the defendant of additional alcohol being formed after the seizure. From 6 to 8 drops of a 40% solution of formaldehyde to a quart of beer is sufficient, and this quantity will not appreciably affect the analysis.

**Arsenic in Beer.**—In 1900 a very disastrous epidemic of arsenical poisoning occurred in Manchester, England, involving several thousand cases, many of which were fatal. The arsenic was traced to sulphuric acid, which entered into the manufacture of commercial glucose used in the beer, the acid found so highly arsenical being made from a certain variety of Swedish pyrites, abnormally high in arsenic. The evidence was conclusive that the beer was the sole cause of the trouble. While the presence of arsenic was in this case accidental, carelessness was shown on the part of those having to do with the purity of the materials entering into the composition of the beer. Further details are given by Kelynack and Kirby.\* Fortunately no other instances are on record of arsenical poisoning from malted liquors. A large number of samples of American beer have been examined in the laboratory of the Food and Drug Department of the Massachusetts State Board of Health, and only insignificant traces of arsenic have in any case been found.

**Temperance Beers and Ales.**—Many varieties of these so-called temperance drinks are home-made, as well as sold on the market. They are usually slightly fermented infusions of various roots and herbs, including ginger or sassafras, with molasses or sugar and yeast, and more often contain less than 1% of alcohol by volume. Among them are included spruce beer, and the various root beers, such as ginger beer and ginger ale. The latter beverages are generally carbonated. Numerous brands

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\* Arsenical Poisoning in Beer Drinkers, London, 1901.



of bottled beer are manufactured, which contain virtually the same body and characteristic flavor as lager beer, but less alcohol. Indeed the composition of many of these beverages is identical with that of lager beer, excepting in alcoholic content, being made by substantially the same process and out of the same ingredients, but with the alcohol finally removed by steaming, so that the liquor comes within the limits of a temperature beverage. Of this class is *Uno beer*, which ranges from 0.6 to 0.9 per cent in alcohol.

#### METHODS OF ANALYSIS OF MALT LIQUORS.\*

**Preparation of Sample.**—Transfer the contents of the bottle or bottles to a large flask and shake vigorously to hasten the escape of carbon dioxide, care being taken that the liquor is not below  $15^{\circ}\text{C}$ ., since below this temperature the carbon dioxide is retained by the beer and is liable to form bubbles in the pycnometer.

**Specific Gravity.**—See page 686.

**Ash.**—Determine in 25 cc. by evaporation and ignition at dull redness.

**Determination of Alcohol.**—*From the Specific Gravity of the Distillate.*—Proceed as described on page 687, employing 100 cc. of the liquor, and determining the specific gravity at  $15.5^{\circ}\text{C}$ . If the liquor is markedly acid, add first 0.1 to 0.2 gram of precipitated calcium carbonate.

*From the Refraction of the Distillate.*—Prepare the distillate as described on page 687, except that it is made up to the mark at  $17.5^{\circ}\text{C}$ . Determine the refraction at  $17.5^{\circ}\text{C}$ . by means of the immersion refractometer, and calculate the alcohol by the table on page 748.

**Determination of Extract.**—In cases where extreme accuracy is desired, the result obtained by evaporating at  $100^{\circ}$  a weighed amount of the beer cannot be accepted, on account of the dehydration of the maltose at a temperature exceeding  $75^{\circ}\text{C}$ . Unless the evaporation is conducted at that temperature (a difficult operation), a closer approximation to the truth is obtained, especially with beer high in sugar, by calculation as follows:

*From the Specific Gravity.*—Evaporate a measured quantity of the beer to one-fourth its volume on the water-bath, make up with water to its original measure, and determine the specific gravity of the dealcoholized beer. Then by means of Schultz and Ostermann's table, pp. 749-753, calculate the extract corresponding.

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\* Barnard, U. S. Dept. of Agric., Bur. of Chem., Circ. 33.



ACKERMANN AND STEINMANN'S TABLE FOR OBTAINING THE PERCENTAGE OF ALCOHOL IN THE DISTILLATE OF BEER FROM THE IMMERSION REFRACTOMETER READINGS.\*

Refractometer Reading.	Alcohol by Weight, Per Cent.	Alcohol by Volume, Per Cent.	Refractometer Reading.	Alcohol by Weight, Per Cent.	Alcohol by Volume, Per Cent.	Refractometer Reading.	Alcohol by Weight, Per Cent.	Alcohol by Volume, Per Cent.	Refractometer Reading.	Alcohol by Weight, Per Cent.	Alcohol by Volume, Per Cent.
15.0	0.00	0.00	17.2	1.38	1.74	19.4	2.74	3.46	21.6	4.02	5.06
15.1	0.06	0.08	17.3	1.44	1.82	19.5	2.80	3.53	21.7	4.07	5.13
15.2	0.13	0.16	17.4	1.51	1.90	19.6	2.86	3.61	21.8	4.13	5.20
15.3	0.19	0.24	17.5	1.57	1.98	19.7	2.91	3.68	21.9	4.18	5.26
15.4	0.25	0.32	17.6	1.63	2.05	19.8	2.97	3.75	22.0	4.22	5.32
15.5	0.32	0.40	17.7	1.68	2.12	19.9	3.04	3.83	22.1	4.28	5.39
15.6	0.38	0.48	17.8	1.74	2.20	20.0	3.10	3.90	22.2	4.33	5.46
15.7	0.44	0.56	17.9	1.81	2.28	20.1	3.15	3.97	22.3	4.39	5.53
15.8	0.50	0.64	18.0	1.87	2.36	20.2	3.20	4.04	22.4	4.44	5.59
15.9	0.57	0.72	18.1	1.93	2.44	20.3	3.26	4.11	22.5	4.49	5.65
16.0	0.64	0.80	18.2	2.00	2.52	20.4	3.33	4.19	22.6	4.54	5.72
16.1	0.70	0.88	18.3	2.06	2.60	20.5	3.38	4.26	22.7	4.59	5.78
16.2	0.77	0.96	18.4	2.13	2.68	20.6	3.43	4.33	22.8	4.64	5.85
16.3	0.83	1.04	18.5	2.19	2.76	20.7	3.50	4.41	22.9	4.70	5.92
16.4	0.88	1.12	18.6	2.25	2.84	20.8	3.56	4.48	23.0	4.76	6.00
16.5	0.95	1.19	18.7	2.31	2.92	20.9	3.61	4.55	23.1	4.81	6.07
16.6	1.01	1.27	18.8	2.37	2.99	21.0	3.67	4.63	23.2	4.86	6.13
16.7	1.05	1.33	18.9	2.43	3.07	21.1	3.73	4.71	23.3	4.92	6.20
16.8	1.13	1.43	19.0	2.49	3.14	21.2	3.78	4.77	23.4	4.97	6.27
16.9	1.19	1.51	19.1	2.55	3.22	21.3	3.84	4.84	23.5	5.02	6.33
17.0	1.25	1.58	19.2	2.61	3.29	21.4	3.90	4.92			
17.1	1.32	1.66	19.3	2.68	3.37	21.5	3.96	4.99			

\* Zeits. gesamte Brauwesen, 28, 1905, p. 259.

*From the Refraction.—Method of Ackermann and Foggenburg.*—Determine the refraction of the liquor at 17.5° C. by means of the immersion refractometer. Determine also the refraction of the distillate from 100 cc. of the liquor at 17.5° C., after making up to its original volume. In order to secure accurate results, care should be taken to cool the prism of the instrument to exactly 17.5° C. by immersing for five minutes in the water-bath previous to taking the refraction of the liquids. If determinations are made on a number of samples, this cooling is not necessary except before taking the reading of the first of the series.

Calculate the grams of extract (*E*) from the refraction of the liquor (*R*) and of the distillate (*R'*) by the following formula:

$$E = 0.25705(R - R').$$

The extract is more conveniently obtained from Ackermann's table given on page 755.



EXTRACT IN BEER WORT.\*  
[ACCORDING TO SCHULTZ AND OSTERMANN.]

Specific Gravity at 15° C.	Extract.		Specific Gravity at 15° C.	Extract.		Specific Gravity at 15° C.	Extract.		Specific Gravity at 15° C.	Extract.	
	Per Cent by Weight	Grams per 100 cc.		Per Cent by Weight	Grams per 100 cc.		Per Cent by Weight	Grams per 100 cc.		Per Cent by Weight	Grams per 100 cc.
1.0000	0.00	0.00	1.0065	1.60	1.70	1.0130	3.35	3.30	1.0105	5.06	5.16
1.0001	0.01	0.01	1.0066	1.72	1.73	1.0131	3.36	3.42	1.0106	5.07	5.17
1.0002	0.02	0.02	1.0067	1.74	1.75	1.0132	3.41	3.46	1.0107	5.12	5.22
1.0003	0.03	0.03	1.0068	1.77	1.78	1.0133	3.43	3.48	1.0108	5.15	5.25
1.0004	0.10	0.10	1.0069	1.79	1.80	1.0134	3.46	3.51	1.0109	5.17	5.27
1.0005	0.13	0.13	1.0070	1.82	1.83	1.0135	3.48	3.53	1.0200	5.20	5.30
1.0006	0.16	0.16	1.0071	1.84	1.85	1.0136	3.51	3.56	1.0201	5.23	5.34
1.0007	0.18	0.18	1.0072	1.87	1.88	1.0137	3.54	3.59	1.0202	5.25	5.36
1.0008	0.21	0.21	1.0073	1.90	1.91	1.0138	3.56	3.61	1.0203	5.28	5.39
1.0009	0.24	0.24	1.0074	1.92	1.93	1.0139	3.59	3.64	1.0204	5.30	5.41
1.0010	0.26	0.26	1.0075	1.95	1.96	1.0140	3.61	3.66	1.0205	5.33	5.44
1.0011	0.29	0.29	1.0076	1.97	1.98	1.0141	3.64	3.69	1.0206	5.35	5.46
1.0012	0.31	0.31	1.0077	2.00	2.02	1.0142	3.66	3.71	1.0207	5.38	5.49
1.0013	0.34	0.34	1.0078	2.02	2.04	1.0143	3.69	3.74	1.0208	5.40	5.51
1.0014	0.37	0.37	1.0079	2.05	2.07	1.0144	3.72	3.77	1.0209	5.43	5.54
1.0015	0.39	0.39	1.0080	2.07	2.09	1.0145	3.74	3.79	1.0210	5.45	5.56
1.0016	0.42	0.42	1.0081	2.10	2.12	1.0146	3.77	3.83	1.0211	5.48	5.60
1.0017	0.45	0.45	1.0082	2.12	2.14	1.0147	3.79	3.85	1.0212	5.50	5.62
1.0018	0.47	0.47	1.0083	2.15	2.17	1.0148	3.82	3.88	1.0213	5.53	5.65
1.0019	0.50	0.50	1.0084	2.17	2.19	1.0149	3.85	3.91	1.0214	5.55	5.67
1.0020	0.52	0.52	1.0085	2.20	2.22	1.0150	3.87	3.93	1.0215	5.57	5.69
1.0021	0.55	0.55	1.0086	2.23	2.25	1.0151	3.90	3.96	1.0216	5.60	5.72
1.0022	0.58	0.58	1.0087	2.25	2.27	1.0152	3.92	3.98	1.0217	5.62	5.74
1.0023	0.60	0.60	1.0088	2.28	2.30	1.0153	3.95	4.01	1.0218	5.65	5.77
1.0024	0.63	0.63	1.0089	2.30	2.32	1.0154	3.97	4.03	1.0219	5.67	5.79
1.0025	0.66	0.66	1.0090	2.33	2.35	1.0155	4.00	4.06	1.0220	5.70	5.83
1.0026	0.68	0.68	1.0091	2.35	2.37	1.0156	4.03	4.09	1.0221	5.72	5.85
1.0027	0.71	0.71	1.0092	2.38	2.40	1.0157	4.05	4.11	1.0222	5.75	5.88
1.0028	0.73	0.73	1.0093	2.41	2.43	1.0158	4.08	4.14	1.0223	5.77	5.90
1.0029	0.76	0.76	1.0094	2.43	2.45	1.0159	4.10	4.17	1.0224	5.80	5.93
1.0030	0.79	0.79	1.0095	2.46	2.48	1.0160	4.13	4.20	1.0225	5.82	5.95
1.0031	0.81	0.81	1.0096	2.48	2.50	1.0161	4.16	4.23	1.0226	5.84	5.97
1.0032	0.84	0.84	1.0097	2.51	2.53	1.0162	4.18	4.25	1.0227	5.87	6.00
1.0033	0.87	0.87	1.0098	2.53	2.55	1.0163	4.21	4.28	1.0228	5.89	6.02
1.0034	0.89	0.89	1.0099	2.56	2.59	1.0164	4.23	4.30	1.0229	5.92	6.06
1.0035	0.92	0.92	1.0100	2.58	2.61	1.0165	4.26	4.33	1.0230	5.94	6.08
1.0036	0.94	0.94	1.0101	2.61	2.64	1.0166	4.28	4.35	1.0231	5.97	6.11
1.0037	0.97	0.97	1.0102	2.64	2.67	1.0167	4.31	4.38	1.0232	5.99	6.13
1.0038	1.00	1.00	1.0103	2.66	2.69	1.0168	4.34	4.41	1.0233	6.02	6.16
1.0039	1.02	1.02	1.0104	2.69	2.72	1.0169	4.36	4.43	1.0234	6.04	6.18
1.0040	1.05	1.05	1.0105	2.71	2.74	1.0170	4.39	4.46	1.0235	6.07	6.21
1.0041	1.08	1.08	1.0106	2.74	2.77	1.0171	4.42	4.50	1.0236	6.09	6.23
1.0042	1.10	1.10	1.0107	2.76	2.79	1.0172	4.44	4.52	1.0237	6.11	6.25
1.0043	1.13	1.13	1.0108	2.79	2.82	1.0173	4.47	4.55	1.0238	6.14	6.29
1.0044	1.15	1.15	1.0109	2.82	2.85	1.0174	4.50	4.58	1.0239	6.16	6.31
1.0045	1.18	1.18	1.0110	2.84	2.87	1.0175	4.53	4.61	1.0240	6.19	6.34
1.0046	1.21	1.22	1.0111	2.87	2.90	1.0176	4.55	4.63	1.0241	6.21	6.36
1.0047	1.23	1.24	1.0112	2.89	2.92	1.0177	4.58	4.66	1.0242	6.24	6.39
1.0048	1.26	1.27	1.0113	2.92	2.95	1.0178	4.61	4.69	1.0243	6.26	6.41
1.0049	1.29	1.30	1.0114	2.94	2.97	1.0179	4.63	4.71	1.0244	6.29	6.44
1.0050	1.31	1.32	1.0115	2.97	3.00	1.0180	4.66	4.74	1.0245	6.31	6.46
1.0051	1.34	1.35	1.0116	2.99	3.02	1.0181	4.69	4.77	1.0246	6.34	6.50
1.0052	1.36	1.37	1.0117	3.02	3.06	1.0182	4.71	4.80	1.0247	6.36	6.52
1.0053	1.39	1.40	1.0118	3.05	3.09	1.0183	4.74	4.83	1.0248	6.39	6.55
1.0054	1.41	1.42	1.0119	3.07	3.11	1.0184	4.77	4.86	1.0249	6.41	6.57
1.0055	1.44	1.45	1.0120	3.10	3.14	1.0185	4.79	4.88	1.0250	6.44	6.60
1.0056	1.46	1.47	1.0121	3.12	3.16	1.0186	4.82	4.91	1.0251	6.47	6.63
1.0057	1.49	1.50	1.0122	3.15	3.19	1.0187	4.85	4.94	1.0252	6.50	6.66
1.0058	1.51	1.52	1.0123	3.17	3.21	1.0188	4.88	4.97	1.0253	6.52	6.68
1.0059	1.54	1.55	1.0124	3.20	3.24	1.0189	4.90	4.99	1.0254	6.55	6.72
1.0060	1.56	1.57	1.0125	3.23	3.27	1.0190	4.93	5.02	1.0255	6.58	6.75
1.0061	1.59	1.60	1.0126	3.25	3.29	1.0191	4.96	5.05	1.0256	6.61	6.78
1.0062	1.62	1.63	1.0127	3.28	3.32	1.0192	4.98	5.08	1.0257	6.63	6.80
1.0063	1.64	1.65	1.0128	3.30	3.34	1.0193	5.01	5.11	1.0258	6.66	6.83
1.0064	1.67	1.68	1.0129	3.33	3.37	1.0194	5.04	5.14	1.0259	6.69	6.86

\* Calculated from results obtained by drying below 75° C.



## EXTRACT IN BEER WORT—(Continued).

Specific Gravity at 15° C.	Extract.		Specific Gravity at 15° C.	Extract.		Specific Gravity at 15° C.	Extract.		Specific Gravity at 15° C.	Extract.	
	Per Cent by Weight	Grams per 100 cc.		Per Cent by Weight	Grams per 100 cc.		Per Cent by Weight	Grams per 100 cc.		Per Cent by Weight	Grams per 100 cc.
1.0260	6.71	6.88	1.0325	8.27	8.54	1.0390	9.92	10.31	1.0455	11.53	12.05
1.0261	6.74	6.92	1.0326	8.29	8.56	1.0391	9.95	10.34	1.0456	11.55	12.08
1.0262	6.77	6.95	1.0327	8.32	8.59	1.0392	9.97	10.36	1.0457	11.57	12.10
1.0263	6.80	6.98	1.0328	8.34	8.61	1.0393	9.99	10.38	1.0458	11.60	12.13
1.0264	6.82	7.00	1.0329	8.37	8.65	1.0394	10.02	10.41	1.0459	11.62	12.15
1.0265	6.85	7.03	1.0330	8.40	8.68	1.0395	10.04	10.44	1.0460	11.65	12.19
1.0266	6.88	7.06	1.0331	8.43	8.71	1.0396	10.06	10.46	1.0461	11.67	12.21
1.0267	6.91	7.09	1.0332	8.45	8.73	1.0397	10.09	10.49	1.0462	11.70	12.24
1.0268	6.93	7.12	1.0333	8.48	8.76	1.0398	10.11	10.51	1.0463	11.72	12.26
1.0269	6.96	7.15	1.0334	8.51	8.79	1.0399	10.13	10.53	1.0464	11.75	12.30
1.0270	6.99	7.18	1.0335	8.53	8.82	1.0400	10.16	10.57	1.0465	11.77	12.32
1.0271	7.01	7.20	1.0336	8.56	8.85	1.0401	10.18	10.59	1.0466	11.79	12.34
1.0272	7.04	7.23	1.0337	8.59	8.88	1.0402	10.20	10.61	1.0467	11.82	12.37
1.0273	7.07	7.26	1.0338	8.61	8.90	1.0403	10.23	10.64	1.0468	11.84	12.39
1.0274	7.10	7.29	1.0339	8.64	8.93	1.0404	10.25	10.66	1.0469	11.87	12.43
1.0275	7.12	7.32	1.0340	8.67	8.96	1.0405	10.27	10.69	1.0470	11.89	12.45
1.0276	7.15	7.35	1.0341	8.70	9.00	1.0406	10.30	10.72	1.0471	11.92	12.48
1.0277	7.18	7.38	1.0342	8.72	9.02	1.0407	10.32	10.74	1.0472	11.94	12.50
1.0278	7.21	7.41	1.0343	8.75	9.05	1.0408	10.35	10.77	1.0473	11.97	12.54
1.0279	7.23	7.43	1.0344	8.78	9.08	1.0409	10.37	10.79	1.0474	11.99	12.56
1.0280	7.26	7.46	1.0345	8.80	9.10	1.0410	10.40	10.83	1.0475	12.01	12.58
1.0281	7.28	7.48	1.0346	8.83	9.14	1.0411	10.42	10.85	1.0476	12.04	12.61
1.0282	7.30	7.51	1.0347	8.86	9.17	1.0412	10.45	10.88	1.0477	12.06	12.64
1.0283	7.33	7.54	1.0348	8.88	9.19	1.0413	10.47	10.90	1.0478	12.09	12.67
1.0284	7.35	7.56	1.0349	8.91	9.22	1.0414	10.50	10.93	1.0479	12.11	12.69
1.0285	7.37	7.58	1.0350	8.94	9.25	1.0415	10.52	10.96	1.0480	12.14	12.72
1.0286	7.39	7.60	1.0351	8.97	9.28	1.0416	10.55	10.99	1.0481	12.16	12.74
1.0287	7.42	7.63	1.0352	8.99	9.31	1.0417	10.57	11.01	1.0482	12.19	12.78
1.0288	7.44	7.65	1.0353	9.02	9.34	1.0418	10.60	11.04	1.0483	12.21	12.80
1.0289	7.46	7.68	1.0354	9.05	9.37	1.0419	10.62	11.06	1.0484	12.23	12.82
1.0290	7.48	7.70	1.0355	9.07	9.39	1.0420	10.65	11.10	1.0485	12.26	12.85
1.0291	7.51	7.73	1.0356	9.10	9.42	1.0421	10.67	11.12	1.0486	12.28	12.88
1.0292	7.53	7.75	1.0357	9.13	9.46	1.0422	10.70	11.15	1.0487	12.31	12.91
1.0293	7.55	7.77	1.0358	9.15	9.48	1.0423	10.72	11.17	1.0488	12.33	12.93
1.0294	7.57	7.79	1.0359	9.18	9.51	1.0424	10.75	11.21	1.0489	12.36	12.96
1.0295	7.60	7.82	1.0360	9.21	9.54	1.0425	10.77	11.23	1.0490	12.38	12.99
1.0296	7.62	7.85	1.0361	9.24	9.57	1.0426	10.80	11.26	1.0491	12.41	13.02
1.0297	7.64	7.87	1.0362	9.26	9.60	1.0427	10.82	11.28	1.0492	12.43	13.04
1.0298	7.66	7.89	1.0363	9.29	9.63	1.0428	10.85	11.31	1.0493	12.45	13.06
1.0299	7.69	7.92	1.0364	9.31	9.65	1.0429	10.88	11.35	1.0494	12.48	13.10
1.0300	7.71	7.94	1.0365	9.34	9.68	1.0430	10.90	11.37	1.0495	12.50	13.12
1.0301	7.73	7.96	1.0366	9.36	9.70	1.0431	10.93	11.40	1.0496	12.53	13.15
1.0302	7.75	7.98	1.0367	9.38	9.72	1.0432	10.95	11.42	1.0497	12.55	13.17
1.0303	7.77	8.01	1.0368	9.41	9.76	1.0433	10.98	11.46	1.0498	12.58	13.21
1.0304	7.80	8.04	1.0369	9.43	9.78	1.0434	11.00	11.48	1.0499	12.60	13.23
1.0305	7.82	8.06	1.0370	9.45	9.80	1.0435	11.03	11.51	1.0500	12.63	13.26
1.0306	7.84	8.08	1.0371	9.48	9.83	1.0436	11.05	11.53	1.0501	12.65	13.28
1.0307	7.86	8.10	1.0372	9.50	9.85	1.0437	11.08	11.56	1.0502	12.67	13.31
1.0308	7.89	8.13	1.0373	9.52	9.88	1.0438	11.10	11.59	1.0503	12.70	13.34
1.0309	7.91	8.15	1.0374	9.55	9.91	1.0439	11.13	11.62	1.0504	12.72	13.36
1.0310	7.93	8.18	1.0375	9.57	9.93	1.0440	11.15	11.64	1.0505	12.75	13.39
1.0311	7.95	8.20	1.0376	9.59	9.95	1.0441	11.18	11.67	1.0506	12.77	13.42
1.0312	7.98	8.23	1.0377	9.62	9.98	1.0442	11.20	11.70	1.0507	12.80	13.45
1.0313	8.00	8.25	1.0378	9.64	10.00	1.0443	11.23	11.73	1.0508	12.82	13.47
1.0314	8.02	8.27	1.0379	9.66	10.03	1.0444	11.25	11.75	1.0509	12.85	13.50
1.0315	8.04	8.29	1.0380	9.69	10.06	1.0445	11.28	11.78	1.0510	12.87	13.53
1.0316	8.07	8.33	1.0381	9.71	10.08	1.0446	11.30	11.80	1.0511	12.90	13.56
1.0317	8.09	8.35	1.0382	9.73	10.10	1.0447	11.33	11.84	1.0512	12.92	13.58
1.0318	8.11	8.37	1.0383	9.76	10.13	1.0448	11.35	11.86	1.0513	12.94	13.60
1.0319	8.13	8.39	1.0384	9.78	10.16	1.0449	11.38	11.89	1.0514	12.97	13.64
1.0320	8.16	8.42	1.0385	9.81	10.19	1.0450	11.40	11.91	1.0515	12.99	13.66
1.0321	8.18	8.44	1.0386	9.83	10.21	1.0451	11.43	11.95	1.0516	13.02	13.69
1.0322	8.20	8.46	1.0387	9.85	10.23	1.0452	11.45	11.97	1.0517	13.04	13.71
1.0323	8.22	8.49	1.0388	9.88	10.26	1.0453	11.48	12.00	1.0518	13.07	13.75
1.0324	8.25	8.52	1.0389	9.90	10.29	1.0454	11.50	12.02	1.0519	13.09	13.77



## EXTRACT IN BEER WORT—(Continued).

Specific Gravity at 15° C.	Extract.		Specific Gravity at 15° C.	Extract.		Specific Gravity at 15° C.	Extract.		Specific Gravity at 15° C.	Extract.	
	Per Cent by Weight	Grams per 100 cc.		Per Cent by Weight	Grams per 100 cc.		Per Cent by Weight	Grams per 100 cc.		Per Cent by Weight	Grams per 100 cc.
1.0520	13.12	13.80	1.0585	14.75	15.61	1.0650	16.25	17.31	1.0715	17.81	19.08
1.0521	13.14	13.82	1.0586	14.78	15.65	1.0651	16.27	17.33	1.0716	17.84	19.12
1.0522	13.16	13.85	1.0587	14.81	15.68	1.0652	16.30	17.36	1.0717	17.86	19.14
1.0523	13.19	13.88	1.0588	14.83	15.70	1.0653	16.32	17.39	1.0718	17.88	19.16
1.0524	13.21	13.90	1.0589	14.86	15.74	1.0654	16.35	17.42	1.0719	17.90	19.19
1.0525	13.24	13.94	1.0590	14.89	15.77	1.0655	16.37	17.44	1.0720	17.93	19.22
1.0526	13.26	13.96	1.0591	14.91	15.79	1.0656	16.40	17.48	1.0721	17.95	19.24
1.0527	13.29	13.99	1.0592	14.94	15.82	1.0657	16.42	17.50	1.0722	17.97	19.27
1.0528	13.31	14.01	1.0593	14.96	15.85	1.0658	16.45	17.53	1.0723	17.99	19.29
1.0529	13.34	14.05	1.0594	14.99	15.88	1.0659	16.47	17.56	1.0724	18.02	19.32
1.0530	13.36	14.07	1.0595	15.02	15.91	1.0660	16.50	17.59	1.0725	18.04	19.35
1.0531	13.38	14.09	1.0596	15.04	15.94	1.0661	16.52	17.61	1.0726	18.06	19.37
1.0532	13.41	14.12	1.0597	15.07	15.97	1.0662	16.54	17.63	1.0727	18.08	19.39
1.0533	13.43	14.15	1.0598	15.09	15.99	1.0663	16.57	17.67	1.0728	18.11	19.43
1.0534	13.46	14.18	1.0599	15.11	16.02	1.0664	16.59	17.69	1.0729	18.13	19.45
1.0535	13.48	14.20	1.0600	15.14	16.05	1.0665	16.62	17.73	1.0730	18.15	19.47
1.0536	13.51	14.23	1.0601	15.16	16.07	1.0666	16.64	17.75	1.0731	18.17	19.50
1.0537	13.53	14.26	1.0602	15.18	16.09	1.0667	16.67	17.78	1.0732	18.20	19.53
1.0538	13.56	14.29	1.0603	15.20	16.12	1.0668	16.69	17.80	1.0733	18.22	19.55
1.0539	13.58	14.31	1.0604	15.23	16.15	1.0669	16.72	17.84	1.0734	18.24	19.58
1.0540	13.61	14.34	1.0605	15.25	16.17	1.0670	16.74	17.86	1.0735	18.26	19.60
1.0541	13.63	14.37	1.0606	15.27	16.20	1.0671	16.76	17.88	1.0736	18.29	19.64
1.0542	13.66	14.40	1.0607	15.29	16.22	1.0672	16.79	17.92	1.0737	18.31	19.66
1.0543	13.68	14.42	1.0608	15.31	16.24	1.0673	16.81	17.94	1.0738	18.33	19.68
1.0544	13.71	14.46	1.0609	15.34	16.27	1.0674	16.84	17.98	1.0739	18.35	19.71
1.0545	13.73	14.48	1.0610	15.36	16.30	1.0675	16.86	18.00	1.0740	18.38	19.74
1.0546	13.76	14.51	1.0611	15.38	16.32	1.0676	16.89	18.03	1.0741	18.40	19.76
1.0547	13.78	14.53	1.0612	15.40	16.34	1.0677	16.91	18.05	1.0742	18.42	19.79
1.0548	13.81	14.57	1.0613	15.43	16.38	1.0678	16.94	18.09	1.0743	18.44	19.81
1.0549	13.83	14.59	1.0614	15.45	16.40	1.0679	16.96	18.11	1.0744	18.47	19.84
1.0550	13.86	14.62	1.0615	15.47	16.42	1.0680	16.99	18.15	1.0745	18.49	19.87
1.0551	13.88	14.64	1.0616	15.49	16.44	1.0681	17.01	18.17	1.0746	18.51	19.89
1.0552	13.91	14.68	1.0617	15.52	16.48	1.0682	17.03	18.19	1.0747	18.53	19.91
1.0553	13.93	14.70	1.0618	15.54	16.50	1.0683	17.06	18.23	1.0748	18.55	19.94
1.0554	13.96	14.73	1.0619	15.56	16.52	1.0684	17.08	18.25	1.0749	18.57	19.96
1.0555	13.98	14.76	1.0620	15.58	16.55	1.0685	17.11	18.28	1.0750	18.59	19.98
1.0556	14.01	14.79	1.0621	15.60	16.57	1.0686	17.13	18.31	1.0751	18.62	20.02
1.0557	14.03	14.81	1.0622	15.63	16.60	1.0687	17.16	18.34	1.0752	18.64	20.04
1.0558	14.06	14.84	1.0623	15.65	16.62	1.0688	17.18	18.36	1.0753	18.66	20.07
1.0559	14.08	14.87	1.0624	15.67	16.64	1.0689	17.21	18.40	1.0754	18.68	20.09
1.0560	14.11	14.90	1.0625	15.69	16.66	1.0690	17.23	18.42	1.0755	18.70	20.11
1.0561	14.13	14.92	1.0626	15.72	16.70	1.0691	17.25	18.44	1.0756	18.72	20.14
1.0562	14.16	14.96	1.0627	15.74	16.73	1.0692	17.28	18.48	1.0757	18.74	20.16
1.0563	14.18	14.98	1.0628	15.76	16.75	1.0693	17.30	18.50	1.0758	18.76	20.18
1.0564	14.21	15.01	1.0629	15.78	16.77	1.0694	17.33	18.53	1.0759	18.78	20.21
1.0565	14.23	15.03	1.0630	15.80	16.80	1.0695	17.35	18.56	1.0760	18.81	20.24
1.0566	14.26	15.07	1.0631	15.83	16.83	1.0696	17.38	18.59	1.0761	18.83	20.26
1.0567	14.28	15.09	1.0632	15.85	16.85	1.0697	17.40	18.61	1.0762	18.85	20.29
1.0568	14.31	15.12	1.0633	15.87	16.87	1.0698	17.43	18.65	1.0763	18.87	20.31
1.0569	14.33	15.15	1.0634	15.89	16.90	1.0699	17.45	18.67	1.0764	18.89	20.33
1.0570	14.36	15.18	1.0635	15.92	16.93	1.0700	17.48	18.70	1.0765	18.91	20.36
1.0571	14.38	15.20	1.0636	15.94	16.95	1.0701	17.50	18.73	1.0766	18.93	20.38
1.0572	14.41	15.23	1.0637	15.96	16.98	1.0702	17.52	18.75	1.0767	18.95	20.40
1.0573	14.44	15.27	1.0638	15.98	17.00	1.0703	17.54	18.77	1.0768	18.97	20.43
1.0574	14.46	15.29	1.0639	16.01	17.03	1.0704	17.57	18.81	1.0769	19.00	20.46
1.0575	14.49	15.32	1.0640	16.03	17.06	1.0705	17.59	18.83	1.0770	19.02	20.48
1.0576	14.52	15.36	1.0641	16.05	17.08	1.0706	17.61	18.85	1.0771	19.04	20.51
1.0577	14.54	15.38	1.0642	16.07	17.10	1.0707	17.63	18.88	1.0772	19.06	20.53
1.0578	14.57	15.41	1.0643	16.09	17.12	1.0708	17.66	18.91	1.0773	19.08	20.55
1.0579	14.59	15.43	1.0644	16.12	17.16	1.0709	17.68	18.93	1.0774	19.10	20.58
1.0580	14.62	15.47	1.0645	16.14	17.18	1.0710	17.70	18.96	1.0775	19.12	20.60
1.0581	14.65	15.50	1.0646	16.16	17.20	1.0711	17.72	18.98	1.0776	19.14	20.63
1.0584	14.67	15.52	1.0647	16.18	17.23	1.0712	17.75	19.01	1.0777	19.17	20.66
1.0583	14.70	15.56	1.0648	16.21	17.26	1.0713	17.77	19.04	1.0778	19.19	20.68
1.0584	14.73	15.59	1.0649	16.23	17.28	1.0714	17.79	19.06	1.0779	19.21	20.71



## EXTRACT IN BEER WORT—(Continued).

Specific Gravity at 15° C.	Extract.		Specific Gravity at 15° C.	Extract.		Specific Gravity at 15° C.	Extract.		Specific Gravity at 15° C.	Extract.	
	Per Cent by Weight	Grams per 100 cc.		Per Cent by Weight	Grams per 100 cc.		Per Cent by Weight	Grams per 100 cc.		Per Cent by Weight	Grams per 100 cc.
1.0780	19.23	20.73	1.0845	20.70	22.45	1.0910	22.19	24.21	1.0975	23.59	25.89
1.0781	19.25	20.75	1.0846	20.73	22.48	1.0911	22.21	24.24	1.0976	23.61	25.92
1.0782	19.27	20.78	1.0847	20.75	22.50	1.0912	22.23	24.26	1.0977	23.63	25.94
1.0783	19.29	20.80	1.0848	20.77	22.53	1.0913	22.26	24.29	1.0978	23.65	25.97
1.0784	19.31	20.82	1.0849	20.79	22.55	1.0914	22.28	24.31	1.0979	23.67	25.99
1.0785	19.33	20.85	1.0850	20.81	22.58	1.0915	22.30	24.34	1.0980	23.69	26.01
1.0786	19.36	20.88	1.0851	20.83	22.61	1.0916	22.32	24.37	1.0981	23.71	26.04
1.0787	19.38	20.90	1.0852	20.86	22.64	1.0917	22.34	24.39	1.0982	23.73	26.06
1.0788	19.40	20.93	1.0853	20.88	22.66	1.0918	22.37	24.42	1.0983	23.76	26.09
1.0789	19.42	20.95	1.0854	20.90	22.68	1.0919	22.39	24.44	1.0984	23.78	26.11
1.0790	19.44	20.98	1.0855	20.93	22.72	1.0920	22.41	24.47	1.0985	23.80	26.14
1.0791	19.46	21.00	1.0856	20.95	22.75	1.0921	22.43	24.49	1.0986	23.82	26.17
1.0792	19.49	21.03	1.0857	20.98	22.78	1.0922	22.45	24.51	1.0987	23.84	26.19
1.0793	19.51	21.06	1.0858	21.01	22.81	1.0923	22.48	24.54	1.0988	23.86	26.22
1.0794	19.53	21.08	1.0859	21.04	22.84	1.0924	22.50	24.56	1.0989	23.88	26.24
1.0795	19.56	21.11	1.0860	21.06	22.87	1.0925	22.52	24.60	1.0990	23.90	26.27
1.0796	19.58	21.14	1.0861	21.09	22.90	1.0926	22.54	24.62	1.0991	23.92	26.30
1.0797	19.60	21.16	1.0862	21.11	22.93	1.0927	22.56	24.64	1.0992	23.94	26.32
1.0798	19.63	21.20	1.0863	21.13	22.96	1.0928	22.59	24.67	1.0993	23.97	26.35
1.0799	19.65	21.22	1.0864	21.16	22.99	1.0929	22.61	24.70	1.0994	23.99	26.37
1.0800	19.67	21.24	1.0865	21.19	23.02	1.0930	22.63	24.73	1.0995	24.01	26.40
1.0801	19.70	21.28	1.0866	21.22	23.06	1.0931	22.65	24.76	1.0996	24.03	26.42
1.0802	19.72	21.30	1.0867	21.25	23.09	1.0932	22.67	24.78	1.0997	24.05	26.44
1.0803	19.74	21.33	1.0868	21.28	23.12	1.0933	22.69	24.81	1.0998	24.07	26.47
1.0804	19.77	21.36	1.0869	21.30	23.15	1.0934	22.71	24.83	1.0999	24.09	26.49
1.0805	19.79	21.38	1.0870	21.33	23.18	1.0935	22.73	24.86	1.1000	24.11	26.52
1.0806	19.81	21.41	1.0871	21.35	23.21	1.0936	22.75	24.89	1.1001	24.13	26.55
1.0807	19.84	21.43	1.0872	21.37	23.23	1.0937	22.77	24.91	1.1002	24.15	26.57
1.0808	19.86	21.46	1.0873	21.39	23.26	1.0938	22.80	24.93	1.1003	24.17	26.60
1.0809	19.88	21.49	1.0874	21.41	23.28	1.0939	22.82	24.96	1.1004	24.19	26.62
1.0810	19.91	21.52	1.0875	21.43	23.31	1.0940	22.84	24.99	1.1005	24.21	26.65
1.0811	19.93	21.55	1.0876	21.45	23.33	1.0941	22.86	25.01	1.1006	24.23	26.68
1.0812	19.96	21.58	1.0877	21.47	23.36	1.0942	22.88	25.03	1.1007	24.25	26.70
1.0813	19.98	21.60	1.0878	21.49	23.38	1.0943	22.90	25.06	1.1008	24.28	26.73
1.0814	20.00	21.63	1.0879	21.51	23.40	1.0944	22.92	25.08	1.1009	24.30	26.75
1.0815	20.03	21.66	1.0880	21.54	23.43	1.0945	22.94	25.11	1.1010	24.32	26.78
1.0816	20.05	21.69	1.0881	21.56	23.45	1.0946	22.96	25.14	1.1011	24.34	26.81
1.0817	20.07	21.71	1.0882	21.58	23.48	1.0947	22.98	25.16	1.1012	24.36	26.83
1.0818	20.10	21.74	1.0883	21.60	23.50	1.0948	23.00	25.18	1.1013	24.39	26.86
1.0819	20.12	21.77	1.0884	21.62	23.52	1.0949	23.03	25.21	1.1014	24.41	26.88
1.0820	20.14	21.79	1.0885	21.64	23.55	1.0950	23.05	25.24	1.1015	24.43	26.91
1.0821	20.17	21.83	1.0886	21.66	23.58	1.0951	23.07	25.26	1.1016	24.45	26.93
1.0822	20.19	21.85	1.0887	21.68	23.60	1.0952	23.10	25.29	1.1017	24.47	26.95
1.0823	20.21	21.87	1.0888	21.71	23.63	1.0953	23.12	25.31	1.1018	24.49	26.98
1.0824	20.24	21.91	1.0889	21.73	23.66	1.0954	23.14	25.34	1.1019	24.51	27.00
1.0825	20.26	21.93	1.0890	21.75	23.69	1.0955	23.16	25.37	1.1020	24.53	27.03
1.0826	20.28	21.96	1.0891	21.77	23.72	1.0956	23.18	25.39	1.1021	24.55	27.06
1.0827	20.31	21.99	1.0892	21.79	23.74	1.0957	23.20	25.42	1.1022	24.57	27.08
1.0828	20.33	22.01	1.0893	21.82	23.77	1.0958	23.23	25.45	1.1023	24.60	27.11
1.0829	20.35	22.04	1.0894	21.84	23.79	1.0959	23.25	25.47	1.1024	24.62	27.14
1.0830	20.37	22.06	1.0895	21.86	23.82	1.0960	23.27	25.50	1.1025	24.64	27.17
1.0831	20.39	22.08	1.0896	21.89	23.85	1.0961	23.29	25.53	1.1026	24.66	27.19
1.0832	20.41	22.11	1.0897	21.91	23.87	1.0962	23.31	25.55	1.1027	24.68	27.21
1.0833	20.43	22.13	1.0898	21.93	23.90	1.0963	23.33	25.58	1.1028	24.70	27.24
1.0834	20.46	22.16	1.0899	21.96	23.93	1.0964	23.35	25.60	1.1029	24.72	27.26
1.0835	20.48	22.19	1.0900	21.98	23.96	1.0965	23.37	25.63	1.1030	24.74	27.29
1.0836	20.50	22.21	1.0901	22.00	23.98	1.0966	23.39	25.66	1.1031	24.76	27.32
1.0837	20.52	22.24	1.0902	22.02	24.01	1.0967	23.41	25.68	1.1032	24.78	27.34
1.0838	20.54	22.26	1.0903	22.04	24.03	1.0968	23.44	25.71	1.1033	24.81	27.37
1.0839	20.56	22.29	1.0904	22.06	24.05	1.0969	23.46	25.73	1.1034	24.83	27.39
1.0840	20.59	22.32	1.0905	22.08	24.08	1.0970	23.48	25.76	1.1035	24.85	27.42
1.0841	20.62	22.35	1.0906	22.10	24.11	1.0971	23.50	25.79	1.1036	24.87	27.45
1.0842	20.64	22.38	1.0907	22.12	24.13	1.0972	23.52	25.81	1.1037	24.89	27.47
1.0843	20.66	22.40	1.0908	22.15	24.16	1.0973	23.55	25.84	1.1038	24.92	27.50
1.0844	20.68	22.42	1.0909	22.17	24.18	1.0974	23.57	25.86	1.1039	24.94	27.53



## EXTRACT IN BEER WORT—(Concluded).

Specific Gravity at 15° C.	Extract.		Specific Gravity at 15° C.	Extract.		Specific Gravity at 15° C.	Extract.		Specific Gravity at 15° C.	Extract.	
	Per Cent by Weight	Grams per 100 cc.		Per Cent by Weight	Grams per 100 cc.		Per Cent by Weight	Grams per 100 cc.		Per Cent by Weight	Grams per 100 cc.
1.1040	24.06	27.56	1.1095	26.16	29.03	1.1150	27.20	30.43	1.1205	28.38	31.81
1.1041	24.08	27.58	1.1096	26.18	29.06	1.1151	27.31	30.45	1.1206	28.40	31.83
1.1042	25.00	27.60	1.1097	26.20	29.08	1.1152	27.33	30.47	1.1207	28.42	31.86
1.1043	25.03	27.63	1.1098	26.23	29.11	1.1153	27.35	30.50	1.1208	28.44	31.88
1.1044	25.05	27.66	1.1099	26.25	29.13	1.1154	27.37	30.52	1.1209	28.46	31.90
1.1045	25.07	27.69	1.1100	26.27	29.16	1.1155	27.38	30.55	1.1210	28.48	31.93
1.1046	25.09	27.72	1.1101	26.29	29.19	1.1156	27.40	30.57	1.1211	28.50	31.95
1.1047	25.11	27.74	1.1102	26.31	29.21	1.1157	27.42	30.59	1.1212	28.52	31.98
1.1048	25.14	27.77	1.1103	26.33	29.24	1.1158	27.44	30.62	1.1213	28.54	32.00
1.1049	25.16	27.79	1.1104	26.35	29.26	1.1159	27.46	30.64	1.1214	28.56	32.03
1.1050	25.18	27.82	1.1105	26.37	29.29	1.1160	27.48	30.67	1.1215	28.58	32.05
1.1051	25.20	27.85	1.1106	26.39	29.32	1.1161	27.50	30.69	1.1216	28.60	32.08
1.1052	25.22	27.87	1.1107	26.41	29.34	1.1162	27.52	30.72	1.1217	28.62	32.11
1.1053	25.24	27.90	1.1108	26.44	29.37	1.1163	27.54	30.75	1.1218	28.64	32.13
1.1054	25.27	27.93	1.1109	26.46	29.39	1.1164	27.56	30.77	1.1219	28.66	32.15
1.1055	25.29	27.96	1.1110	26.48	29.42	1.1165	27.58	30.80	1.1220	28.68	32.18
1.1056	25.31	27.98	1.1111	26.50	29.44	1.1166	27.60	30.82	1.1221	28.70	32.20
1.1057	25.33	28.00	1.1112	26.52	29.46	1.1167	27.62	30.85	1.1222	28.72	32.23
1.1058	25.35	28.03	1.1113	26.54	29.49	1.1168	27.64	30.87	1.1223	28.74	32.25
1.1059	25.38	28.06	1.1114	26.56	29.51	1.1169	27.66	30.89	1.1224	28.76	32.27
1.1060	25.40	28.09	1.1115	26.58	29.54	1.1170	27.68	30.92	1.1225	28.78	32.30
1.1061	25.42	28.12	1.1116	26.60	29.57	1.1171	27.70	30.94	1.1226	28.80	32.32
1.1062	25.44	28.14	1.1117	26.62	29.59	1.1172	27.72	30.97	1.1227	28.82	32.35
1.1063	25.46	28.17	1.1118	26.64	29.61	1.1173	27.74	31.00	1.1228	28.84	32.37
1.1064	25.48	28.19	1.1119	26.66	29.64	1.1174	27.76	31.02	1.1229	28.86	32.40
1.1065	25.50	28.22	1.1120	26.68	29.67	1.1175	27.78	31.05	1.1230	28.88	32.43
1.1066	25.52	28.25	1.1121	26.70	29.69	1.1176	27.80	31.07	1.1231	28.90	32.45
1.1067	25.54	28.27	1.1122	26.72	29.71	1.1177	27.82	31.09	1.1232	28.92	32.48
1.1068	25.57	28.30	1.1123	26.75	29.74	1.1178	27.84	31.12	1.1233	28.94	32.50
1.1069	25.59	28.32	1.1124	26.77	29.77	1.1179	27.86	31.15	1.1234	28.96	32.53
1.1070	25.61	28.35	1.1125	26.79	29.80	1.1180	27.88	31.18	1.1235	28.98	32.56
1.1071	25.63	28.38	1.1126	26.81	29.83	1.1181	27.90	31.20	1.1236	29.00	32.58
1.1072	25.65	28.40	1.1127	26.83	29.85	1.1182	27.92	31.23	1.1237	29.02	32.60
1.1073	25.67	28.43	1.1128	26.85	29.88	1.1183	27.94	31.25	1.1238	29.04	32.63
1.1074	25.69	28.45	1.1129	26.87	29.90	1.1184	27.96	31.27	1.1239	29.06	32.65
1.1075	25.71	28.48	1.1130	26.89	29.93	1.1185	27.98	31.30	1.1240	29.08	32.68
1.1076	25.73	28.51	1.1131	26.91	29.95	1.1186	28.00	31.32	1.1241	29.10	32.71
1.1077	25.75	28.53	1.1132	26.93	29.97	1.1187	28.02	31.35	1.1242	29.12	32.73
1.1078	25.78	28.56	1.1133	26.95	30.00	1.1188	28.04	31.37	1.1243	29.14	32.76
1.1079	25.80	28.58	1.1134	26.97	30.02	1.1189	28.07	31.40	1.1244	29.16	32.78
1.1080	25.82	28.61	1.1135	26.99	30.06	1.1190	28.09	31.43	1.1245	29.18	32.81
1.1081	25.84	28.64	1.1136	27.01	30.08	1.1191	28.11	31.45	1.1246	29.20	32.83
1.1082	25.86	28.66	1.1137	27.03	30.10	1.1192	28.13	31.48	1.1247	29.22	32.86
1.1083	25.89	28.69	1.1138	27.05	30.13	1.1193	28.15	31.51	1.1248	29.24	32.89
1.1084	25.91	28.72	1.1139	27.07	30.15	1.1194	28.17	31.53	1.1249	29.26	32.91
1.1085	25.93	28.75	1.1140	27.09	30.18	1.1195	28.19	31.56	1.1250	29.28	32.94
1.1086	25.96	28.78	1.1141	27.11	30.20	1.1196	28.21	31.59	1.1251	29.30	32.96
1.1087	25.98	28.80	1.1142	27.13	30.22	1.1197	28.23	31.61	1.1252	29.32	32.99
1.1088	26.01	28.83	1.1143	27.15	30.25	1.1198	28.25	31.63	1.1253	29.34	33.02
1.1089	26.03	28.86	1.1144	27.17	30.27	1.1199	28.27	31.65	1.1254	29.36	33.04
1.1090	26.05	28.89	1.1145	27.19	30.31	1.1200	28.28	31.68	1.1255	29.38	33.07
1.1091	26.07	28.92	1.1146	27.21	30.33	1.1201	28.30	31.70	1.1256	29.40	33.09
1.1092	26.09	28.94	1.1147	27.23	30.35	1.1202	28.32	31.73	1.1257	29.42	33.12
1.1093	26.12	28.97	1.1148	27.25	30.37	1.1203	28.34	31.75	1.1258	29.45	33.14
1.1094	26.14	29.00	1.1149	27.27	30.40	1.1204	28.36	31.78	1.1259	29.47	33.17



**Original Gravity of Beer Wort and its Determination.**—Following a long-established custom of the English excise, the duty on beer has been based on the specific gravity of the original wort, by which is meant the wort of the beer before any of its sugar has been lost by fermentation.

From the content of alcohol in the beer the sugar originally present in the wort may be calculated, assuming that the alcohol amounts to about half the sugar used up in fermentation.

Obtain the specific gravity of the beer, dealcoholized and made up to its original volume, as in the calculation of the extract. This is called the "extract gravity." Note the specific gravity corresponding to the alcohol found, i.e., the specific gravity of the distillate in the alcohol determination, when made up to the original volume, and subtract this from 1. The difference is known arbitrarily as the "degree of spirit indication."

From the table of Graham, Hofmann, and Redwood,\* given below, the "degrees of gravity lost" corresponding to the "spirit indication" are ascertained. This figure is added to the "extract gravity" to find the "original gravity of the wort."

SUGAR USED UP IN FERMENTATION.

Degrees of "Spirit In- dication"	0.0000	0.0001	0.0002	0.0003	0.0004	0.0005	0.0006	0.0007	0.0008	0.0009
0.000	.....	0.0003	0.0006	0.0009	0.0012	0.0015	0.0018	0.0021	0.0024	0.0027
.001	.0030	.0033	.0037	.0041	.0044	.0048	.0051	.0055	.0059	.0062
.002	.0066	.0070	.0074	.0078	.0082	.0086	.0090	.0094	.0098	.0102
.003	.0107	.0111	.0115	.0120	.0124	.0129	.0133	.0138	.0142	.0147
.004	.0151	.0155	.0160	.0164	.0168	.0173	.0177	.0182	.0186	.0191
.005	.0195	.0199	.0204	.0209	.0213	.0218	.0222	.0227	.0231	.0236
.006	.0241	.0245	.0250	.0255	.0260	.0264	.0269	.0274	.0278	.0283
.007	.0288	.0292	.0297	.0302	.0307	.0312	.0317	.0322	.0327	.0332
.008	.0337	.0343	.0348	.0354	.0359	.0365	.0370	.0375	.0380	.0386
.009	.0391	.0397	.0402	.0407	.0412	.0417	.0422	.0427	.0432	.0437
.010	.0442	.0447	.0451	.0456	.0460	.0465	.0470	.0475	.0480	.0485
.011	.0490	.0496	.0501	.0506	.0512	.0517	.0522	.0527	.0533	.0538
.012	.0543	.0549	.0554	.0559	.0564	.0569	.0574	.0579	.0584	.0589
.013	.0594	.0600	.0605	.0611	.0616	.0622	.0627	.0633	.0638	.0643
.014	.0648	.0654	.0659	.0665	.0671	.0676	.0682	.0687	.0693	.0699
.015	.0705	.0711	.0717	.0723	.0729	.0735	.0741	.0747	.0753	.0759

\* Report on Original Gravities, 1852; Allen's Coml. Org. Anal., 4 Ed., Vol. I, p. 151.



ACKERMANN'S TABLE FOR OBTAINING THE EXTRACT IN BEER FROM THE IMMERSION REFRACTOMETER  
READING OF THE BEER ( $R$ ) AND OF THE DISTILLATE OF THE BEER ( $R'$ ).\*

$R-R'$	Extract in 100 cc. Grams.	$R-R'$	Extract in 100 cc. Grams.	$R-R'$	Extract in 100 cc. Grams.	$R-R'$	Extract in 100 cc. Grams.	$R-R'$	Extract in 100 cc. Grams.	$R-R'$	Extract in 100 cc. Grams.	$R-R'$	Extract in 100 cc. Grams.	$R-R'$	Extract in 100 cc. Grams.
15.0	3.76	17.0	4.37	19.0	4.88	21.0	5.40	23.0	5.91	25.0	6.43	27.0	6.94	29.0	7.45
1	3.88	1	4.40	1	4.91	1	5.42	1	5.94	1	6.45	1	6.97	1	7.48
2	3.91	2	4.42	2	4.93	2	5.45	2	5.96	2	6.48	2	6.98	2	7.51
3	3.93	3	4.45	3	4.96	3	5.47	3	5.99	3	6.50	3	7.01	3	7.53
4	3.96	4	4.47	4	4.98	4	5.50	4	6.01	4	6.53	4	7.03	4	7.56
5	3.98	5	4.50	5	5.01	5	5.53	5	6.04	5	6.55	5	7.06	5	7.58
6	4.01	6	4.52	6	5.04	6	5.55	6	6.07	6	6.58	6	7.08	6	7.61
7	4.04	7	4.55	7	5.06	7	5.58	7	6.09	7	6.61	7	7.11	7	7.63
8	4.06	8	4.57	8	5.09	8	5.60	8	6.12	8	6.63	8	7.14	8	7.66
9	4.09	9	4.60	9	5.11	9	5.63	9	6.14	9	6.66	9	7.16	9	7.69
16.0	4.11	18.0	4.63	20.0	5.14	22.0	5.65	24.0	6.17	26.0	6.68	28.0	7.20	30.0	7.71
1	4.14	1	4.65	1	5.17	1	5.68	1	6.19	1	6.71	1	7.22	1	7.74
2	4.16	2	4.68	2	5.19	2	5.71	2	6.22	2	6.73	2	7.25	2	7.76
3	4.19	3	4.70	3	5.22	3	5.73	3	6.25	3	6.76	3	7.27	3	7.79
4	4.22	4	4.73	4	5.24	4	5.76	4	6.27	4	6.79	4	7.30	4	7.81
5	4.24	5	4.75	5	5.27	5	5.78	5	6.30	5	6.81	5	7.33	5	7.84
6	4.27	6	4.78	6	5.29	6	5.81	6	6.32	6	6.84	6	7.35	6	7.87
7	4.29	7	4.81	7	5.32	7	5.83	7	6.35	7	6.86	7	7.38	7	7.89
8	4.32	8	4.83	8	5.35	8	5.86	8	6.37	8	6.89	8	7.40	8	7.92
9	4.34	9	4.86	9	5.37	9	5.89	9	6.40	9	6.91	9	7.43	9	7.94

\* Zeits. gesamte Brauwesen, 29, 1906, p. 146.



*Example.*—Suppose the “extract gravity” is 1.0389 and the specific gravity of the alcoholic distillate is 0.9902, both at 15.6. Then  $1 - 0.9902 = 0.0098$ , the “degree of spirit indication.” From the above table the corresponding “degree of gravity lost” is found to be 0.0432.

$0.0432 + 1.0389 = 1.0821$ , the original gravity of the wort.

The calculation in the above simplified form is accurate for normal beer wherein the free acid present, expressed as acetic, does not exceed 0.1%. In case of beer that has developed free acid much in excess of the above limit, a correction should be added to the degrees of spirit indication. This correction, which in practice it is rarely necessary to apply except in extreme cases of old or sour beer, is calculated as follows:

If  $a$  represents the grams of free acid (as acetic) in 100 cc., then the correction to be added to the spirit indication  $= 0.0013a - 0.00014$ .

*Example.*—Supposing the “extract gravity” to be 1.0413, the specific gravity of the alcoholic distillate to be 0.9890, and the free acid as acetic to be 0.35%. Then  $1 - 0.9890 = 0.0110$ , the degree of spirit indication.

$0.35 \times 0.0013 - 0.00014 = 0.0003$ , correction to be added to the spirit indication.

$0.0110 + 0.0003 = 0.0113$ , corrected spirit indication.

From the above table the corresponding degrees of gravity lost are 0.0506:

$0.0506 + 1.0413 = 1.0919$ , the original gravity of the wort.

**Determination of Degree of Fermentation.**—This is calculated by the formula  $D = \frac{200A}{B}$ , in which  $D$  = degree of fermentation,  $A$  = per cent of alcohol by weight, and  $B$  = the original extract.

**Determination of Reducing Sugars.**—Dealcoholize 25 cc. of the beer and make up to 100 cc. Determine reducing sugars by the Defren-O’Sullivan or Munson-Walker method, and calculate as maltose.

**Determination of Dextrin.**—Dilute 50 cc. of the beer to 200 cc., hydrolize by heating in a boiling water-bath for  $2\frac{1}{2}$  hours with 20 cc. of hydrochloric acid (specific gravity 1.125), nearly neutralize the free acid with sodium hydroxide, make up to 300 cc., filter, and determine the dextrose by copper reduction. Multiply the amount of reducing sugars as maltose by 0.95, subtract this from the dextrose, and multiply the difference by 0.9, thus obtaining the dextrin in the beer.

**Determination of Glycerol.**—Proceed as directed on page 734 under wine. The milk of lime is added during evaporation after the carbon dioxide has been expelled. It is advisable that the filtrate, after being



evaporated to a syrupy consistency, be treated again with 5 cc. of absolute alcohol and two portions of 7.5 cc. each of absolute ether. If clear, continue as directed. If not clear, it is necessary to repeat the treatment with lime.

**Determination of Total, Fixed, and Volatile Acids.**—A measured volume of the beer, say 10 cc., is freed from carbon dioxide by bringing to boiling. It is then cooled and titrated with tenth-normal sodium hydroxide, using neutral litmus solution as an indicator. Each cubic centimeter of tenth-normal alkali is equivalent to 0.009 gram of lactic acid, in which the total acidity is usually expressed.

Fixed acid, also expressed as lactic, though small quantities of succinic, tannic, and malic acids are usually also present, is determined as follows: Dealcoholize a measured amount of the beer, say 10 cc., by evaporation to one-fourth its volume, dilute with water to the original volume, and titrate with tenth-normal alkali, as before.

Volatile acid is expressed as acetic, and is usually calculated by difference between total and fixed acid. Each cubic centimeter of tenth-normal alkali is the equivalent of 0.006 gram acetic acid.

**Determination of Proteins.**—Fifty cc. of the beer are first treated with 5 cc. of dilute sulphuric acid, and concentrated by boiling to syrupy consistency. Then proceed by the Gunning method, p. 58.  $N \times 6.25 =$  proteins.

**Determination of Phosphoric Acid.**—Unless the sample is very dark-colored, sufficiently close results can usually be obtained by direct titration of the beer itself with uranium acetate solution. For very accurate results the ash should be used. Prepare a solution of uranium acetate of such strength that 20 cc. will correspond to 0.1 gram  $P_2O_5$ . This solution is best standardized against pure, crystallized, uneffloresced, powdered hydrogen sodium phosphate, 10.085 grams of which are dissolved in water and made up to a liter. 50 cc. of this solution contains 0.1 gram phosphoric anhydride, if the salt is pure. If the solution is of proper strength, 50 cc. evaporated to dryness and ignited in a tared platinum dish should have an ash weighing 0.1874 gram. For preliminary trial about 35 grams of uranium acetate are dissolved in water, 25 cc. of glacial acetic acid, or its equivalent in weaker acid added, and the solution made up to a liter with water.

To standardize, 50 cc. of the standard phosphate solution prepared as above are heated to  $90^\circ$  or  $100^\circ$  C., and the uranium solution run in from a burette till the resulting precipitate of hydrogen uranium phos-



plate is complete. The end-point is determined by transferring a few drops of the solution to a porcelain plate, and touching with a drop of freshly prepared potassium ferrocyanide solution. When the slightest excess of uranium acetate has been added, a reddish-brown color is produced by the ferrocyanide. The uranium acetate solution is purposely made rather stronger than necessary at first, and by repeated trials is brought by dilution with water to the required strength (20 cc. equivalent to 50 cc. of the phosphate solution).

Fifty cc. of the beer are heated to 90° or 100° C. and titrated with the uranium acetate solution under the same conditions and in precisely the same manner as when standardizing that solution. Each cubic centimeter of the uranium acetate corresponds to 0.01% of  $P_2O_5$ .

For the phosphoric acid determination in the ash, 50 cc. of the beer are incinerated in the regular manner, and the ash moistened with concentrated hydrochloric acid. The acid is then evaporated off on the water-bath, after which the ash is boiled with 50 cc. of distilled water, and titrated with the standard uranium solution.

**Determination of Carbon Dioxide.\***—In the case of beer and other carbonated drinks put up in corked bottles, the carbon dioxide may be readily determined by piercing the cork with a metal champagne tap, which is connected by a flexible tube, first with a safety flask and then with an absorption apparatus somewhat after the style of that used in the determination of carbon dioxide in baking powder, the liberated carbon dioxide being absorbed for weighing in a concentrated solution of potassium hydroxide contained in a tared Liebig bulb. The beer-bottle thus connected is immersed in a vessel of water, which is heated over a gas-flame, after all the carbon dioxide that will escape spontaneously has been allowed to do so. Before weighing the absorbed carbon dioxide, the beer-bottle is replaced by a soda-lime tube, and a current of air drawn through the tubes.

Beer and ale put up in bottles having patent metallic or rubber stoppers cannot thus be treated. In this case a measured quantity, say 200 cc., of the sample is transferred as quickly as possible to a large flask provided with an outlet-tube having a glass stopper, this being connected up with the safety-flask and absorption-tubes. In this case heat may be directly, though cautiously, applied to the flask containing the beer by means of a gas-flame, after all the carbon dioxide has gone over that will

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 95; Bul. 107 (rev.), p. 92.



do so spontaneously. Exactly the same apparatus as that shown in Fig. 71 may be used to advantage for determination of carbon dioxide in beer, except that a larger distilling-flask should be used in the case of beer.

**Detection of Bitter Principles.**—Elaborate schemes have been worked out for the systematic treatment of beer and ale for bitter principles. Nearly all of these are complicated and somewhat unsatisfactory. The presence of alkaloids in malt liquors, deliberately introduced during the process of manufacture, is now so rare that the analyst need seldom look for them, except in cases of suspected poisoning, when the scheme of Dragendorf\* or of Otto-Stas should be employed. While it is somewhat difficult to positively identify the various alkaloids, it is usually easy to prove their absence in clear solutions, if on treatment with either of the general alkaloidal reagents, Mayer's solution, or iodine in potassium iodide, no precipitate is formed.

It is comparatively easy to prove the mere presence or absence of hop substitutes. The bitter principle of hops is readily soluble in ether, when a sample of the beer evaporated to syrupy consistency is extracted therewith, while the bitters of quassia and aloes, common hop substitutes, are insoluble in ether. Though many varieties of bitters might be employed that are soluble in ether, the *absence* of a bitter taste from the ether extract is evidence of the absence of hops.

The most marked difference analytically between hops and their substitutes in malt liquors lies in the fact that the bitter principle of hops is completely precipitated therefrom by treatment of the beer with lead acetate (either basic or neutral), leaving no bitter taste in the filtrate after concentration, while if any of the hop substitutes are present, the concentrated filtrate from the lead acetate treatment will have a bitter taste. The excess of lead should be removed from the filtrate, before concentration and tasting, by treatment with hydrogen sulphide. If the residue from the ether or chloroform extraction of the concentrated filtrate from a beer after treatment with lead acetate be found to be bitter, there is positive evidence that a foreign substitute has been employed.

The following are characteristic reactions that may help to identify some of the common hop substitutes: †

*Quassiin* is readily soluble by chloroform from acid solution. If a residue containing quassiin be moistened with a weak alcoholic solution

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\* Gerichtlich-Chemische Ermittlung von Giften, St. Petersburg, 1876.

† Allen, Analyst, 12, 1887, p. 107.



of ferric chloride and gently heated, a marked mahogany-brown coloration is produced.

On treatment of quassia with bromine and sodium hydroxide or ammonia, a bright-yellow color is shown.

*Chiretta* is readily dissolved by ether from its aqueous solution. Its ether residue, when treated with bromine and ammonia, gives a straw color, slowly changing to a dull purple-brown. This is not true of its chloroform residue, so that it is not to be mistaken for quassia (Allen).

*Gentian Bitter* may be extracted by treatment of the acid liquor with chloroform. When the residue containing gentian bitter is treated with concentrated sulphuric acid, in the cold, no color is produced, but on warming gently a carmine-red color is shown; if further treated with ferric chloride solution, a green-brown color is formed.

*Aloes*.—This substance is separated from beer by treating the dried residue from 200 cc. of the beer with warm ammonia, filtering, cooling, and treating the filtrate with hydrochloric acid. The resin of aloes is precipitated and collected on a filter. It is insoluble in cold water, ether, chloroform, or petroleum ether, but is soluble in alcohol. It has a very characteristic odor, which serves to identify it. The hot-water solution gives a curdy precipitate on treatment with lead acetate.

*Capsicin* is extracted by treatment of the acid liquor with chloroform. It is recognizable by its sharp, pungent taste.

**Detection of Arsenic.**—*By the Marsh Method.*—Measure 100 cc. of the beer (freed from carbon dioxide by agitation) into a seven-inch porcelain evaporating-dish, add 20 cc. pure concentrated nitric acid, and 3 cc. pure concentrated sulphuric acid, and cautiously heat till vigorous chemical action sets in, accompanied by frothing and swelling of the beer. Turn the flame low or remove it altogether, and stir vigorously till the frothing ceases, after which the liquid may be boiled freely. At this stage transfer to a large casserole, and continue the boiling till nearly all the nitric acid is driven off. Then, holding the casserole by the handle, continue the heating till the mass chars and the fumes of sulphuric acid are given off, giving the casserole a rotary motion to prevent sputtering. The residue should be reduced to a dry, black, pulverulent char soon after the sulphuric acid fumes begin to come off freely. If still liquid, pieces of filter-paper should be stirred in while still heating, till the residue is dry, avoiding an excess of paper.

Cool, add 50 cc. of water, and remove the masses of char from the sides



of the dish by the stirring-rod. Heat to boiling and filter. Use the filtrate for the Marsh apparatus, adding it gradually.

The arsenic mirror may be weighed in the usual manner, if of sufficient size.

*Reinsch's Test.\**—Two hundred cc. of the beer are acidified with 1 cc. of pure, concentrated, arsenic-free hydrochloric acid, and evaporated to half its volume. 15 cc. more of hydrochloric acid are then added, and a piece of pure burnished copper foil half an inch long and a quarter of an inch wide is immersed in the liquid and kept in it for an hour while simmering, replacing from time to time the water lost by evaporation. If after the lapse of an hour the copper still remains bright, no arsenic is present.

If the copper shows a deposit, remove, wash with water, alcohol, and ether, and dry. Then place the copper in a subliming-tube, and heat over a low flame. Tetrahedral crystals, apparent under the microscope, show the presence of arsenic. Blackening of the copper does not in itself prove arsenic.

**Detection and Determination of Preservatives.** —See Chapter XVIII. Sulphurous acid may be determined by direct titration, as in the case of wine.

#### MALT EXTRACT.

True malt extract is a syrupy fluid having a specific gravity of from 1.3 to 1.6, and made up in accordance with the following directions of the 1880 Pharmacopœia: Upon 100 parts of coarsely powdered malt contained in a suitable vessel, pour 100 parts of water, and macerate for six hours. Then add 400 parts of water, heated to about 30° C. and digest for an hour at a temperature not exceeding 55° C. Strain the mixture with strong pressure. Finally, by means of a water-bath or vacuum apparatus, at a temperature not exceeding 55° C., evaporate the strained liquid rapidly to the consistence of thick honey.

Keep the product in well-closed vessels in a cool place.

Such an extract has a residue of at least 70%, consisting chiefly of maltose, and contains about 2% of diastase. It should furthermore be capable of converting its own weight of starch at 55° C. in less than ten minutes.

The following are analyses of three samples of pure malt extract: †

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\* Jour. Soc., Chem. Ind., 20, p. 646.

† Penn. Dept. of Agric. An. Rep., 1898, p. 85.



	Specific Gravity.	Alcohol.	Extract.	Free Acids as Acetic.	Volatile Acids.	Albuminoids.	Maltose.	Dextrin.	Ash.	Phosphoric Acid.	Diastatic Action.
A	1.387	0	72.31	0.231	0.033	3.329	62.52	5.25	1.21	0.483	Complete in less than 5 min.
B	1.421	0	76.65	0.275	0.021	3.116	65.41	6.94	1.19	0.556	" " " " 10 "
C	1.498	0	79.81	0.386	0.053	4.872	61.32	12.39	1.23	0.428	" " " " 5 "

There are on the market many so-called malt extracts widely advertised for their tonic and medicinal virtues, having the taste and consistency of beer or ale. In fact they are virtually beer, differing therefrom mainly in respect to price. Such "malt extracts" have no diastase, and their value as nutrients depends almost entirely on their sugar content.

Harrington\* has analyzed twenty-one of the best known of these alleged malt extracts, the maximum, minimum, and mean results of his analyses being as follows:

	Specific Gravity.	Alcohol.	Total Residue.	Ash.
Maximum.....	1.0555	7.13	13.63	0.53
Minimum.....	1.0149	0.74	5.13	0.20
Mean.....	.....	3.94	8.78	

None of them contained any diastase, and several were preserved with salicylic acid.

#### DISTILLED LIQUORS.

These beverages differ from those hitherto considered, by reason of their high alcoholic content and low extract or residue. Indeed, when first distilled they are entirely without residue, but from long storage in casks, they absorb certain extractives from the wood, that impart more or less flavor as well as color.

When any fermented alcoholic infusion is subjected to distillation under ordinary circumstances, a distillate results which consists of a mixture with water of a large number of alcohols, chief among which is ethyl alcohol. The high boiling alcohols—amyl, butyl, propyl, etc., with their esters—exist in the distillate in small amount, constituting what is known as fusel oil. The various distilled liquors of commerce

\* Boston Medical and Surgical Journal, Dec. 31, 1896.



are made by just such a process of distillation, the product varying widely in flavor and character with the basis from which it was distilled.

The so-called pot-still (the old-fashioned copper still and worm) is well adapted for the production of potable spirits such as whiskey, brandy, gin, and rum, as these products should contain the congeneric substances which give the liquors their special character; it is not, however, suited for the manufacture of pure alcohol, because repeated distillation would be required for purification.

Now, however, by the use of improved apparatus, such as the Coffey still, involving the principle of fractional condensation, it is possible to obtain what is known as "silent spirit," or ethyl alcohol, free from fusel oil. With proper appurtenances for rectifying, one can now obtain 95% alcohol by two distillations.

**Standards for Spirits.**—The following are the standards adopted by the Joint Committee of the Association of Official Agricultural Chemists and the Association of State and National Food and Dairy Departments:

*Distilled Spirit* is the distillate obtained from a fermented mash of cereals, molasses, sugars, fruits, or other fermentable substance, and contains all the volatile flavors, essential oils, and other substances derived directly from the material used, and the higher alcohols, ethers, acids, and other volatile bodies congeneric with ethyl alcohol produced during fermentation, which are carried over at the ordinary temperature of distillation, and the principal part of which are higher alcohols estimated as amylic.

*Alcohol, Cologne Spirit, Neutral Spirit, Velvet Spirit, or Silent Spirit*, is distilled spirit from which all, or practically all, of its constituents except ethyl alcohol and water, are separated, and contains not less than 94.9% (189.8 proof) by volume of ethyl alcohol.

**Composition of Fusel Oil.**—Fusel oil varies considerably in composition with the source from which it is derived. Amyl alcohol, being in all cases its chief constituent, is frequently known commercially as fusel oil. The alcohols found in fusel oil with their formulas, specific gravity, and boiling-points are as follows:

	Formula.	Specific Gravity.	Boiling-point.
Ethyl alcohol.....	$C_2H_5OH$	.794	78.4° C.
Propyl ".....	$C_3H_7OH$	.820	97° C.
Butyl ".....	$C_4H_9OH$	.803	115° C.
Amyl ".....	$C_5H_{11}OH$	.811	130° C.
Hexyl ".....	$C_6H_{13}OH$		



The following acids have been found in fusel oil, usually combined with the alcohols to form compound ethers:

Acetic. . . . .	$\text{HC}_2\text{H}_3\text{O}_2$	Caproic. . . . .	$\text{HC}_6\text{H}_{11}\text{O}_2$
Propionic. . . . .	$\text{HC}_3\text{H}_5\text{O}_2$	Ænanthylic. . . . .	$\text{HC}_7\text{H}_{13}\text{O}_2$
Butyric. . . . .	$\text{HC}_4\text{H}_7\text{O}_2$	Caprylic. . . . .	$\text{HC}_8\text{H}_{15}\text{O}_2$
Valerianic. . . . .	$\text{HC}_5\text{H}_9\text{O}_2$	Pelargonic. . . . .	$\text{HC}_9\text{H}_{17}\text{O}_2$

**Aging.**—Freshly distilled liquors all contain notable quantities of substances, which render them harsh and unfit for use, but during aging, they become in several years mellow and palatable. The chemical changes which take place during aging are discussed under whiskey.

### WHISKEY.

**Process of Manufacture.**—Whiskey is the liquor resulting from the distillation of a fermented infusion of grain, the process being carried out in a pot-still, or some other form of still, constructed so that the resulting liquor contains not only the alcohol, but also the greater part of the congeneric substances which are vaporized with the alcohol. The fermented infusion known as the “mash” is obtained by steeping in water the starch-containing material, usually barley, rye, corn (maize), or oats mixed with malt, and subjecting the mixture to the action of the diastase contained in the malt, in much the same manner as the mashing process in the brewing of beer, except that for whiskey the process of saccharous fermentation is carried further, with a view to obtaining a maximum yield of maltose and a minimum of dextrin. Yeast is afterwards added, and alcoholic fermentation allowed to proceed with proper precautions.

The fermented wort from whatever source obtained is subjected to distillation, purposely avoiding rectification or separation of the fusel oil and other congeneric substances which are valuable as flavors. The product of the first distillation is called “low wines,” and is redistilled; the product of the second distillation is commonly divided into three fractions, of which the middle portion, or “clean spirit” is retained for the whiskey, while the first (“foreshots”) and the last fraction (“faints”) are mixed with the next portion of low wine to be redistilled. If the whiskey is too high in alcohol, it is diluted to the proper strength.

As new whiskey is crude and harsh in taste, it is subjected to “aging,” or storing in casks for a number of years. The aging process softens and refines the flavor, but recent investigations have proved that this



is not due, as formerly believed, to transformation of fusel oil into esters although the esters increase in amount during aging, as do also the acids—especially the volatile acids—the aldehydes, and the furfural. As a matter of fact, the percentage of fusel oil increases instead of diminishes on aging, due to the evaporation of water and, in a lesser degree, of alcohol through the wood; the actual amount, however, remains practically the same as at the start (see table, p. 769). When first distilled, whiskey is perfectly colorless, but during the aging it extracts more or less color and some flavor from the casks in which it is stored. This color is especially pronounced in American whiskies, owing to the prevailing custom of charring the inside of the cask. Its flavor varies considerably with the nature of the grain used in its preparation.

**U. S. Rulings.**—The following decision of President Roosevelt, based on an opinion of Attorney-General Bonaparte, was promulgated by Secretary Wilson, April 11, 1907:

“Straight whiskey will be labeled as such.

“A mixture or two or more straight whiskies will be labeled ‘blended whiskey,’ or ‘whiskies.’

“A mixture of straight whiskey and ethyl alcohol, provided that there is a sufficient amount of straight whiskey to make it genuinely a ‘mixture,’ will be labeled as compound of, or compounded with, pure grain distillate.

“Imitation whiskey will be labeled as such.”

This decision was overruled by President Taft, whose opinion is the basis of Food Inspection Decision No. 113 (Feb. 16, 1910), signed by the secretaries of the Treasury, Agriculture, and Commerce and Labor. The chief points of this decision follow:

“All unmixed distilled spirits from grain, colored and flavored with harmless color and flavor, in the customary ways, either by the charred barrel process, or by the addition of caramel and harmless flavor, if of potable strength and not less than 80° proof, are entitled to the name whiskey without qualification.

“Whiskies of the same or different kinds (i.e., straight, rectified, redistilled, and neutral spirits whiskies) are like substances and mixtures of such, with or without harmless color or flavor used for purposes of coloring and flavoring only, are blends.

“Potable alcoholic distillates from sources other than grain (e.g., cane, fruit, or vegetables), colored and flavored, are imitations and mixtures of such with grain distillate are compounds.



"A distillate of grain (e.g., corn) flavored to simulate a whiskey of another kind (e.g., rye) is an imitation of that whiskey."

Attorney-General Wickersham (F. I. D. No. 127) has further decided that the name "Canadian Club whiskey" is distinctive and it is therefore unnecessary to place the word "blend" on the label.

**Joint Standards.**—The following are the standards of the Joint Committee of the A. O. A. C. and the A. S. N. F. D. D.:

*New Whiskey* is the properly distilled spirit from the properly prepared and properly fermented mash of malted grain, or of grain the starch of which has been hydrolyzed by malt; it has an alcoholic strength corresponding to the excise laws of the various countries in which it is produced, and contains in 100 liters of proof spirit not less than 100 grams of the various substances other than ethyl alcohol derived from the grain from which it is made, and of those produced during fermentation, the principal part of which consists of higher alcohols estimated as amylic.

*Whiskey (Potable Whiskey)* is new whiskey which has been stored in wood not less than four years without any artificial heat save that which may be imparted by warming the storehouse to the usual temperature, and contains in 100 liters of proof spirit not less than 200 grams of the substance found in new whiskey, save as they are changed or eliminated by storage, and of those produced as secondary bodies during aging; and, in addition thereto, the substances extracted from the casks in which it has been stored. It contains, when prepared for consumption as permitted by the regulations of the Bureau of Internal Revenue, not less than 45% by volume of ethyl alcohol, and, if no statement is made concerning its alcoholic strength, it contains not less than 50% of ethyl alcohol by volume, as prescribed by law.

*Rye Whiskey* is a whiskey in the manufacture of which rye, either in a malted condition or with sufficient barley or rye malt to hydrolyze the starch, is the only grain used.

*Bourbon Whiskey* is a whiskey made in Kentucky from a mash of Indian corn and rye, and barley malt, of which Indian corn forms more than 50%.

*Corn Whiskey* is whiskey made from malted Indian corn or of Indian corn the starch of which has been hydrolyzed by barley malt.

*Blended Whiskey* is a mixture of two or more whiskeys.

*Scotch Whiskey* is whiskey made in Scotland solely from barley malt, in the drying of which peat has been used. It contains in 100 liters of



proof spirit not less than 150 grams of the various substances prescribed for whiskey exclusive of those extracted from the cask.

*Irish Whiskey* is whiskey made in Ireland, and conforms in the proportions of its various ingredients to Scotch whiskey, save that it may be made of the same materials as prescribed for whiskey, and the malt used is not dried over peat.

**Composition.**—Whiskey consists chiefly of alcohol and water, with relatively small amounts of fusel oil, acids, esters, aldehydes, and furfural. Its extract, derived mainly from the casks in which it is stored, should consist only of small amounts of tannin, sugar, and coloring matter.

*British Whiskies.*—Scotch and Irish whiskies are aged in uncharred barrels, hence they are of a lighter color than the American product. Scotch whiskey is further characterized by its smoky taste, due to the peat over which it is dried. The following analyses by Vasey \* illustrate the composition of Scotch and Irish whiskey of different ages, of neutral spirits used in compounding ("blending") and adulterating, and of the compounded liquors:

	Grams per 100 Liters of Absolute Alcohol.				
	Volatile Acids.	Esters.	Aldehydes.	Furfural.	Fusel Oil.
Pot-still Scotch whiskey, 8 years old . . . . .	48.0	89.7	14.2	4.0	200.0
Pot-still Scotch whiskey, 25 years old . . . . .	64.8	125.1	66.1	5.4	180.0
Irish whiskey, new . . . . .	20.9	7.7	6.5	0.4	174.0
Irish whiskey, 7 years old . . . . .	41.8	20.9	11.2	3.4	204.0
Neutral spirit for "blending" . . . . .	8.4	23.8	4.9	0.4	trace
"Blended" Scotch . . . . .	39.1	106.8	14.3	3.5	108.5
"Scotch," probably all neutral spirits . . . . .	16.8	8.2	10.0	none	none

It will be noted that the congeneric substances in whiskey increase on aging, although in the case of fusel oil this apparent increase is doubtless due merely to concentration dependent on evaporation. The sample of neutral spirits contained only small amounts of the congeneric substances, while the "blended" whiskies were deficient in most of these substances.

*American Whiskies.*—These have a deeper color than the British whiskies (due to the charred barrel) and a rich fruity flavor without the suggestion of smoke.

\* Potable Spirits, pp. 82, 83, and 87.



In the table below are given analyses by Shepard\* of fourteen leading brands, including both rye and bourbon, varying in age from four to eight years; also of two samples of neutral spirits used for compounding and adulterating.

A summary of the results obtained by Crampton and Tolman† in the analysis of fourteen brands of rye and seventeen brands of bourbon whiskey at differing stages of aging appear in the table on p. 769. The barrels were kept in U. S. bonded warehouses during aging, and samples

	Age, Years.	Alcohol Per Cent by Volume.	Grams per 100 Liters of the Liquor.								
			Extract.	Ash.	Acids.			Esters.	Aldehydes.	Furfural.	Fusel Oil.
					Total.	Fixed.	Volatile.				
Rye .....	8	50.1	189.8	7.7	92.0	12.8	79.2	81.8	17.5	3.0	84.9
Bourbon .....	8	50.1	181.5	7.3	68.4	9.3	59.1	60.7	17.5	3.2	102.6
Standard .....	5	50.0	160.4	7.2	66.8	10.2	56.6	55.9	10.0	2.4	160.4
Hand-made sour mash.	4½	49.8	162.1	7.4	67.1	10.2	56.9	74.8	12.0	2.6	130.9
	4	50.0	148.5	7.3	62.4	7.5	54.9	55.9	15.0	2.6	152.0
Hand-made sour mash.	4	49.9	132.7	5.8	49.2	7.5	41.7	39.6	8.0	1.0	107.4
	6	50.4	138.6	6.4	74.8	8.6	66.2	61.6	10.5	1.3	192.7
Hand-made sour mash.	6	50	153.7	9.7	58.8	9.9	48.9	69.6	14.0	0.7	137.1
“ “ “	7	50	180.0	10.0	74.4	9.9	64.5	70.8	12.5	2.5	117.0
Bourbon .....	5½	49.9	129.3	5.1	60.9	7.2	53.7	49.3	9.5	0.8	141.7
Special reserve .....	7	49.8	212.0	8.0	93.0	13.5	79.5	94.0	22.5	5.0	119.5
Sour mash .....	5	50.1	124.5	7.2	58.2	7.2	51.0	64.0	9.5	0.5	95.3
	7½	49.8	177.2	7.3	66.5	9.0	57.5	76.6	10.0	1.7	193.6
	4	50.1	139.4	6.5	50.3	6.3	44.0	54.6	7.5	1.5	152.0
Neutral spirits.....		95.6	10.3	0.9	7.5	1.2	6.3	15.4	2.5	.....	30.0
“ “ .....		94.4	3.2	0.0	6.3	1.4	4.9	64.2	11.0	.....	39.6

were withdrawn at intervals of a year for eight years. As the minimum figures for certain constituents are abnormal, the next to the minimum figures are also given. It will be noted that during the first few years there was a marked increase in actual amounts of all the constituents determined, except fusel oil, over and above that due to concentration, but after three or four years the acids and esters do not materially change. The rye whiskeys contained larger amounts of solids, acids, esters, etc., than the bourbons, but this was attributed to the fact that heated warehouses are used for rye, and unheated for bourbon whiskey. The authors state that the characteristic aroma of American whiskey,

\* The Constants of Whiskey, S. Dak. Food and Dairy Commission, March, 1906.

† Jour. Am. Chem. Soc., 30, 1908, p. 98.



## SUMMARY OF ANALYSES OF AMERICAN WHISKIES OF DIFFERENT AGES

		Proof.	Grams per 100 Liters of 100 Proof Spirits.						
			Color	Extract.	Acids.	Esters.	Alde- hydes.	Fur- fural.	Fusel Oil.
RYE WHISKEY.									
New:	Average ...	101.2	0.0	13.3	4.4	16.3	5.4	1.0	90.4
	Maximum .	102.0	0.0	30.0	72.0	21.8	15.0	1.9	161.8
	Minimum *	100.0	0.0	5.0	12.0	4.3	0.7	trace	{ 61.8 43.7
One year old:	Average ...	102.5	8.8	119.7	46.6	37.0	7.0	1.8	111.5
	Maximum .	104.0	13.8	171.0	60.5	64.8	15.5	3.3	194.0
	Minimum *	101.0	{ 7.2 6.6	{ 93.0 92.0	{ 31.1 5.8	{ 6.8 6.8	{ 2.8	{ 0.4	{ 80.4 66.4
Two years old:	Average ...	104.9	11.6	144.7	51.9	54.0	10.5	2.2	112.4
	Maximum .	109.0	16.7	199.0	75.6	75.1	18.7	5.7	214.0
	Minimum *	100.0	{ 8.8 8.6	{ 121.0 94.0	{ 44.3 11.0	{ 41.5 31.2	{ 5.4	{ 0.7	{ 83.4 82.2
Three years old:	Average ...	107.7	13.2	171.4	62.7	61.5	12.5	1.5	112.7
	Maximum .	112.0	18.3	224.0	81.8	79.8	20.8	6.1	202.0
	Minimum *	104.0	{ 11.4 10.1	{ 145.0 119.0	{ 52.3 16.4	{ 47.6 34.3	{ 6.5	{ 0.7	{ 79.0 60.0
Four years old:	Average ...	111.2	14.0	185.0	65.9	69.3	13.9	2.8	125.1
	Maximum .	118.0	18.9	238.0	83.8	89.1	22.1	6.7	203.5
	Minimum *	105.0	{ 11.6 11.3	{ 156.0 153.0	{ 58.6 17.3	{ 57.7 36.3	{ 6.4	{ 0.7	{ 83.8 67.8
Eight years old:	Average ...	123.8	18.6	256.0	82.9	89.1	16.0	3.4	154.2
	Maximum .	132.0	24.2	339.0	112.0	126.6	26.5	9.2	280.3
	Minimum *	112.0	{ 13.8 13.7	{ 214.0 200.0	{ 73.7 31.7	{ 68.4 40.9	{ 7.9	{ 0.8	{ 109.0 107.1
BOURBON WHISKEY.									
New:	Average ...	101.0	0.0	26.5	10.0	18.4	3.2	0.7	100.9
	Maximum .	104.0	0.0	161.0	29.1	53.2	7.9	2.0	171.3
	Minimum *	100.0	0.0	4.0	12.0	13.0	1.0	trace	{ 71.3 42.0
One year old:	Average ...	101.8	7.1	99.6	41.1	28.6	5.8	1.6	110.1
	Maximum .	103.0	10.9	193.0	55.3	55.9	8.6	7.9	173.4
	Minimum *	100.0	{ 5.4 4.6	{ 61.0 54.0	{ 24.7 7.2	{ 17.2 10.4	{ 2.7	{ trace	{ 58.0 42.8
Two years old:	Average ...	102.2	8.6	126.8	45.6	40.0	8.4	1.6	108.9
	Maximum .	104.0	11.8	214.0	61.7	59.8	12.0	9.1	197.1
	Minimum *	100.0	{ 6.9 5.7	{ 81.0 78.0	{ 25.5 23.3	{ 24.4 11.2	{ 5.9	{ 0.4	{ 86.2 42.8
Three years old:	Average ...	103.0	10.0	149.3	54.3	48.1	10.5	1.7	112.4
	Maximum .	106.0	13.8	245.0	64.8	73.0	22.1	9.5	221.8
	Minimum.*	100.0	{ 8.9 7.0	{ 95.0 90.0	{ 38.4 32.1	{ 27.2 12.1	{ 5.9	{ 0.6	{ 88.0 43.5
Four years old:	Average ...	104.3	10.8	151.9	58.4	53.5	11.0	1.9	123.9
	Maximum .	108.0	14.8	249.0	73.0	80.6	22.0	9.6	237.1
	Minimum *	100.0	{ 8.6 7.4	{ 101.0 92.0	{ 40.4 40.4	{ 28.2 13.8	{ 6.9	{ 0.8	{ 95.0 43.5
Eight years old:	Average ...	111.1	14.2	210.3	76.4	65.6	12.9	2.1	143.5
	Maximum .	124.0	20.9	326.0	91.4	93.6	28.8	10.0	241.8
	Minimum *	102.0	{ 12.3 10.5	{ 152.0 141.0	{ 64.1 53.7	{ 37.7 22.1	{ 8.7	{ 1.0	{ 110.0 47.6

\* Minimum and next to the minimum.



also the oily appearance and the "body" (solids), are due to the charred barrels.

Thirty-seven samples of whiskey, purchased by the glass from various Massachusetts saloons, were examined by the Massachusetts State Board of Health in 1894, with the following results:

	Per Cent Alcohol by Weight.	Per Cent Extract.
Maximum.....	45.96	1.68
Minimum. ....	30.70	0.08
Mean.....	36.51	0.50

Seven of these samples had an excess of tannic acid, three had no tannic acid at all, and two or three had insoluble residues.

**Adulteration of Whiskey.**—Imitation whiskey is often concocted by diluting alcohol or neutral spirit to the proper strength, coloring with caramel, sometimes adding for body prune juice, and adding for flavor certain essential oils, such as oil of wintergreen, and artificial fruit essences, such as cœnanthic and pelargonic ethers. As a rule, a small amount of pure whiskey is mixed with the artificial to give it flavor.

What has long been known as "blended whiskey" is either an imitation pure and simple, or a compound of whiskey and neutral spirits, artificially colored and flavored. According to the U. S. decisions, the term "blended whiskey" is restricted to a mixture of different kinds of grain distillate, colored and flavored.

Among Fleischman's recipes for "blended" whiskey is the following, which he claims to be the very lowest grade:

Spirits .....	32 gallons
Water .....	16 "
Caramel .....	4 ounces
Beading oil .....	1 ounce

"Beading oil" is prepared by mixing 48 ounces oil of sweet almonds with 12 ounces C. P. sulphuric acid, neutralizing with ammonia, adding double the volume of proof spirits, and distilling. This preparation is so called because it is largely used for putting an artificial bead on cheap liquors.

A little creosote is sometimes added to give a burnt taste in sem-



blance of Scotch whiskey. Pungent materials such as cayenne pepper are said to be used as adulterants, but no record is known of any substance being used more injurious than the alcohol. Sugar is a frequent adulterant.

Some doubt exists as to the injurious effects of fusel oil on the system.

The following analyses by Ladd\* show the composition of neutral spirits, and imitation whiskey consisting of neutral spirits diluted with water, colored with caramel and flavored:

	Alcohol Per Cent by Volume.	Grams per 100 Liters of the Liquor.								
		Extract.	Ash.	Acids.			Esters.	Aldehydes.	Furfural.	Fusel Oil.
				Total.	Fixed.	Volatile.				
Neutral spirits .....	94.0	2.4	0.0	7.2	0.0	7.2	26.4	6.0	trace	28.0
Imitation whiskey, rye .....	40.1	366.4†	4.4	43.2	9.0	34.2	3.5	trace	0.4	
“ “ malt .....	45.8	854.0†	2.0	20.4	3.0	17.4	14.0	trace	1.0	37.0
“ “ rye .....	45.0	456.0†	5.5	9.6	3.0	6.6	5.2	trace	0.8	42.3

† Includes caramel color.

### BRANDY AND COGNAC.

Brandy is the product of the distillation of fermented grape juice or wine. In a broader sense the term brandy is sometimes applied to liquor distilled from the juices of other fruits, such as apples, peaches, cherries, etc. The finest grades of brandy, such as pure cognac and armagnac (named from towns in France in which they were originally distilled), are made from choice white wine by the use of pot stills, and naturally command a high price. Brandy of a lower grade is distilled from the cheaper wines, and sometimes from the fermented marc, or refuse, of the grape, as well as from the lees and “scrapings” of the casks. The best brandies are sometimes rectified by a second distillation. Like whiskey, the fresh brandy is colorless, and would so remain if stored in glass or stone. The color is due to the wooden casks in which it is stored. Brandy consists of nearly pure alcohol and water, having a characteristic flavor, differing somewhat according to the nature and quality of the wine from which it was prepared. The chief flavor of pure cognac is due to ænanthic ether.

\* N. Dak. Agric. Exp. Sta. Rep., 1906, Part II, p. 145.



**Composition.**—Vasey \* gives the following analyses of cognac and of brandy adulterated with neutral spirits:

	Cognac Ten Years Old.	Brandy Mixed with Neutral Spirits.			
Volatile acids.....	74.5	79.4 grams per 100 liters of absolute alcohol			
Esters.....	109.3	32.4	"	"	"
Aldehydes.....	16.6	7.4	"	"	"
Furfural.....	1.7	0.6	"	"	"
Fusel oil.....	124.2	49.0	"	"	"

Thirty-seven samples of brandy, collected from Massachusetts bar-rooms in 1894 and examined by the State Board of Health, showed the following results:

	Per Cent Alcohol by Weight.	Per Cent Extract.
Maximum.....	50.70	3.00
Minimum.....	21.30	0.10
Mean.....	40.54	0.93

Three of these samples were artificially prepared mixtures of alcohol and water, one showed the presence of cloves, five contained tannin in excess, nine were excessively acid, and two had insoluble residues.

**Joint Standards.**—The following are the standards of the A. O. A. C. and the A. S. N. F. D. D.:

*New Brandy* is a properly distilled spirit made from wine, and contains in 100 liters of proof spirit not less than 100 grams of the volatile flavors, oils, and other substances, derived from the material from which it is made, and of the substances congeneric with ethyl alcohol produced during fermentation and carried over at the ordinary temperatures of distillation, the principal part of which consists of the higher alcohols estimated as amylic.

*Brandy (Potable Brandy)* is new brandy stored in wood for not less than four years without any artificial heat save that which may be imparted by warming the storehouse to the usual temperature, and contains in 100 liters of proof spirit not less than 150 grams of the substances found in new brandy, save as they are changed or eliminated by storage, and of those produced as secondary bodies during aging;

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\* Analysis of Potable Spirits, p. 20



and, in addition thereto, the substances extracted from the casks in which it has been stored. It contains, when prepared for consumption, as permitted by the regulations of the Bureau of Internal Revenue, not less than 45% by volume of ethyl alcohol, and, if no statement is made concerning its alcoholic strength, it contains not less than 50% by volume of ethyl alcohol as prescribed by law.

*Cognac, Cognac Brandy*, is brandy produced in the departments of the Charente and Charente Inferieure, France, from wine produced in those departments.

**Adulteration of Brandy.**—Much of the brandy sold on the market is a compound or imitation, having for its basis alcohol reduced to the requisite strength, flavored either by the admixture of real brandy, or by various preparations such, for example, as syrup of raisins, prune juice, rum, acetic ether, cœnanthic ether, infusion of green walnut-hulls, infusion of bitter almond shells, catechu, balsam of Tolu, etc.

Fleischmann gives the following recipe for artificial brandy of the cheapest grade:

Spirits .....	45 gallons
Coloring (caramel) .....	6 ounces
Cognac oil . . . . .	$\frac{1}{8}$ ounce

“Cognac oil” is made up of melted cocoanut oil 16 ounces, sulphuric acid 8 ounces, alcohol 16 ounces, mixed and distilled.

While commercial brandy often fails to meet the pharmacopœial requirements, and may contain any of the above flavoring materials, not one sample has been found among the many examined by the Massachusetts Board of Health during upwards of twenty years containing a more injurious ingredient than alcohol.

Genuine new brandy may be “aged” or “improved” for immediate use, according to Duplais, by adding to 100 liters the following:

Old rum.....	2.00 liters
Old kirsch*.....	1.75 “
Infusion of walnut-hulls.....	.75 liter
Syrup of raisins. . . . .	2.00 liters

The addition of sugar and caramel to brandy is very common. The

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\* Brandy distilled from cherry wine.



lack of flavor resulting from the employment of "silent spirit," or from watering the product, may be compensated for by the employment of so-called cognac essences sold for the purpose, containing mixtures of the aromatic compounds named above.

### RUM.

Rum is the liquor distilled from fermented molasses or cane juice, or from the scum and other waste juices from the manufacture of raw sugar. The molasses wort is mixed with the residue from a previous fermentation and allowed to ferment for a number of days, after which it is distilled twice and stored in wood for a long time, to remove the disagreeable odor, which in the new product is especially marked. The characteristic flavor of old rum is due to a mixture of butyric and acetic ether, principally the former. Pineapples and guavas are often put in the still to impart a fruity flavor. The best varieties of rum come from Jamaica and Vera Cruz.

**Composition.**—The following analysis of rum is by Vasey:\*

Volatile acids.....	28.0	grams	per 100	liters of absolute alcohol
Esters.....	399.0	"	"	"
Aldehydes.....	8.4	"	"	"
Furfural.....	2.8	"	"	"
Fusel oil.....	90.6	"	"	"

Thirty-nine samples of rum, sold at retail in Massachusetts in 1894, were examined by the State Board of Health with the following results:

	Per Cent Alcohol by Weight.	Per Cent Extract.
Maximum.....	42.9	3.93
Minimum. ....	24.7	0.04
Mean.....	37.1	0.51

Of these, two samples were new rum, and several were entirely artificial.

**Joint Standards.**—The following are the joint standards of the A. O. A. C. and the A. S. N. F. D. D.:

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\* Analysis of Potable Spirits, p. 85.



*New Rum* is properly distilled spirit made from the properly fermented clean, sound juice of the sugar cane, the clean, sound massacuite made therefrom, clean, sound molasses from the massecuite, or any sound clean intermediate product save sugar, and contains in 100 liters of proof spirit not less than 100 grams of the volatile flavors, oils, and other substances derived from the materials of which it is made, and of the substances congeneric with the ethyl alcohol produced during fermentation, which are carried over at the ordinary temperatures of distillation, the principal part of which is higher alcohols estimated as amylic.

*Rum (Potable Rum)* is new rum stored not less than four years in wood without any artificial heat save that which may be imparted by warming the storehouse to the usual temperature, and contains in 100 liters of proof spirit not less than 175 grams of the substances found in new rum, save as they are changed or eliminated by storage, and of those produced as secondary bodies, during aging; and, in addition thereto, the substances extracted from the casks. It contains, when prepared for consumption as permitted by the regulations of the Bureau of Internal Revenue, not less than 45% by volume of ethyl alcohol, and if no statement is made concerning its alcoholic strength, it contains not less than 50% by volume of ethyl alcohol as prescribed by law.

More or less factitious rum is sold on the market, made up of alcohol diluted to the right strength, colored with caramel, and flavored by the addition of "rum essence." Prune juice is sometimes added.

Fleischman gives the following recipe for low-grade artificial rum:

Spirits. ....	40	gallons
New England rum. ....	5	"
Prune juice. ....	$\frac{1}{2}$	gallon
Caramel. ....	12	ounces
Rum essence. ....	8	"

The "rum essence" is made up by distilling 32 ounces of a mixture of 2 ounces black oxide of manganese, 4 ounces pyroligneous acid, 32 ounces alcohol, and 4 ounces sulphuric acid. To this is added 32 ounces of acetic ether, 8 ounces of butyric ether, 16 ounces saffron extract, and  $\frac{1}{8}$  ounce oil of birch.



## GIN.

Gin is an alcoholic liquor, flavored with the volatile oil of juniper and sometimes with other aromatic substances, such as coriander, grains of paradise, anise, cardamom, orange-peel, and fennel. The choicest variety is known as Schiedam schnapps, named from the town of Schiedam in Holland, where there are upwards of 200 distilleries devoted to the manufacture of gin. The mash used for this variety is fermented by yeast from malted barley and rye, after which it is distilled and redistilled in pot stills with juniper berries and sometimes hops.

Juniper berries, to which the most characteristic flavor of gin is due, are dark blue in color, and possess a pungent taste. They grow on the slender evergreen shrub *Juniperus communis*. Gin differs from the other distilled liquors by being water-white. To this end it is kept in glass and not in wood.

Much of the gin of commerce is made by redistilling corn or grain whiskey with oil of juniper, and frequently one or several of the above-named flavoring materials. Sugar is often added, and sometimes in the cheaper productions oil of turpentine is substituted for juniper oil.

**Composition.**—The following analysis of unsweetened gin is by Vasey:\*

Volatile acids.....	0.0	grams	per 100	liters of absolute alcohol
Esters.....	37.3	"	"	"
Aldehydes.....	1.8	"	"	"
Furfural.....	0.0	"	"	"
Fusel oil.....	44.6	"	"	"

Thirty-three samples of gin, purchased in Massachusetts saloons and analyzed by the State Board of Health in 1894, gave the following results in per cent of alcohol by weight: Maximum 42.5, minimum 29.5, mean 38.2.

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\* Analysis of Potable Spirits, p. 85.



## METHODS OF ANALYSIS OF DISTILLED LIQUORS.

Specific gravity and alcohol are determined as described on pp. 686-706. The following methods with the exception of the qualitative test for fusel oil, Mitchell's method, and McGill's opalescence test are those of the A. O. A. C.

**Determination of Extract.**—Weigh or measure (at 15.6° C.) 100 cc. of the sample, evaporate nearly to dryness on the water-bath, then transfer to a water-oven, and dry at the temperature of boiling water for 2½ hours.

**Determination of Acids.**—Titrate 100 cc. (or 50 cc. diluted to 100 cc. if the sample is dark in color) with tenth-normal alkali, using phenolphthalein as indicator. 1 cc. of tenth-normal alkali is equal to 0.006 of acetic acid.

**Determination of Esters.**—Dilute 200 cc. of the sample with 25 cc. of water and distil slowly into a graduated 200-cc. flask until nearly filled to the mark. Complete the volume, shake, and use aliquot portions for the determination of esters, aldehydes, and furfural.

Exactly neutralize 50 cc. of the distillate with tenth-normal alkali, using phenolphthalein as indicator, and add from 25 to 50 cc. of the tenth-normal alkali in excess of that required for neutralization. Either boil for one hour with a reflux condenser, or allow to stand overnight in a stoppered flask, and heat with a tube condenser for one-half hour below the boiling-point. Cool, and titrate with tenth-normal acid, using phenolphthalein as indicator. Multiply the number of cc. of tenth-normal alkali used in the saponification by 0.0088, thus obtaining the grams of esters calculated as ethyl acetate.

**Determination of Aldehydes.**—1. *Reagents.*—(a) *Alcohol Free from Aldehydes.*—Prepare by first redistilling the ordinary 95% alcohol over caustic soda or potash, then add from 2 to 3 grams per liter of *m*-phenylenediamine hydrochloride, digest at ordinary temperature for several days (or reflux on the steam-bath for several hours), and then distil slowly, rejecting the first 100 cc. and the last 200 cc.

(b) *Sulphite-fuchsin Solution.*—Dissolve 0.50 gram of pure fuchsin in 500 cc. of water, then add 5 grams of SO<sub>2</sub> dissolved in water, make up to a liter, and allow to stand until colorless. Prepare this solution in small quantities, as it retains its strength for only a very few days.



(c) *Standard Acetic Aldehyde Solution*.—Prepare according to the directions of Vasey\* as follows: Grind aldehyde ammonia in a mortar with ether, and decant the ether. Repeat this operation several times, then dry the purified salt in a current of air and finally in a vacuum over sulphuric acid. Dissolve 1.386 grams of this purified ammonium aldehyde in 50 cc. of 95% alcohol, to this add 22.7 cc. of normal alcoholic sulphuric acid, then make up to 100 cc. and add 0.8 cc. to compensate for the volume of the ammonium sulphate precipitate. Allow this to stand over night and filter. This solution contains 1 gram of acetic aldehyde in 100 cc. and will retain its strength.

The standard found most convenient for use is 2 cc. of this strong aldehyde solution diluted to 100 cc. with 50% alcohol by volume. One cc. of this solution is equal to 0.0002 gram of acetic aldehyde. This solution should be made up fresh every day or so, as it loses its strength.

2. *Process*.—Determine the aldehyde in the distillate prepared for esters. Dilute from 5 to 10 cc. of the distillate to 50 cc. with aldehyde-free alcohol (50% by volume), add 25 cc. of the fuchsin solution, and allow to stand for fifteen minutes at 15° C. The solutions and the reagents should be at 15° C. before they are mixed. Prepare standards of known strength in the same way.

**Determination of Furfural.**—*Standard Furfural Solution*.—Dissolve 1 gram of redistilled furfural in 100 cc. of 95% alcohol. This strong solution will keep. Standards are made by diluting 1 cc. of this solution to 100 cc. with 50% by volume alcohol. One cc. of this solution contains 0.0001 gram furfural.

*Process*.—Dilute from 10 to 20 cc. of the distillate, prepared as described under esters, to 50 cc. with furfural-free alcohol (50% by volume). To this add 2 cc. of colorless anilin and 0.5 cc. of hydrochloric acid (specific gravity 1.125), and keep for fifteen minutes in a water-bath at about 15° C. Prepare standards of known strength in the same way.

**Detection of Fusel Oil.**—In the process of dealcoholizing a liquor by evaporation in an open dish over the water-bath, one may readily detect fusel oil, if present, by its harsh and nauseating odor, if the nose is applied just at the moment when the last traces of alcohol are going off. At this stage any considerable trace of fusel oil will be especially apparent by the effect on the throat of the one who smells it, causing

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\* Analysis of Potable Spirits, p. 30.



an uncontrollable desire to cough. Other ways of applying the odor test consist in pouring a small portion of the spirit into the hand, and allowing it to evaporate slowly therefrom, or in rinsing out a warm glass with the liquor, observing the odor in each case.

Goebel suggests the following test, based on the detection of the volatile acids: Agitate about 30 cc. of the liquor with 2 or 3 cc. of a dilute solution of potassium hydroxide; evaporate over the water-bath to the volume of 2 or 3 cc., cool, and to the residue add 5 or 6 cc. of concentrated sulphuric acid. If fusel oil be present, the characteristic odors of valerianic and butyric acids will be apparent.

**Determination of Fusel Oil.**—*Allen-Marquardt Method.*—Add to 100 cc. of whiskey 20 cc. of half-normal sodium hydroxide, and saponify the mixture by boiling for one hour under a reflux condenser.\* Connect the flasks with a distilling apparatus, distil 90 cc., add 25 cc. of water, and continue the distillation until an additional 25 cc. is collected.

Approximately saturate the distillate with finely ground sodium chloride, and add a saturated solution of sodium chloride until the specific gravity is 1.10.

Extract this salt solution four times with carbon tetrachloride,† using 40, 30, 20, and 10 cc. respectively, and wash the carbon tetrachloride three times with 50-cc. portions of a saturated solution of sodium chloride, and twice with saturated solution of sodium sulphate. Then transfer the carbon tetrachloride to a flask containing 5 cc. of concentrated sulphuric acid, 45 cc. of water, and 5 grams of potassium bichromate, and boil for eight hours under a reflux condenser.

Add 30 cc. of water, and distil until only about 20 cc. remain; add 80 cc. of water, and distil until but 5 cc. are left. Neutralize the distillate to methyl orange, and titrate with sodium hydroxide, using phenolphthalein as indicator. One cc. of tenth-normal sodium hydroxide is equivalent to 0.0088 gram of amyl alcohol.

Rubber stoppers can be used in the saponification and first distillation, but corks covered with tinfoil must be used in the oxidation and second distillation. Corks and tinfoil must be renewed frequently.

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\* Or 100 cc. of the liquor may be mixed with 20 cc. of half-normal sodium hydroxide, allowed to stand overnight at room temperature, and distilled directly.

† Purify 5 liters of carbon tetrachloride by boiling for several hours under a reflux condenser with 200 cc. of sulphuric acid and 25 grams of potassium bichromate in 200 cc. of water; separate from the oxidizing mixture by distillation, and redistil over barium carbonate.



*Tolman and Hillyer's Modification of the Allen-Marquardt Method.*—Proceed with the Allen-Marquardt method to the point where the carbon tetrachloride solution of the higher alcohols is ready to be oxidized. Add 50 cc. of a solution of 200 grams of pulverized potassium bichromate in 1800 cc. of water and 200 cc. of concentrated sulphuric acid, very carefully measured with pipette or burette, and start the eight-hour oxidation. Take great care to prevent any isolation of spots of bichromate on the flask during the oxidation. Decomposition of the bichromate from overheating can best be prevented by slow boiling over several thicknesses of asbestos board. After the oxidation is complete, separate the bichromate solution from the carbon tetrachloride in a separatory funnel, care being taken to wash the carbon tetrachloride free from bichromate. Make up the bichromate solution to 500 cc. Place 200 cc. of this solution in a liter flask, add 20 cc. of concentrated hydrochloric acid, 100 cc. of potassium iodide solution (1:1), and 50 cc. of approximately three-fourths normal thiosulphate not standardized. Make this last addition by means of a burette. (If a high content of fusel oil is present, 50 cc. of thiosulphate may be excessive and a smaller amount should be used, the same quantity being added to the sample and to the blank.) Run blanks containing exactly the same amount of reagents with each series, and treat them in the same way, starting them at the point where the carbon tetrachloride is washed with sodium chloride. The titration of this blank, to which has been added exactly the same amount of three-fourths normal thiosulphate, gives the value of the bichromate solution. The difference in cubic centimeters of tenth-normal thiosulphate used in titrating the blank and the samples gives the amount of bichromate reduced by the higher alcohols. This difference in cubic centimeters of tenth-normal thiosulphate multiplied by the factor 0.001773 gives grams of higher alcohols present.

*Mitchell and Smith Method.*\*—This is more rapid than the Allen-Marquardt method and gives more nearly the true amount of fusel oil.

Saponify, distil, shake with sodium chloride, and extract with carbon tetrachloride, as in the Allen-Marquardt method. To the carbon tetrachloride extract, contained in the separatory funnel, add 10 cc. of potassium hydroxide solution (1:1). Cool the mixture in ice-water to approximately 0° C. Similarly cool 100 cc. of a solution of potassium permanganate solution (20 grams to the liter), accurately measured in

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\* A. O. A. C. Proc. 1908, U. S. Dept. of Agric., Bur. of Chem., Bul. 122, p. 199.



a flask. To the contents of the separatory funnel add the bulk of the permanganate solution, but without rinsing, retaining the residue to be added at a later stage. Remove the mixture from the bath, and shake vigorously for five minutes; set aside for thirty minutes, with occasional shaking, permitting the liquid to warm to room temperature (20 to 25° C.)

Accurately measure into a liter Erlenmeyer flask 100 cc. of a solution of hydrogen peroxide slightly (about 2%) stronger than the permanganate solution, acidulate with 100 cc. of an approximately 25% sulphuric acid solution, and slowly add the contents of the separatory funnel with constant shaking, keeping the acid solution constantly in excess. Rinse the separatory funnel and the flask containing the residue of permanganate with water and add to the peroxide solution. Finally titrate the excess of hydrogen peroxide with standard potassium permanganate solution (10 grams to the liter).

Run a blank determination, using the same amounts of the stronger permanganate, potassium hydroxide, hydrogen peroxide, and sulphuric acid solutions, and titrating the residual peroxide with the standard potassium permanganate as before.

The difference in the amounts of permanganate consumed, in grams, times 0.696, gives the amount of amyl alcohol.

**Detection of Methyl Alcohol.**—*Leach and Lythgoe Immersion Refractometer Method.*\*—Determine at 20° C. the refraction of the distillate obtained in the determination of alcohol by the immersion refractometer. If on reference to the table the refraction shows the percentage of alcohol agreeing with that obtained from the specific gravity, it may be safely assumed that no methyl alcohol is present. If, however, there is an appreciable amount of methyl alcohol, the low refractometer reading will at once indicate the fact. If the absence from the solution of other refractive substances than water and the alcohols is assured, this qualitative test by difference in refraction is conclusive.

The addition of methyl to ethyl alcohol decreases the refraction in direct proportion to the amount present; hence the quantitative calculation is readily made by interpolation in the table, using the figures for pure ethyl and methyl alcohol of the same alcoholic strength as the sample.

*Example.*—Suppose the distillate made up to the original volume of the measured portion taken for the alcohol determination has a

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\* Jour. Am. Chem. Soc., 27, 1905, p. 964.



specific gravity of 0.9736, corresponding to 18.38% alcohol by weight, and has a refraction of 35.8 at 20° C. by the immersion refractometer; by interpolation in the refractometer table the readings of ethyl and methyl alcohol corresponding to 18.38% alcohol are 47.2 and 25.4, respectively, the difference being 21.8;  $47.2 - 35.8 = 11.4$ ;  $(11.4 \div 21.8) 100 = 52.3$ , showing that 52.3 of the alcohol present is methyl alcohol.

SCALE READINGS ON ZEISS IMMERSION REFRACTOMETER AT 20° C.,  
CORRESPONDING TO EACH PER CENT BY WEIGHT OF METHYL AND  
ETHYL ALCOHOLS.

Per Cent Alcohol by Weight.	Scale Readings.		Per Cent Alcohol by Weight.	Scale Readings.		Per Cent Alcohol by Weight.	Scale Readings.		Per Cent Alcohol by Weight.	Scale Readings.	
	Methyl Al- cohol.	Ethyl Al- cohol.		Methyl Al- cohol.	Ethyl Al- cohol.		Methyl Al- cohol.	Ethyl Al- cohol.		Methyl Al- cohol.	Ethyl Al- cohol.
0	14.5	14.5	26	30.3	61.9	51	39.7	91.1	76	29.0	101.0
1	14.8	16.0	27	30.9	63.7	52	39.6	91.8	77	28.3	100.9
2	15.4	17.6	28	31.6	65.5	53	39.6	92.4	78	27.6	100.9
3	16.0	19.1	29	32.2	67.2	54	39.5	93.0	79	26.8	100.8
4	16.6	20.7	30	32.8	69.0	55	39.4	93.6	80	26.0	100.7
5	17.2	22.3	31	33.5	70.4	56	39.2	94.1	81	25.1	100.6
6	17.8	24.1	32	34.1	71.7	57	39.0	94.7	82	24.3	100.5
7	18.4	25.9	33	34.7	73.1	58	38.6	95.2	83	23.6	100.4
8	19.0	27.8	34	35.2	74.4	59	38.3	95.7	84	22.8	100.3
9	19.6	29.6	35	35.8	75.8	60	37.9	96.2	85	21.8	100.1
10	20.2	31.4	36	36.3	76.9	61	37.5	96.7	86	20.8	99.8
11	20.8	33.2	37	36.8	78.0	62	37.0	97.1	87	19.7	99.5
12	21.4	35.0	38	37.3	79.1	63	36.5	97.5	88	18.6	99.2
13	22.0	36.9	39	37.7	80.2	64	36.0	98.0	89	17.3	98.9
14	22.6	38.7	40	38.1	81.3	65	35.5	98.3	90	16.1	98.6
15	23.2	40.5	41	38.4	82.3	66	35.0	98.7	91	14.9	98.3
16	23.9	42.5	42	38.8	83.3	67	34.5	99.1	92	13.7	97.8
17	24.5	44.5	43	39.2	84.2	68	34.0	99.4	93	12.4	97.2
18	25.2	46.5	44	39.3	85.2	69	33.5	99.7	94	11.0	96.4
19	25.8	48.5	45	39.4	86.2	70	33.0	100.0	95	9.6	95.7
20	26.5	50.5	46	39.5	87.0	71	32.3	100.2	96	8.2	94.9
21	27.1	52.4	47	39.6	87.8	72	31.7	100.4	97	6.7	94.0
22	27.8	54.3	48	39.7	88.7	73	31.1	100.6	98	5.1	93.0
23	28.4	56.3	49	39.8	89.5	74	30.4	100.8	99	3.5	92.0
24	29.1	58.2	50	39.8	90.3	75	29.7	101.0	100	2.0	91.0
25	29.7	60.1									

*Trillat Method.\**—To 50 cc. add 50 cc. of water and 8 grams of lime, and fractionally distil by the aid of Glinksy bulb tubes. Dilute the

\* A. Trillat, Analyst, 24, 1899, pp. 13, 211-212.



first 15 cc. of the distillate to 150 cc., mix with 15 grams of potassium bichromate and 70 cc. of sulphuric acid (1:5), and allow to stand for one hour with occasional shaking.

Distil, reject the first 25 cc., and collect 100 cc. Mix 50 cc. of the distillate with 1 cc. of rectified dimethyl-anilin, transfer to a stout, tightly-stoppered flask, and keep on bath at 70 to 80° C. for three hours with occasional shaking. Make distinctly alkaline with sodium hydroxide, and distil the excess of dimethyl-anilin, stopping the distillation when 25 cc. have passed over.

Acidify the residue in the flask with acetic acid, shake, and test a few cc. by adding four or five drops of water with lead dioxide in suspension (1 gram in 100 cc.). If methyl alcohol be present, a blue coloration occurs which is increased by boiling.

Note.—Ethyl alcohol thus treated yields a blue coloration, changing immediately to green, afterwards to yellow, and becoming colorless when boiled.

*Riche and Bardy Methoa.\**—The following method for the detection of methyl alcohol in commercial spirit of wine depends on the formation of methyl-anilin violet:

Place 10 cc. of the sample, previously rectified over potassium carbonate if necessary, in a small flask with 15 grams of iodine and 2 grams of red phosphorus. Keep in ice-water for from ten to fifteen minutes until action has ceased. Distil on a water-bath the methyl and ethyl iodides formed into about 30 cc. of water. Wash with dilute alkali to eliminate free iodine. Separate the heavy oily liquid which settles, and transfer to a flask containing 5 cc. of anilin. The flask should be placed in cold water, in case the action should be violent, or, if necessary, the reaction may be stimulated by gently warming the flask. After one hour boil the product with water, and add about 20 cc. of a 15% solution of soda; when the bases rise to the top as an oily layer, fill the flask up to the neck with water, and draw them off with a pipette. Oxidize 1 cc. of the oily liquid by adding 10 grams of a mixture of 100 parts of clean sand, 2 of common salt, and 3 of cupric nitrate; mix thoroughly, introduce into a glass tube, and heat to 90° C. for eight or ten hours. Exhaust the product with warm alcohol, filter, and make up with alcohol to 100 cc. If the sample of spirits be pure, the liquid is of a red tint, but in the presence of 1% of methyl alcohol, it has a distinct violet shade; with

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\* Allen's Commercial Organic Analysis, 3d ed., I, p. 80.



2.5% the shade is very distinct, and still more so with 5%. To detect more minute quantities of methyl alcohol, dilute 5 cc. of the colored liquid to 100 cc. with water, and dilute 5 cc. of this again to 400 cc. Heat the liquid thus obtained in porcelain, and immerse a fragment of white merino (free from sulphur) in it for half an hour. If the alcohol be pure, the wool will remain white, but if methylated, the fiber will become violet, the depth of tint giving a fair approximate indication of the proportion of methyl alcohol present.

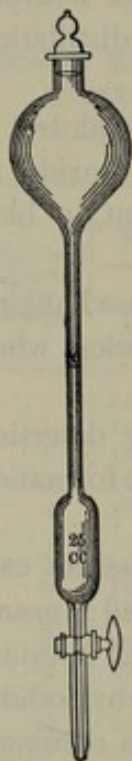


FIG. 116.—Separatory Funnel for Detection of Caramel.

**Detection of Caramel.**—*Crampton and Simon's Method.*\*—Evaporate 50 cc. of the liquor nearly but not quite to dryness in an evaporating-dish on the water-bath. Wash with water into a 50-cc. graduated glass-stoppered flask, add 25 cc. of absolute alcohol, and fill to the mark with water. Shake, and transfer 25 cc. of the solution to a separatory funnel of the type presented in Fig. 116, the stem of which terminates in a 25-cc. graduated bulb pipette, provided with a stop-cock as shown.

Add 50 cc. of ether, and shake carefully at intervals during half an hour. After complete separation, make up the lower aqueous layer with water to the 25-cc. mark, which may be done by siphoning it in through a rubber tube from an elevated flask, controlling the supply by the stop-cock. Shake the separatory funnel, and again allow the layers to separate, draw off the aqueous layer, and compare with the color of the original liquor. Express the amount of color removed as per cent of the total amount. Ether will readily dissolve the natural color due to oakwood (mainly flavescein), while caramel is insoluble in ether; hence uncolored liquors are partially decolorized by this treatment, while those colored with caramel show little change.

*Amthor Test, Modified by Lasché.*†—Add 10 cc. of paraldehyde to 5 cc. of the sample contained in a test tube and shake. Add absolute alcohol, a few drops at a time, shaking after each addition until the mixture becomes clear. Allow to stand. Turbidity after ten minutes is an indication of caramel.

\* Jour. Am. Chem. Soc., 22 1900, p. 810.

† The Brewer Distiller, May, 1903.



**Determination of Water-insoluble Color in Whiskies.**—Evaporate 50 cc. of the sample just to dryness. Take up with cold water, using approximately 15 cc., filter, and wash until the filtrate amounts to nearly 25 cc. To this filtrate add 25 cc. of absolute alcohol or 26.3 cc. of 95% by volume alcohol, and make up to 50 cc. by the addition of water. Mix thoroughly and compare in a colorimeter with the original material. Calculate the per cent of color insoluble in water from these readings.

**Determination of Color Insoluble in Amyl Alcohol.**—*Modified Marsh Test.*—Evaporate 50 cc. of the whiskey just to dryness on the steam-bath. Add 26.3 cc. of 95% alcohol to dissolve the residue. Transfer to a 50-cc. flask and make up to volume with water to obtain a uniform alcohol concentration. Place 25 cc. of this solution in a separatory funnel, and add 20 cc. of the Marsh reagent, shaking lightly so as not to form an emulsion. (This reagent consists of 100 cc. of pure amyl alcohol, 3 cc. of syrupy phosphoric acid, and 3 cc. of water; shake before using.) Allow the layers to separate, and repeat this shaking and standing twice again. After the layers have clearly separated, draw off the lower or watery layer which contains the caramel into a 25-cc. cylinder, and make up to volume with 50% by volume alcohol. Compare this solution in a colorimeter with the untreated 25 cc. Calculate the result of this reading to the per cent of color insoluble in amyl alcohol.

**Opalescence in Diluted Alcohol Distillate.**—McGill \* has shown that in the case of liquors made from thoroughly rectified grain spirit, there is little or no opalescence produced when the alcoholic distillate (i.e., that used in determining the alcohol) is diluted with an equal volume of water, while in the case of liquors distilled from alcoholic infusions without rectification, the opalescence is marked. He ascribes the opalescence to the presence of minute amounts of volatile oils present in wine marc, grains, and other sources of these liquors, soluble in strong, but insoluble in dilute alcohol. Whether due to this or to the separation of minute traces of fusel oil on dilution, the presence or absence of turbidity certainly furnishes a rough distinguishing test, indicating in some cases the exclusive use of rectified spirit.

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\* Bul. 27, Canadian Inland Rev. Dept.



## LIQUEURS AND CORDIALS.

These are manufactured beverages, usually high in alcohol and sugar, flavored with a wide variety of aromatic herbs or essences, and often strongly colored. Red colors most frequently used for this purpose are cochineal, cudbear, and red sandal and Brazil woods; for yellow colors, caramel and saffron-yellow are employed; for blue, indigo; and for green, chlorophyll and malachite green.

Some of the oldest of the liqueurs, such as chartreuse and bénédictine, derive their names from certain monasteries of Europe, in which they have been made for many years.

*Absinthe* is one of the best-known cordials, made by redistilling 40% alcohol in which wormwood, anise, sweet flag, and marjoram leaves have been macerated. Sometimes coriander and fennel are also used. It is highly intoxicating.

*Curaçao* is made by distilling dilute spirits in which Curaçao orange-peel,\* cinnamon and often other spices have been soaked, and by adding sugar to the resulting liqueur.

De Brevans gives the following recipe for curaçoa:

Rasped skins of.....	18 or 20 oranges
Cinnamon.....	4 grams
Mace.....	2 "
Alcohol (85%).....	5 liters
White sugar.....	1750 grams

Macerate for fourteen days, distill without rectification, and color with caramel.

*Angostura* owes its flavor to Angostura bark and various spices.

*Maraschino* had originally for its basis the fermented juice of the sour Italian cherry, to which honey was added. It is more commonly made by distilling a mixture in alcohol of ripe wild cherries, raspberries, cherry leaves, peach nuts, and orris. Finally sugar is added.

*Chartreuse* and *Bénédictine* contain much sugar, and are flavored with the volatile oils of angelica, hyssop, nutmeg, and peppermint.

*Noyau*, or *Crème de Noyau*, is a preparation distilled from brandy, bitter almonds, mace and nutmeg. Sugar and coloring matter, usually pink, are added to the final product.

*Crème de Menthe*, according to De Brevans, is made by distilling a mixture of

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\* This is a very rare and highly-prized orange, growing in the island of Curacao.



Peppermint.....	600 grams
Balm.....	40 "
Sage.....	10 "
Cinnamon.....	20 "
Orris root.....	10 "
Ginger.....	15 "
Alcohol (80%) .....	5030 cc.

producing finally 10 liters of the liquor, after 3750 grams of white sugar have been introduced.

The better grades of crème de menthe were formerly colored with an alcoholic solution of chlorophyll, derived by macerating bruised green leaves of various plants with alcohol, but at present, coal-tar dyes are used. Frequently the desired shade is secured by mixing a green (e.g., Light Green S.F.), a blue-green (e.g., Malachite Green), or a blue (e.g., Indigo Carmine) with a yellow color.

The following analyses, due to König, show the chemical composition of the best-known cordials:

	Specific Gravity.	Alcohol by Volume.	Alcohol by Weight.	Extract.	Cane Sugar.	Other Extractives.	Ash.
Absinthe.....	0.9116	58.93	.....	0.18	.....	0.32	.....
Bénédictine.....	1.0709	52	38.5	36.00	32.57	3.43	0.043
Ginger.....	1.0481	47.5	36.0	27.79	25.92	1.87	0.141
Crème de menthe.....	1.0447	48.0	36.5	28.28	27.63	0.65	0.068
Anisette de Bordeaux...	1.0847	42.0	30.7	34.82	37.44	0.38	0.040
Curaçoa.....	1.0300	55.0	42.5	28.60	28.50	0.10	0.040
Kümmel.....	1.0830	33.9	24.8	32.02	31.18	0.84	0.058
Angostura.....	0.9540	49.7	.....	5.85	4.16	1.69	.....
Chartreuse.....	1.0799	43.18	.....	36.11	34.35	1.76	.....

**Analysis of Cordials and Liqueurs.**—The character of the essences and flavoring principles used in these beverages is so widely varied that no regular systematic plan for identifying them can be made applicable to all cases. The senses of smell and taste are most useful, both when applied directly to the liqueur itself and to the dry extract, for suggestions as to the main ingredients employed. Coloring-matters, sugars, acids, and alcohol are determined as with other liquors, except that in the case of alcohol all volatile oils must first be separated out by treatment with magnesia, as directed for alcohol in lemon extract. Presence of volatile oils is shown, if on treatment of a few cubic centimeters of the sample in a test-tube with water a precipitate is formed.

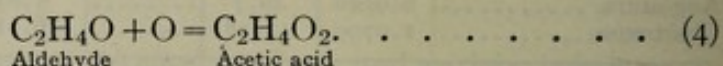
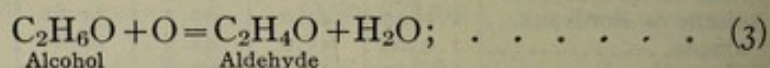
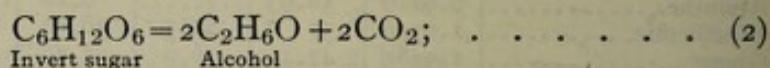
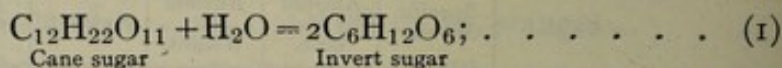


## CHAPTER XVI

### VINEGAR.

TRUE vinegar is the product of the acetic fermentation of an alcoholic liquid under the influence of the organism *Mycoderma aceti*, existing in the "mother-of-vinegar." While vinegar may be made directly from a dilute solution of pure alcohol, it is commonly obtained from fruit juice, wine, or other saccharine liquid that has first undergone alcoholic fermentation.

Of the following equations (1) and (2) illustrate the processes of inversion and alcoholic fermentation respectively, while (3) and (4) show the double process of acetic fermentation, wherein the alcohol is oxidized, first to acetaldehyde and finally to acetic acid:



**Varieties.**—The principal varieties of vinegar are the following: *Cider vinegar*, *wine vinegar*, *malt or beer vinegar*, *spirit or distilled vinegar*, *glucose vinegar*, *sugar or molasses vinegar*, and *wood vinegar or diluted pyroligneous acid*, the four last being frequently used as adulterants of the others.

Cider and distilled vinegar are the principal varieties used in the United States and Canada, malt vinegar is the common variety in Great Britain and wine vinegar on the continent.

In addition to the acetic acid, its chief active principle, vinegar contains small amounts of other substances present in the raw material or formed during the alcoholic and acetic fermentations. To the former class belong the acids of the apple and grape and to the latter, succinic and lactic acids.



Alcohol, in greater or less amount depending on the completeness of acetification, is present in all true vinegar. Reducing sugar and other carbohydrates in minute amount, glycerol, coloring matters, aromatic ethers, and mineral salts occur in all kinds but distilled and wood vinegar. Other minor constituents of cider vinegar are pentosans,\* furfural,† and acetyl-methylcarbinol.‡ Distilled vinegar contains only traces of solids. Wood vinegar contains considerable amounts of formic acid; cider vinegar and malt vinegar none or only traces.§

**Manufacture of Vinegar.**—Cider and wine vinegar were formerly made almost entirely by the slow process of cask fermentation, the fruit juice being allowed to undergo both alcoholic and acetic fermentation in barrels with open bung-holes in a warm cellar, or exposed to the sun. Two or three years are required for this process. Sometimes fresh cider or wine is added to the barrels at regular intervals of two or three weeks, thus causing a series of progressive fermentations. The acetic fermentation is hastened by adding old vinegar, or mother-of-vinegar. While farmers and some manufacturers still continue to use the slow process, quick or "generator" vinegar processes are now much used not only for malt, beer, and spirit vinegar but also for cider and wine vinegar.

There are two types of generators: (1) *Tank Generators*, provided with false bottoms, containing beech shavings, birch twigs, corn cobs, or other woody material, previously saturated with old vinegar, through which the alcoholic liquor percolates and is brought in contact with a current of air passed up from below, and (2) *Rotating Generators* consisting of rectangular tanks containing the alcoholic liquor into which dip slowly revolving drums containing beech shavings. In the former process the acetification is completed in two or three days, in the latter, in about four weeks.

The alcoholic liquid from which genuine malt vinegar is made is derived from the wort obtained by mashing malt, or a mixture of malt and cereals. Sugar and glucose vinegar are prepared by the alcoholic and acetic fermentation of diluted molasses, or other sugar by-product, and glucose respectively. Spirit vinegar is derived from diluted whiskey, brandy, or alcohol.

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\* Tolman and Hartman, Jour. Ind. Eng. Chem., 9, 1917, p. 759.

† Anderson, Tech. Quart., 6, 1914, p. 214.

‡ Farnstainer, Zeits. Unters. Nahr. Genussm., 2, 1899, p. 198; 15, 1908, p. 321; Browne, Jour. Amer. Chem. Soc., 25, 1903, p. 29; Pastureau, Jour. pharm. chim., 21, 1905, p. 593; Balcom, Jour. Amer. Chem. Soc., 39, 1917, p. 309.

§ Crawford, Jour. Ind. Eng. Chem., 5, 1913, p. 846; Rowatt, Lab. Int. Rev. Dept. Canada, Bul. 64, 1917.



**CHARACTERISTICS AND COMPOSITION OF THE VARIOUS VINEGARS.**

—**Cider Vinegar** is brownish yellow in color, and possesses an odor of apples. It is chiefly distinguished from other vinegar by the presence of more or less malic acid, by the character of its sugars, and by the predominance of potash in the ash. Its specific gravity varies from 1.013 to 1.015. Its acidity varies from 3 to 6 per cent, and its solids from 1½ to 3 per cent. Cider vinegar under polarized light is always lævorotatory.

The following are summarized data of analyses made by H. C. Lythgoe in the writer's laboratory of twenty-two samples of cider vinegar of known purity.

	Acetic Acid.	Total Solids.	Ash.	Alkalinity of Ash. <sup>1</sup>	P <sub>2</sub> O <sub>5</sub> in Ash of 100 Grams Vinegar.	
					Soluble (mgr.).	Insoluble (mgr.).
Maximum.....	5.86	3.20	0.42	36.1	31.7	31.5
Minimum. ....	3.92	1.84	0.20	22.2	12.1	6.5
Average.....	4.84	2.49	0.34	29.7	19.2	15.6

	Reducing Sugars.		Polarization, Degrees Ventzke 200-mm. Tube.	Malic Acid.	Per Cent Ash in Total Solids.	Per Cent Reducing Sugars in Total Solids.	Ratio of Soluble to Total P <sub>2</sub> O <sub>5</sub> .	Alkalinity of 1 Gram of Ash, cc. $\frac{N}{10}$ Acid.
	Before Inversion.	After Inversion.						
Maximum....	0.51	0.53	-3.6	0.16	19.0	16.6	66.9	125.0
Minimum. ...	0.15	0.15	-0.3	0.08	10.0	7.3	50.0	69.0
Average.....	0.25	0.25	-1.3	0.11	13.8	10.7	56.3	90.0

<sup>1</sup> Number of cubic centimeters of tenth-normal acid to neutralize the ash of 100 grams of vinegar.

Twenty-two samples of pure cider vinegar were analyzed by A. W. Smith\* with the following results:

	Acetic Acid.	Total Solids.	Ash.	Alkalinity of Ash. <sup>1</sup>	Soluble P <sub>2</sub> O <sub>5</sub> .	Insoluble P <sub>2</sub> O <sub>5</sub> .	Total P <sub>2</sub> O <sub>5</sub> .
Maximum.....	7.61	4.45	0.51	55.2	22.7	19.4	39.0
Minimum. ....	3.24	2.00	0.31	28.4	13.6	4.2	19.8
Average.....	4.46	2.83	0.39	38.8	19.1	10.1	28.6

<sup>1</sup> Number of cubic centimeters of tenth-normal acid required to neutralize the ash from 100 grams of vinegar.

Van Slyke,† in extensive experiments on the home manufacture of vinegar, found that the fixed acid in seventeen samples of fresh apple juice

\* Jour. Am. Chem. Soc., 20 (1898), p. 6.

† N. Y. Agr. Exp. Sta., Bul. 258, 1904.



was 0.55 per cent; after six months it was reduced to 0.39 per cent, after eight months to 0.13 per cent, after fifteen months to 0.06 per cent and after twenty-four months to 0.02 per cent, the greatest loss being after the alcoholic fermentation had ceased and before the acetic fermentation was considerable.

Tolman and Goodnow\* have shown that vinegar made in the tank type of generator differs little in composition from that of the hard cider except for conversion of alcohol into acetic acid, a marked loss in fixed acids, and a gain in pentosans. For example, in one experiment the hard cider contained 0.15 per cent of fixed acids and 0.15 per cent of pentosans, whereas the vinegar contained 0.06 per cent of fixed acids and 0.19 per cent of pentosans. In another experiment the percentages in the hard cider were 0.19 and 0.12 and in the vinegar 0.06 and 0.18 per cent respectively.

Hartman and Tolman,† from experiments carried out with the rotating generator process extending through two years, concluded: (1) During fermentation a large part of the malic acid of the apple juice is destroyed to form lactic acid. (2) During acetification the remaining malic acid is almost entirely oxidized. (3) The fixed acid in the vinegar is chiefly lactic acid.

In the final vinegar made from a mixture of first and second pressings they obtained, among other results, in the filtered and cleared stock respectively as follows: solids 1.31 and 1.21, non-sugar solids 1.19 and 1.10, volatile acid as acetic 6.44 and 6.58, malic acid 0.0335 and 0.0440, succinic acid 0.0085 and 0.0110, lactic acid 0.225 and 0.285, formic acid 0.0004 and 0.0004, and acetates as acetic acid 0.043 and 0.039 per cent.

Bender‡ found that the glycerol content of cider vinegar made by the generator process in New Jersey ranged from 0.25 to 0.45 per cent. Goodnow‡ found the range for generator vinegar produced in Michigan and New York to be 0.24 to 0.46 and 0.25 to 0.31 per cent respectively. Both investigators made daily tests for several months of both the cider stock and the finished vinegar.

The composition of cider vinegar ash is found by Doolittle and Hess § to be as follows:

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\* Jour. Ind. Eng. Chem., 5, 1913, p. 928.

† Ibid., 9, 1917, p. 759.

‡ U. S. Dept. of Agric. Notice of Judgment No. 1159.

§ Jour. Amer. Chem. Soc., 22, 1900, p. 220.



Calcium oxide.....	CaO.....	3.4 to 8.21
Magnesium oxide.....	MgO.....	1.88 to 3.44
Potassium oxide.....	K <sub>2</sub> O.....	46.33 to 65.64
Sodium oxide.....	Na <sub>2</sub> O.....	None
Sulphuric anhydride.....	SO <sub>3</sub> .....	4.66 to 16.29
Phosphoric anhydride.....	P <sub>2</sub> O <sub>5</sub> .....	3.29 to 6.66
Iron oxide.....	Fe <sub>2</sub> O <sub>3</sub> .....	None to trace
	CO <sub>2</sub> and loss...	0.00 to 40.44

**Wine Vinegar** is light yellow if made from white wine, and red if from red wine. The former is the highest prized. Wine vinegar varies in specific gravity from 1.0129 to 1.0213, and contains from 6 to 9 per cent of acetic acid. It is characterized chiefly by the bitartrate of potassium (cream of tartar) which true wine vinegar always possesses. Free tartaric acid is also usually present. Wine vinegar is the principal vinegar of France and Germany. In the United States the term white wine vinegar is usually applied to distilled or spirit vinegar, which is much cheaper than the real wine vinegar and altogether inferior to it.

Wine vinegar is slightly lævo-rotary with polarized light.

The composition of genuine white wine vinegar is shown by the following summary of the analyses of twenty-two samples, made in the Municipal Laboratory of Paris:

	Specific Gravity.	Total Solids.	Sugar.	Bitartrate of Potash.	Ash.	Acidity (as Acetic).
Maximum.....	1.0213	3.19	0.46	0.36	0.69	7.38
Minimum.....	1.0129	1.38	0.06	0.07	0.16	4.44
Mean.....	1.0175	1.93	0.22	0.17	0.32	7.38

Weigmann gives the following average of analyses of red wine vinegar:

Specific Gravity.	Acetic Acid.	Total Tartaric Acid.	Free Tartaric Acid.	Cream of Tartar.	Alcohol.	Extract.	Glycerin.	Ash.	Phosphoric Acid.
1.0143	7.79	0.216	0.006	0.057	1.19	0.863	0.141	0.118	0.012

**Malt or Beer Vinegar** is of a brown color, and its odor is suggestive of sour beer. It varies in specific gravity from 1.015 to 1.025; its acidity is about the same as cider vinegar, but the extract is much larger, varying from 4 to 6 per cent. Malt vinegar contains considerable nitrogenous



matter, and notable quantities of phosphates, dextrin, and maltose. It contains no cream of tartar. Malt vinegar is largely used in Great Britain.

Hehner gives the following data of the analyses of seven samples of vinegar undoubtedly made from malt only:\*

	Acidity.	Total Solids.	Ash.	Phosphoric Anhydride.	Alkalinity ( $\text{Na}_2\text{CO}_3$ ).
Maximum.....	6.48	4.23	0.47	.13	.089
Minimum.....	2.88	1.68	0.22	.067	.017
Mean.....	4.23	2.70	0.34	.105	.024

Allen gives the results of the analyses of three samples of genuine vinegar brewed from a mixture of malted and unmalted barley as follows:†

	Specific Gravity.	Acetic Acid.	Total Solids.	Ash.	Alkalinity as $\text{K}_2\text{O}$ .	Phosphoric Acid.	Nitrogen.	Albuminoids.
1.....	1.0170	6.39	2.57	0.34	0.091	0.077	.099	.624
2.....	1.0228	5.26	3.96	0.40	0.118	0.093	.095	.598
3.....	1.0160	4.86	2.31	0.47	.....	0.057	.099	.624

Wyatt and Schlichting ‡ consider the U. S. limit for phosphoric acid in the soluble ash (page 804) too high and have shown that malt vinegar is often lævorotatory. A shipment of malt vinegar pronounced an imitation by the United States authorities analyzed, according to Wyatt and Schlichting, as follows: sp.gr. at 60° F. 1.0164, acetic acid 4.65, lactic acid 0.47, total acidity calculated as acetic 4.97, total solids 2.03, dextrin trace, ash 0.46, alkalinity of ash ( $\text{K}_2\text{O}$ ) 0.06, phosphoric acid 0.08, protein ( $\text{N} \times 6.25$ ) 0.50, opticity (angular degrees, 200 mm. tube) -0.307. Chapman confirmed their findings. Russell and Hodgson further criticise the United States Standards as permitting sophistication. They propose a minimum of 0.05 per cent for phosphoric acid; in authentic samples they found a range of 0.047 to 0.092 per cent.

\* Analyst, 16, p. 82. See also Analyst, 18, p. 240.

† Ibid., 19, p. 15.

‡ Eighth Int. Cong. App. Chem., 14, 1912, p. 277.



**Distilled, Spirit, or Alcohol Vinegar.**—This vinegar, being made from diluted alcohol, is nearly colorless, unless artificially colored, as it often is, with caramel. As stated on page 792, the "white wine" vinegar (incorrectly so-called) commonly sold in the United States is of this class. Its specific gravity ranges from 1.008 to 1.013. Spirit vinegar contains from 3 to 10 per cent of acetic acid. Its content of total solids is insignificant, and it contains only traces of ash. It always contains non-acetified alcohol and aldehyde. It has no optical activity with polarized light.

Twelve samples of distilled vinegar analyzed in the Municipal Laboratory of Paris gave according to Girard \* the following results:

	Specific Gravity.	Total Solids.	Sugar.	Ash.	Acidity.
Maximum.....	1.0131	0.58	Trace	.09	7.98
Minimum.....	1.0082	0.16	"	Trace	4.98
Average.....	1.0100	0.35	"	.04	6.34

An analysis of a sample of typical distilled or spirit vinegar as produced in the United States is given in the table on page 806.

**Glucose Vinegar** is made from the acetification of alcohol, obtained from the fermentation of commercial glucose. This vinegar usually possesses the odor and taste of fermented starch. It is low in total solids, the extract consisting almost entirely of untransformed glucose, and the vinegar therefrom contains all the ingredients of the product from which it was made, viz., dextrin, maltose, and dextrose, as well as chloride of sodium. It is decidedly dextro-rotatory with polarized light both before and after inversion.

**Molasses Vinegar.**—This is largely the product of the acetic fermentation of sugar-house wastes, and sometimes of the accidental acetic fermentation of molasses itself, after it has undergone alcoholic fermentation for the manufacture of rum. This variety of vinegar is sometimes used as an adulterant of cider vinegar. With polarized light molasses vinegar is dextrorotatory before, and levorotatory after inversion.

\* Analyse des matières alimentaires, Paris, 1904, p. 271.



**Wood Vinegar** is prepared by the purification of pyroligneous acid, which may be accomplished by saturating the crude acid with lime or soda, adding hydrochloric or sulphuric acid, and distilling. It is further purified by redistillation with potassium bichromate, and filtration through bone-black. Acetic acid is sometimes added to impart flavor.

The extract and ash of wood vinegar are very small. Its specific gravity averages 1.007 according to Blyth. Empyreumatic or tarry products are nearly always present in vinegar of this class.

#### ANALYSIS OF VINEGAR.

**Specific Gravity.**—This is obtained either with the hydrometer, pycnometer, or Westphal balance.

**Determination of Total Solids.**—Weigh 10 grams of the sample in a tared platinum dish 50 mm. in diameter, evaporate to dryness on a boiling-water bath and dry for two and one-half hours in a water oven at the temperature of boiling water. Cool in a desiccator and weigh.

**Determination of Ash.**—Transfer the dish containing the last residue or extract to a muffle, and burn at a low red heat to an ash, or the ignition may be accomplished with care over a direct flame turned low. Cool the dish and weigh.

**Determination of Solubility and Alkalinity of the Ash.**—*Smith's Method.*\*—Twenty-five cc. of the vinegar are evaporated to dryness in a tared platinum dish, ignited, cooled, and the ash weighed. The ash is then repeatedly extracted with hot water by washing into a Gooch crucible provided with a layer of asbestos (previously ignited in the crucible, cooled, and weighed) or upon an ash-free filter. Dry the Gooch or filter, ignite, cool, and weigh the insoluble ash. The aqueous extract is titrated directly with tenth-normal acid, using methyl orange as an indicator, or treated by adding an excess of tenth-normal hydrochloric acid, boiling and titrating back with tenth-normal sodium hydroxide, using phenolphthalein. Express the alkalinity in terms of 100 grams of the vinegar, by multiplying by 4 the number of cubic centimeters of acid required to neutralize.

**Determination of Phosphoric Acid.**†—Extract repeatedly the insoluble ash as obtained in the preceding section with hot water acidulated with nitric acid, and acidify with nitric acid the neutralized solution of the

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\* Jour. Amer. Chem. Soc., 20, p. 5.

† U. S. Dept. of Agric., Bur. of Chem., Bul. 46, p. 12.



soluble ash. Add to each solution 15 grams of ammonium nitrate, heat to boiling, and precipitate the phosphoric acid with 50 cc. of ammonium molybdate reagent after which digest for an hour at a temperature of about  $65^{\circ}$ , filter, and wash with cold water. Dissolve the precipitate on the filter with ammonia and hot water, and wash into a beaker to a bulk of not more than 100 cc. Nearly neutralize with hydrochloric acid, cool, and add slowly magnesia mixture (prepared as usual), drop by drop while stirring vigorously. After fifteen minutes add 30 cc. of ammonia (specific gravity 0.96), let stand for at least two hours, filter on a Gooch crucible, wash with 2.5% ammonia till practically free from chlorides, ignite, and weigh as  $Mg_2P_2O_7$ . Express results in terms of milligrams of phosphoric anhydride in the soluble and insoluble vinegar ash from 100 cc. of vinegar.

Phosphoric acid in the soluble and insoluble ash may be conveniently determined also by the uranium acetate method, page 757.

**Determination of Nitrogen.**—Concentrate from 50 to 100 cc. of vinegar to a syrupy consistency, and proceed as directed under the Kjeldahl or Gunning method, page 58.

**Determination of Total Acidity.**—Six cc. of vinegar are carefully measured from a pipette into a white porcelain dish and diluted with water. Using phenolphthalein as an indicator, titrate with tenth-normal sodium hydroxide. The number of cubic centimeters of the latter required to neutralize, divided by 10, expresses the acidity in terms of percentage of acetic acid.

*Approximate Determination of Vinegar Acidity by Lime Water.*—It has generally been considered difficult for vinegar dealers and others who desire to estimate the acidity of their vinegar to do this themselves, in that it has been necessary to obtain for the purpose a carefully standardized alkaline solution, the exact strength of which it is impossible for them to determine.

It has been found that very satisfactory, though of course not absolutely accurate, results may be obtained by the use of ordinary lime water, which any one may easily prepare by making a saturated solution of ordinary air-slaked lime. The strength of such a solution is very nearly constant, and has been found to be about  $\frac{1}{21.4}$  of the normal. If, therefore, it is not easy to obtain exactly normal or tenth-normal alkali, approximate figures may be obtained by employing such a saturated lime water. If 2.75 cc. of vinegar are titrated with lime water contained in a burette, using phenolphthalein as an indicator, the number of cubic centimeters



of the lime water necessary to neutralize the vinegar, divided by 10, gives the percentage of acetic acid in the vinegar. To make sure that the lime water is saturated, an excess of lime should always be present in the reagent bottle.

**Determination of Volatile and Fixed Acids.**—Thirty cc. of the vinegar are transferred to a distilling-flask and subjected to distillation, using a current of steam. Receive the distillate in a 25-cc. graduated cylinder. After 15 cc. have passed over, test from time to time the drops of distillate as they fall into the receiving vessel with litmus-paper, and when free from acid discontinue the distillation. Note the volume of the distillate, mix by shaking, and transfer one-fifth to a white porcelain dish. Titrate as in the case of total acidity, expressing the volatile acids as acetic.

Calculate the fixed acid, expressed in the case of cider vinegar as malic, by subtracting the percentage of volatile acid from the percentage of total acid, and multiplying the result by the factor 1.117. In the case of wine vinegar, express as tartaric acid by using the factor 1.25. To express acidity in terms of sulphuric acid, multiply the percentage of acetic acid by 0.817.

**Determination of Alcohol.**—Alcohol is present in very small amounts in fruit vinegar that has not been completely acetified. Frear recommends concentrating the distillates as follows: Neutralize 100 cc. of the sample and distill off 40 cc. Then redistill the distillate till 20 cc. have gone over. Cool to 15.6° C. and make up to 20 cc. with distilled water. Determine the specific gravity with a 10-cc. pycnometer, and ascertain from the table on page 690 the per cent by weight of alcohol corresponding to the specific gravity. The percentage in the last distillate, divided by 5, expresses the amount of alcohol in the vinegar.

**Detection of Free Mineral Acids.**—The ash of genuine cider vinegar is always alkaline. If the ash is neutral, free mineral acids are doubtless present. For their detection the following is a modification of Brann's method of procedure:

Add to 50 cc. of the vinegar in an Erlenmeyer flask a small bit of starch the size of a wheat-grain, and shake to disseminate it through the fluid. Boil for some minutes, cool, and add a drop of iodine solution. If a blue coloration occurs, no mineral acid is present. In the presence of an appreciable amount of mineral acid, the starch will be converted to dextrin and sugar, and no coloration will be produced by the iodine.

*Frear's Method.*—Add 5 or 10 cc. of water to 5 cc. of the vinegar, and



to the mixture add a few drops of a solution of methyl violet (one part of methyl violet 2B in 100,000 parts of water). In the presence of mineral acids, a blue or green coloration will be produced.

**Determination of Free Mineral Acids.**—*Hehner's Method*.<sup>\*</sup>—To a weighed quantity of the sample add an excess of decinormal alkali, evaporate to dryness, incinerate, and titrate the ash with decinormal acid. The difference between the number of cubic centimeters of alkali added in the first place, and the number of cubic centimeters needed to titrate the ash, represents the equivalent of the free acid present.

**Detection and Determination of Sulphuric Acid.**—This is determined as barium sulphate by the addition of barium chloride solution. A slight cloudiness on the addition of the reagent indicates the presence of small quantities of sulphate as an impurity, rather than free sulphuric acid. If a minute quantity of free sulphuric acid be present, a rather heavy white cloud on the addition of the barium chloride will be formed, which slowly settles out. According to Brannt, if the quantity of sulphuric acid is more than one part in a thousand, the sulphate of barium formed by addition of the reagent produces a copious precipitate that rapidly falls to the bottom of the receptacle. This may be filtered, washed, ignited, and weighed in the usual manner.

**Detection of Free Hydrochloric Acid.**—Distill off half of a measured volume of vinegar into the receiving-flask of a distillation apparatus, and to the distillate add a few drops of nitrate of silver reagent. A precipitate indicates hydrochloric acid.

**Detection of Malic Acid (*Free or Combined*).**—Absence of malic acid may be assured, if no precipitate occurs with neutral acetate of lead, when a few drops of a solution of this reagent are added to the vinegar. In the presence of malic acid, as in the case of a pure cider vinegar, the precipitate which is formed with lead acetate is flocculent, forms at once, and is of considerable amount. In pure cider vinegar the precipitate will settle to the bottom of the test-tube, leaving a clear supernatant liquid within ten minutes. Unfortunately the acetate of lead test is a negative one, in that several organic acids other than malic will cause a precipitate, as, for instance, tartaric and saccharic acids, the former being found in wine and the latter in molasses vinegar. Malt vinegar also gives a copious precipitate with lead acetate, due to phosphoric acid.

The writer employs the following test † for detecting malic acid in

<sup>\*</sup> Analyst, 1, 1877, p. 105.

† An. Rep. Mass. State Board of Health, 1902, p. 485. Food and Drug Reprint, p. 33.



vinegar: Add a few drops of a 10% solution of calcium chloride to some of the vinegar in a test-tube, and make the mixture slightly alkaline with ammonia. Filter off the precipitate that occurs at this point, to the filtrate add two or three volumes of 95% alcohol, and heat to boiling. A copious, flocculent precipitate of calcium malate will form, if malic acid be present, settling to the bottom of the tube in a few minutes. A precipitate will occur in malt and glucose vinegar, due to dextrin.

To confirm the presence of malic acid, filter, wash the precipitate with a little alcohol, dry, dissolve it in strong nitric acid in a porcelain evaporating-dish, and evaporate to dryness over the water-bath, forming calcium oxalate. Boil the residue with sodium carbonate, filter, acidify the filtrate with acetic acid, boil to expel the carbon dioxide, and add a solution of calcium sulphate. A precipitate of calcium oxalate confirms the presence of malic acid.

The quantitative determination of malic acid is seldom necessary.

**Lead Precipitate.**—*Hortvet Number.*—The quantitative measurement of the precipitate formed with lead acetate, or subacetate, is of considerable importance. Even though the precipitate formed may not be due as was long thought to malic acid, but may be due to phosphoric acid (though this has not been fully proved), it nevertheless remains a fact that the qualitative lead acetate test is one of the most important of all in judging the purity of cider vinegar.

The lead precipitate is best measured as follows: To 25 cc. of the vinegar add 2.5 cc. of U. S. P. subacetate of lead solution. Shake and whirl in a graduated Hortvet tube in the centrifugal machine, and read the volume of the precipitate in the bottom of the tube. The results expressed in cc. on thirty samples of pure cider vinegar are summarized as follows: Highest, 1.4; lowest, 0.5; average, 0.84. The Hortvet number of adulterated cider vinegar runs from a mere trace to 0.5 and sometimes higher.

*Winton's Lead Number.*—This is determined by the method described for maple products, page 658.

Bailey\* obtained by this method the following results:

Cider vinegar (8 samples) . . . . .	0.075 to 0.290
Malt vinegar (3 samples) . . . . .	0.158 to 0.548
Distilled vinegar (1 sample) . . . . .	0.018

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\* A. O. A. C. Proc., 1908. U. S. Dept. of Agric., Bur. of Chem., Bul. 122, p. 27.



Hickey \* follows the same method, except that he employs only 5 cc. of standard lead subacetate solution and determines the lead in 50 cc. of the filtrate. The lead number found by him in twenty samples of cider vinegar varied from 0.076 to 0.166.

**Determination of Acid Tartrate of Potassium.**—*Berthelot and Fleuriens's Method.*†—Twenty-five cc. of the vinegar are evaporated on the water-bath to syrupy consistency, and the residue is dissolved in water and made up to its original volume. It is then transferred to a 250-cc. Erlenmeyer flask, and 100 cc. of a mixture of equal parts of strong alcohol and ether are added, the flask is corked, shaken, and set on ice or in a cold place for forty-eight hours. At the end of this time, if a crystalline precipitate has gathered, the supernatant liquid is decanted upon a filter, and finally the precipitate is washed upon it by a fresh quantity of the ether-alcohol mixture, and the washing continued with this reagent till practically free from acid. The filter and its contents are then transferred to the original flask, and the tartrate is dissolved in boiling water, after which the solution is titrated in the same flask with tenth-normal sodium hydroxide, using phenolphthalein as an indicator. Multiply the number of cubic centimeters of alkali required to neutralize by the factor 0.0188, and the quotient expresses the grams of bitartrate of potash in the sample. Multiply this by 4 to obtain the percentage present.

**Polarization and Determination of Sugar.**—If the vinegar is light-colored and quite free from turbidity, it may sometimes be polarized undiluted in the 100-mm. tube. Vinegar may often be sufficiently clarified for polarization by filtering twice through the same filter. It is, however, best to add 10% of basic lead acetate solution, and to filter before polarizing, thus removing the malic or tartaric acids which may have a slight effect on the polarization. In case of dark-colored or turbid samples, add to 50 cc. of the sample 5 cc. of about equal quantities of lead subacetate and alumina cream, shake, filter, and polarize in a 200-mm. tube, adding 10% to the reading on account of the dilution. The polarization value of the vinegar is conveniently expressed in terms of actual direct reading obtained by the undiluted sample in a 200- or 400-mm. tube.

If the invert reading is desired for calculation of sucrose or commercial glucose, subject the sample to inversion with hydrochloric acid and heat, as in the case of sugars.

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\* Ibid.

† Girard, *Analyse des Matières Alimentaires*, Paris, 1904, p. 144.



For the determination of sucrose, use Clerget's formula (page 611), calculating the true direct and invert readings from the direct and invert readings of the undiluted vinegar on the basis of the normal weight of the sample, by multiplying the obtained readings by 0.26 in the case of the Soleil-Ventzke instrument.

**Determination of Reducing Matter before and after Inversion.**—Measure two portions of 25 cc. each into 100-cc. flasks. Dilute one portion with 25 cc. of water, add 5 cc. of concentrated hydrochloric acid and invert in the usual manner. Neutralize both portions with sodium hydroxide, clear with normal lead acetate, remove the excess of lead with potassium sulphate or carbonate, and make up to the mark. Determine reducing sugars in each portion by the Munson and Walker method (page 622) and calculate as invert sugar.

The above method gives the total reducing matters before and after inversion. To determine the non-volatile reducing matters evaporate the 25 cc. portions to dryness then take up in 50 cc. of water and proceed as described.

**Determination of Pentosans.**—Place 100 cc. of the vinegar in a flask, add 43 cc. of concentrated hydrochloric acid (sp.gr. 1.19) and proceed as described on page 286.

**Determination of Glycerol.**—The glycerol is extracted by essentially the same process as is used for dry wines (p. 703) and determined by the Hehner method modified by Richardson and Jaffé \* and Low. These processes have been adapted to vinegar analysis by Ross † as follows:

*Standard Solutions.*—1. *Strong Bichromate.*—Dissolve 74.56 grams of dry, recrystallized potassium bichromate in water, add 150 cc. concentrated sulphuric acid, cool, make up to 1000 cc. at 20° C., and determine the specific gravity at 20°/20° C.; 1 cc. = 0.01 gram glycerol. Accurate measurements being difficult owing to changes in room-temperature it is well to use weighed amounts of the solution from a weight burette, dividing by the specific gravity to obtain the volume used. The solution has an apparent expansion in glass of 0.0005 (or 0.05%) for each degree centigrade. The solution may be measured if this correction is made.

2. *Dilute Bichromate.*—Introduce a weighed amount (12.5 times the specific gravity) of the strong bichromate from a weight burette into a 250 cc. glass-stoppered volumetric flask, make up to the mark with water at room temperature; 20 cc. = 1 cc. of the strong solution. If slightly more than 12.5 cc. equivalent is used, make up to the mark and then add the required amount of water to make one-twentieth dilution.

\* Jour. Soc. Chem. Ind., 17, 1898, p. 330.

† Proc. A. O. A. C., 1910. U. S. Dept. of Agric., Bur. of Chem., Bul. 137, p. 61.



3. *Ferrous Ammonium Sulphate*.—Dissolve 30 grams of the crystallized salt in water, add 50 cc. of concentrated sulphuric acid, cool and dilute to 1000 cc. at room temperature; 1 cc. = approximately 1 cc. of the dilute bichromate. Owing to daily changes in strength it should be standardized against the bichromate whenever used.

*Extraction of Glycerol*.—Make all evaporations on a water-bath kept at 85° to 90° C. Evaporate 100 cc. of the vinegar to about 5 cc., add 20 cc. of water and again evaporate to about 5 cc. to expel acetic acid. Add about 5 grams of fine sand and 15 cc. of milk of lime (freshly prepared and containing about 15% of calcium oxide), and evaporate nearly, but not quite, to dryness, with frequent stirring, avoiding formation of dry crust. Rub into a homogeneous paste with 5 cc. of hot water, add 45 cc. of absolute alcohol, washing down paste adhering to the sides of the dish, and stir thoroughly. Heat the mixture on a water-bath with constant stirring to incipient boiling, decant onto a 12.5 cm. fluted filter, wash twice by decantation and finally on the filter with 90% alcohol up to about 150 cc., or, instead of filtering, centrifuge and wash three times. Evaporate to a sirup, dissolve in 10 cc. of absolute alcohol, and wash into a 50 cc. glass-stoppered cylinder with two 5 cc. portions of absolute alcohol. Add three portions of 10 cc. each of absolute ether, thoroughly shaking after each addition. Let stand until clear, then pour off through a filter, and wash the cylinder and filter with mixed absolute alcohol and absolute ether (1:1.5). If a heavy precipitate is observed in the cylinder, it is well to centrifuge at low speed and decant the clear liquid through a filter. Add 20 cc. of the mixture of absolute alcohol and absolute ether to the precipitate in the cylinder, shake thoroughly, centrifuge and decant, repeating three times. Evaporate filtrate and washings at 85°-90° C., to about 5 cc.; dilute and evaporate to 5 cc. three times, using respectively 20, 20 and 10 cc. of water. Wash residue with hot water into a 50 cc. volumetric flask, cool, add silver carbonate freshly precipitated from 0.1 gram of silver sulphate, shake occasionally, and allow to stand 10 minutes; then add 0.5 cc. of lead subacetate solution, shake occasionally, and allow to stand 10 minutes. Make up to the mark, shake well, filter, rejecting the first portion of the filtrate, and pipette off 25 cc. of the clear filtrate into a 250 cc. glass-stoppered volumetric flask. Precipitate the excess of lead with 1 cc. of concentrated sulphuric acid, and determine the glycerol by the following method:

*Determination*.—From a weight burette introduce into the 250 cc. flask, containing the 25 cc. of purified glycerol solution, a weighed amount of the strong bichromate solution (with ordinary vinegar 30-35 cc.) suf-



ficient to leave about 12.5 in excess, carefully add 24 cc. of concentrated sulphuric acid, rotating gently to mix and avoid ebullition, then heat in boiling-water bath for exactly 20 minutes. Dilute at once, cool, and make up to mark at room temperature. The oxidation is a trifle more complete if only 15 cc. of concentrated sulphuric acid are added and the digestion is continued for at least 2 hours.

Standardize the ferrous ammonium sulphate solution against the dilute bichromate by introducing from burettes approximately 20 cc. of each into a beaker containing 100 cc. of water. Complete the titration, using potassium ferricyanide solution (0.5 to 1%) as indicator on a porcelain spot plate. Calculate the volume ( $F$ ) of ferrous ammonium sulphate equivalent to 20 cc. of the dilute and, consequently, to 1 cc. of the strong bichromate solution.

Substitute for the dilute bichromate a burette containing the oxidized glycerol with excess of bichromate solution, and ascertain how many cubic centimeters of it are equivalent to  $F$  cc. of the ferrous ammonium sulphate solution, and therefore to 1 cc. of the strong bichromate. Then 250 divided by this last equivalent equals the number of cubic centimeters excess of the strong bichromate present in the 250 cc. flask after oxidation of the glycerol.

The number of cubic centimeters of strong bichromate added, minus the excess found after oxidation, multiplied by 0.01 equals the weight of glycerol in the 25 cc. of purified solution used in the determination; this result, multiplied by 2, gives the weight of glycerol in grams per 100 cc. of the vinegar.

#### ADULTERATION OF VINEGAR.

**Standards of Purity.**—In England, where the principal vinegar is malt vinegar, the legal standards are considerably different from those in force in France and Germany, where wine vinegar is prevalent. These differ again from the requirements found in the United States and Canada, where cider vinegar is the chief product.

Some of the state food laws fix a standard for the acidity of cider vinegar varying from 3.5 to 4.5 per cent of acetic acid, and in most cases also a minimum standard for total solids or residue of from 1.5 to 2 per cent. Special laws stipulate furthermore in some states that cider vinegar, sold as such, must be *exclusively* the product of pure apple cider.

Following are the U. S. standards for the various vinegars:

*Vinegar, Cider Vinegar, Apple Vinegar*, is the product made by the alcoholic and subsequent acetous fermentations of the juice of apples, is lævo-rotatory, and contains not less than 4 grams of acetic acid, not



less than 1.6 grams of apple solids, of which not more than 50% are reducing sugars, and not less than 0.25 gram of apple ash in 100 cc. (20° C.); and the water-soluble ash from 100 cc. (20° C.) of the vinegar contains not less than 10 milligrams of phosphoric acid ( $P_2O_5$ ), and requires not less than 30 cc. of decinormal acid to neutralize its alkalinity.

*Wine Vinegar, Grape Vinegar*, is the product made by the alcoholic and subsequent acetous fermentations of the juice of grapes, and contains in 100 cc. (20° C.), not less than 4 grams of acetic acid, not less than 1.0 gram of grape solids, and not less than 0.13 gram of grape ash.

*Malt Vinegar* is the product made by the alcoholic and subsequent acetous fermentations, without distillation, of an infusion of barley malt, or cereals whose starch has been converted by malt, is dextro-rotatory, and contains, in 100 cc. (20° C.), not less than 4 grams of acetic acid, not less than 2 grams of solids, and not less than 0.2 gram of ash; and the water-soluble ash from 100 cc. (20° C.), of the vinegar contains not less than 9 milligrams of phosphoric acid ( $P_2O_5$ ), and requires not less than 4 cc. of decinormal acid to neutralize its alkalinity.

*Sugar Vinegar* is the product made by the alcoholic and subsequent acetous fermentations of solutions of sugar, syrup, molasses, or refiners' syrup, and contains in 100 cc. (20° C.), not less than 4 grams of acetic acid.

*Glucose Vinegar* is the product made by the alcoholic and subsequent acetous fermentations of solutions of starch sugar or glucose, is dextro-rotatory, and contains, in 100 cc (20° C.), not less than 4 grams of acetic acid.

*Spirit Vinegar, Distilled Vinegar, Grain Vinegar*, is the product made by the acetous fermentation of dilute distilled alcohol, and contains, in 100 cc. (20° C.), not less than 4 grams of acetic acid.

**Accidental Adulteration** of vinegar may result in the presence of injurious metallic salts, such as of copper, lead, or zinc, derived from vessels or utensils used in the manufacture of vinegar, or even minute traces of arsenic may be found, when glucose has been employed as an ingredient or source of the vinegar, the arsenic being in this case probably due to impure acid used in the manufacture of the glucose.

**Fraudulent Adulteration** may consist merely in diluting cider, malt, or wine vinegar to 4 per cent acid strength or less without a declaration on the label or in substituting distilled vinegar wholly or in part for the more expensive kinds.



Imitation cider vinegar is frequently made up of spirit vinegar, colored with caramel, the solids being reinforced by boiled cider or apple jelly, made often out of exhausted apple pomace, left after the apple stock has been subjected to one and sometimes two pressings. Since sweet cider usually contains over 11% of solids and the standard for cider vinegar requires only 1.6%, it is readily seen how a given volume of cider may be made to contribute the normal amount of solids to at least seven times that volume of an artificial cider vinegar containing distilled vinegar as a base. More skilful mixtures contain about equal volumes of distilled and cider vinegar with sufficient boiled cider and caramel to make up for the deficiency of solids and color. To further mislead the analyst soluble mineral matter and even glycerin are added. Acetic ether is sometimes used to impart flavor.

The table on p. 806 by Balcom \* shows the range in composition of authentic vinegars and the influence of dilution with spirit vinegar, with and without addition of boiled cider, on the composition.

**Character of the Residue.**—The residue of pure cider vinegar should be thick, light brown in color, of a viscid or mucilaginous consistency, somewhat foamy, having an astringent acid, though pleasant, taste, very suggestive of baked apples, which it also resembles in color. The residue of malt or beer vinegar is brown and gummy, has a characteristic odor and contains a considerable quantity of dextrin. The odor of molasses is very apparent in the residue of vinegar from sugar-house wastes. If pyroligneous acid or wood vinegar has been introduced, the dried residue may have a tarry or smoky taste and smell.

The residue of cider vinegar is very soluble in alcohol, while that of malt vinegar is only slightly soluble. Wine vinegar residues dissolve readily in alcohol, except for the granular residue of cream of tartar. If the loop of a clean platinum wire be rubbed in the vinegar residue and ignited in a colorless Bunsen flame, the color imparted will, if the vinegar has been made from pure cider exclusively, consist altogether of the pale-lilac color of a potash salt without any of the yellow sodium flame being visible. In all vinegars other than of pure cider, the sodium flame will predominate, when the residue is burnt as above. Again, the ignited residue left in the loop of wire in the case of a pure cider vinegar will form a fusible bead, having a strong alkaline reaction upon moistened

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\* A. O. A. C. Proc., 1909. U. S. Dept. of Agric., Bur. of Chem., Bul. 132, p. 93.



## COMPARISON OF VINEGARS OF KNOWN CHARACTER WITH COMMERCIAL SAMPLES.

	Total Acid.	Total Solids.	Non-sugar Solids.	Reducing Sugars in Solids. Per cent.	Total Ash.	Alkalinity of Water-soluble Ash. Cc.	Ash in Non-Sugar Solids. Per cent.	Phosphoric Acid (P <sub>2</sub> O <sub>5</sub> ) Mgs. per 100 cc.			Polarization (direct). %
								Water Soluble.	Insoluble.	Total.	
Cider Vinegar.....											
Authentic * { Av....	4.94	2.54	1.90	19.6	0.37	35.7	18.8	17.3	12.0	29.3	-1.5
{ Max....	7.96	4.52	2.89	45.0	0.52	56.0	26.5	39.9	32.0	64.2	-3.6
{ Min....	3.29	1.37	1.26	5.6	0.20	21.5	11.2	6.7	4.3	15.1	-0.2
Commercial †....	4.65	2.40	1.51	37.2	0.32	31.9	21.2	12.4	10.5	22.9	-1.3
Spirit Vinegar.....	4.31	0.18	0.16	11.1	0.02	1.5	10.0	0.2	1.5	1.7	+0.6
Mixture A ‡....	4.51	1.27	0.80	37.0	0.20	17.5	25.0	5.5	7.4	12.9	-0.3
“ B §.....	4.72	2.15	1.05	51.2	0.28	37.0	26.7	8.7	10.4	19.1	
“ C   .....	4.46	2.11	0.91	56.9	0.29	33.0	31.9	11.5	10.8	22.3	-1.0
Molasses Vinegar..	4.66	2.09	1.73	17.0	0.56	11.1	32.4	1.8	15.2	17.0	0.0

\* Compilation of 100 analyses. The figures for each constituent represent from 56 to 94 samples.

† A mixture of 50 samples. All were passed but some were thought to contain a diluent and a few probably had been mixed with boiled cider or a similar material.

‡ Equal parts of two preceding samples.

§ Known mixture of cider and spirit vinegars fortified with boiled cider.

|| Commercial sample evidently of same general character as preceding.

test-paper, and effervescing briskly when immersed in acid. The presence in vinegar of even a slight trace of added mineral acid will prevent the ignited residue from having the alkaline reaction, or effervescing with acid.\*

The odor given off in the first stages of burning this residue to an ash should be noted. With cider vinegar the apple odor is very marked while burning. In vinegar wherein molasses products have been employed, the smell of charred sugar is usually apparent, while with glucose vinegar the smell of burnt corn predominates. On burning the residue of malt vinegar, the odor produced at first is not unlike that of toasted bread. At a later stage in the burning the vapors evolved are very pungent.

**Character of the Ash.**—The ash of pure cider and malt vinegar is quite strongly alkaline, while that of distilled and wood vinegar is only slightly alkaline. In cider and malt vinegar the quantity of phosphoric acid present in the ash is considerable, while only traces are present in distilled or spirit vinegar. Considerably more than half the phosphoric

\* Davenport, 18th An. Rep., Mass. Board of Health, 1887, p. 159.



acid in the ash of cider vinegar is soluble, while no soluble phosphoric acid is present in the ash of spirit vinegar.

The percentage of ash in total solids is of some value in judging the purity of cider vinegar. According to Frear,\* if the ash of the vinegar is less than 10% of the total solids, the vinegar may be suspected of having added unfermented material, while a percentage of ash less than 6 is absolute evidence that the vinegar is not genuine cider vinegar.

Balcom † finds that the percentage of ash in the non-sugar solids is more constant than in the total solids, since the amount of sugar varies greatly in pure vinegar. This figure is of service in detecting the presence of mineral matter when added in conjunction with boiled cider in such amount as not to disturb the normal ratio of ash to total solids.

The alkalinity of 1 gram of the ash of pure cider vinegar should be equivalent to at least 65 cc. of tenth-normal acid. At least 50% of the phosphates in the ash should be soluble in water.

**Character of the Sugars.**—Browne ‡ found that the rotation of the freshly expressed juice of eleven varieties of apple varied from  $-19.24^{\circ}$  to  $-49^{\circ}$  Ventzke, in a 400-mm. tube. Also that five samples of completely fermented cider, examined five or six months after pressing, polarized in a 400-mm. tube from  $-1.76^{\circ}$  to  $-5.28^{\circ}$ . He found also that 20 grams of a pure cider jelly made from concentrated apple juice diluted to 100 cc., had a left-handed rotation amounting to  $21.35^{\circ}$  in a 200-mm. tube, and finally that four cider vinegar samples of known purity showed readings of from  $-0.96^{\circ}$  to  $-2.94^{\circ}$  Ventzke in a 400-mm. tube.

The left-handed rotation of pure cider vinegar is a characteristic so fixed and unalterable that a right-handed polarization of more than  $0.5^{\circ}$  may safely be assumed as evidence of adulteration. The polarization of cider vinegar, expressed in terms of 200 mm. of the undiluted sample should lie between  $-0.1^{\circ}$  and  $-4.0^{\circ}$  Ventzke. If the direct polarization of a sample of vinegar is right-handed, while the invert is left-handed, sugar-house wastes or molasses may be suspected as an adulterant.

If both direct and invert readings are right-handed, commercial glucose is undoubtedly present. If the polarization of the vinegar is far to the left, boiled cider or apple jelly has probably been used to reinforce the solids.

Frear regards the ratio of reducing sugars after inversion to total

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\* Report of Penn. Dept. of Agric., 1898, p. 38.

† Loc. cit.

‡ Bull. 58, Penn. Dept. of Agric., "A Chemical Study of the Apple and Its Products."



solids as a useful factor in discriminating between pure cider vinegar and the common artificial substitutes in which the solids of distilled vinegar are reinforced by apple jelly, or in which glucose or molasses vinegars are used. When the reducing sugars after inversion form more than 25% of the entire solids, the alleged cider vinegar is usually spurious, although in exceptional cases it runs up to 45%. In pure cider vinegar the per cent of reducing sugar is the same after inversion as before. The same is true of glucose vinegar. Vinegar containing added molasses or cane sugar will, however, naturally show an increase in reducing sugar after inversion.

A large content of alcohol in cider vinegar, otherwise showing the constants of pure vinegar except for the low acidity, would indicate incomplete acetification. A high content of nitrogen is characteristic of malt vinegar.

The table below gives a summary of analyses of eighty-four samples of undoubtedly pure cider vinegar examined at the Food and Drug Department of the Massachusetts State Board of Health in 1901.\*

CIDER VINEGAR FOUND PURE.

	Acid (Per Cent).	Solids (Per Cent).	Ash. (Per Cent).	Polarization.
Maximum.....	6.36	4.00	0.58	-5.4
Minimum.....	4.50	2.01	0.19	-0.4
Mean.....	4.84	2.43	0.38	-2.0

The table on page 809 includes samples of adulterated vinegar, sold for cider vinegar, none of which was probably made from cider. It will be noticed that in several of the samples the amount of glucose was abnormally large, as is shown by the very high right-handed polarization, in one case amounting to over 12°.

**Glycerol** is absent in distilled vinegar hence a determination of this constituent is a valuable means of detecting distilled vinegar in cider vinegar. The limits for pure cider vinegar are given on page 791.

**Direct Tests Made on the Vinegar.**—Brannet † applies the test of odor in vinegar as determining its character, by rinsing out a large beaker

\* 32d An. Rep. (1900), p. 661, Food and Drug Reprint, p. 44; 33d An. Rep. (1901), p. 467, Food and Drug Reprint, p. 47; 34th An. Rep. (1902), p. 483, Food and Drug Reprint, p. 31.

† A Practical Treatise on the Manufacture of Vinegar, Phila., 1914, p. 231.



## VINEGAR NOT THE EXCLUSIVE PRODUCT OF PURE APPLE CIDER.

Per Cent Acetic Acid.	Per Cent Total Solids.	Per Cent Ash.	Per Cent Ash in Total Solids.	Polarization in 200-mm. Tube.	Lead Acetate.
5.90	.40	....	....	+1.4	No precipitate
5.14	.36	....	....	.0	" "
5.12	.53	....	....	+ .6	" "
4.83	3.70	.32	8.65	+8.0†	" "
4.82	2.71	.13	4.80	+9.6†	Heavy precipitate*
4.80	1.97	.20	10.15	+ .9	Precipitate
4.80	1.03	.27	14.75	+1.1	"
4.66	2.92	.20	6.49	+2.2	No precipitate
4.60	2.57	....	....	+2.6	" "
4.56	2.60	....	....	+7.0†	" †
4.54	3.97	.19	4.78	+5.6	No precipitate
4.54	3.90	.32	9.72	+5.0	" "
4.54	2.94	.23	7.82	+5.0	" "
4.54	2.70	.23	8.52	+ .4	Precipitate
4.50	3.05	....	....	+2.2	No precipitate
4.50	2.92	.22	7.52	+ .9	" "
4.50	2.69	....	....	+2.8	" "
4.48	3.80	....	....	+12.0†	" "
4.46	2.80	....	....	+2.6	" "
4.42	2.75	....	....	+3.2	Slight precipitate
4.42	2.10	....	....	+9.2	Precipitate
4.40	2.51	.20	11.15	+1.1	"
4.40	.97	....	....	+ .4	No precipitate
4.38	.29	....	....	+1.6	" "
4.32	.70	.09	12.86	....	" "
4.08	3.35	....	....	+1.2	Precipitate
3.98	.55	....	....	+1.8	Slight precipitate

\* Cider vinegar to which apple jelly containing glucose had been added for the purpose of increasing the solids after watering.

† This sample contained a large amount of phosphate, and consequently the test for malates is obscured.

‡ These samples polarized practically the same after as before inversion, indicating much glucose.

with the sample, and after allowing it to stand for some hours, examining the few drops remaining in the beaker. The acetic acid having for the most part become volatilized, the characteristic vinous odor of pure wine vinegar would at this stage be very prominent, while that of cider vinegar would be entirely different. The odor of the two vinegars is very similar in their ordinary state. The peculiar fruity flavor of pure cider vinegar is very characteristic and not readily imitated by cheaper substitutes. Only a very slight turbidity should be produced in pure cider vinegar by the addition of either ammonium oxalate (absence of lime), barium chloride (absence of sulphuric acid or sulphate), and nitrate of silver (absence of hydrochloric acid or chlorides).

The character of the precipitate produced by neutral lead acetate should be particularly noted. Unless it is flocculent and copious, set-



ting out after a few minutes, cider vinegar is not pure, even if a marked turbidity is produced. Added apple jelly from exhausted apple pomace gives such a turbidity, and is to be suspected when not more than a cloudiness is produced on addition of the lead acetate reagent. Pure cider vinegar usually responds to both the lead acetate and the calcium chloride tests for malic acid. Van Slyke,\* Browne† and Mestrezat‡ have shown, however, that malic acid decreases during alcoholic fermentation and may during the subsequent acetous fermentation disappear largely or entirely.

**Wood Vinegar or Pyroligneous Acid** is sometimes rendered apparent by the empyreumatic or tarry taste and odor imparted to the product. When, however, the added acetic acid has been so purified that the tarry taste and odor are lacking, its presence may often be proved by the traces of furfural which always accompany it.

*Test for Furfural.*—A little of the vinegar is subjected to distillation, and to the first few drops of the distillate is added a little colorless anilin solution. A fading crimson color will be produced in presence of furfural. This reaction may sometimes be obtained upon the vinegar itself without distillation, if sufficient added wood vinegar be present.

The first portion of the distillate of wood vinegar reduces permanganate of potassium to a marked degree.

**The Addition of Spices** to vinegar in order to increase the pungency is best detected by first neutralizing the vinegar with sodium carbonate and then tasting. Under these conditions, the admixture of spices is rendered very apparent.

**Detection of Caramel.**—Considerable added caramel in vinegar is apparent from the unnaturally dark color and extremely bitter taste of the residue after evaporation.

Tests for caramel made on the vinegar residue, if long dried at the temperature of the water-bath, are not to be depended on as establishing the presence of added caramel, since at that temperature the decomposition of the sugar may in any event cause a positive test.

Caramel is detected by Crampton and Simon's and Amthor's tests (page 784). A further indication of caramel is the reducing power of the water solution of the precipitate obtained in Amthor's test.

**Examination for Metallic Impurities.**—*Lead and Zinc* are best looked for in the ash of the vinegar in cases where, like cider vinegar, the percent-

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\* N. Y. Agr. Exp. Sta., Bul. 258.

† Penn. Dept. of Agric. Rep., 1901, p. 128.

‡ J. Soc. Chem. Ind., 27, 1908, p. 763; 28, 1909, p. 734.



age of extract is high. A large volume of the vinegar is evaporated to substantial dryness over the water-bath. This may most readily be done in a 100-cc. platinum wine-shell, adding the vinegar in successive portions. To the residue add a small amount of sodium hydroxide, and burn to an ash in a muffle, or over a low flame, using potassium nitrate if necessary, a little at a time. Take up the ash in dilute hydrochloric acid, and examine for lead and zinc as in the case of canned goods.

In the case of vinegar low in extract, as in spirit vinegar, the sample may be evaporated to dryness, the residue dissolved directly in dilute hydrochloric acid without ignition, and the acid solution subjected to direct examination for lead and zinc.

*Copper* is best determined by electrolysis. 100 cc. of the vinegar are evaporated to a volume of about 10 cc. with a little sulphuric acid, filtered into a platinum dish, and subjected to electrolysis, using conveniently the apparatus described on page 634.

*Arsenic*.—Boil down a portion of the vinegar, to which concentrated nitric acid has been added, to a small volume, then add a few cubic centimeters of concentrated sulphuric acid, and continue the heating till fumes of sulphuric acid show the nitric to have been driven off. Cool, dilute with water, and test in the Marsh apparatus.



## CHAPTER XVII.

### ARTIFICIAL FOOD COLORS.

**THE** use of artificial dyestuffs in food products has greatly increased during recent years, both in degree and in variety of colors employed. Where formerly but a few well-known coloring matters, chiefly so-called vegetable colors and occasionally mineral pigments were used for this purpose, a vast array of dyes, chosen largely from the coal-tar colors, are now found in food, so that at present the exact identification of the particular dyestuff employed oftentimes presents a somewhat formidable problem to the analyst. The problem may consist in determining the class to which a commercial food color or combination of colors belongs, or it may consist in isolating the color itself, and afterwards identifying it as far as possible, for the purpose of determining whether or not it is harmless within the meaning of the law.

The effect of imparting to the cheaper varieties of jellies, jams, and ketchups which flood the market such intense and striking colors that these products in no wise resemble their pure uncolored prototypes, has a tendency in many cases to mislead the public into the idea that the genuine products are inferior by contrast, and to create a craving for unnaturally colored varieties. Indeed, the adherents to the free use of coloring matters in food assert that these brilliant hues please the eye and are hence legitimate.

**Objectionable Features.**—With the exception of confectionery and certain dessert preparations, in which dyes may be employed purely for æsthetic considerations only (a fact which is well understood by the consumer), the use of coloring matters in food is mainly for the purpose of deceiving as to their true character. The use of dyestuffs in food is objectionable on two accounts, first as introducing in some cases materials injurious to health, and second, in nearly all cases as deceiving the purchaser by concealing inferiority, or by making the goods



appear of greater value than they really are. In most states the food laws regarding employment of colors are so framed, that the presence of such colors constitutes an offense under one or the other of the above heads, mainly, however, because, by reason of their use, cheaper or inferior materials are made to masquerade for the higher or genuine grades, as, for instance, when alleged currant jelly is found to consist chiefly of apple-stock and commercial glucose, colored with an artificial red dye.

In such cases the analyst has merely to prove conclusively that an artificial color is present, even if he does not identify the dye itself. It is of course more satisfactory to at least show in addition whether the dye present is of vegetable origin, or is of the coal-tar variety, and in most cases this can readily be done, even if it is not easy to identify the exact color.

In localities where laws prevail stipulating that what are commonly known as "mixtures" or "compounds" to be legally sold, must be labeled with the names and percentages of ingredients, the law applies to coloring matters as well as other ingredients, and it may even be ruled in a strict interpretation of the law that the exact dye or dyes employed should appear on the label.

**Toxic Effects of Colors.**—Formerly the use of such pigments as chromate of lead was common in coloring confectionery, but lead chromate is rarely used at present. Other mineral pigments obviously unfit for use in food by reason of their well-known poisonous effects are those which contain salts of arsenic, mercury, lead, and copper. While most of the coal-tar colors are considered harmless in themselves, some are decidedly objectionable, and should not be used in foods. Under the latter class are included, first, those in connection with the manufacture of which arsenic, mercury, or other poisonous mineral ingredients have been used, such for example as arsenical fuchsin, and, second, those which are themselves inherently poisonous, as for instance picric acid. Fuchsin is now largely made without the aid of arsenic acid, and this variety is, perhaps, harmless. The toxic effects of many of the coal-tar colors have not been thoroughly established excepting in a negative way. Weyl has made many experiments on dogs and rabbits in which these animals have been fed with varying amounts of coloring material. In nearly all cases the doses far exceeded the amounts ordinarily taken in food, and the experiments are of value mainly in so far as they show harmless results of certain colors on the animal. It is to be regretted



that physiological experiments cannot more readily be tried on human beings, so as to study the effects of administering to them such amounts as are used in food.

More conclusive results (though still of a negative character) tending to establish the harmlessness of most of the coal-tar colors are given by Grandhomme\* in statistics showing the condition of health of laborers in factories where these dyestuffs are made, in comparison with those engaged in other industries where poisonous materials are handled. From these it appears that the proportion of illness among the anilin-makers is remarkably small.

In the case of coloring confectionery by the use of mineral pigments, a considerable amount of the coloring material must be used, forming without doubt a source of danger in some cases. With coal-tar dyes, on the contrary, the case is different. One ounce of auramine, for instance, has been found sufficient to give a deep-yellow color to 2,000 pounds of confectionery, so that almost an infinitesimal amount of the actual dyestuff is taken into the system. Hence it is that very little danger need be apprehended from the use of most coal-tar colors in food, objectionable as they certainly are as a commercial fraud.

**Injurious and Non-injurious Colors.**—Various countries have enacted specific laws regulating the use of coloring matters in foods, especially England, France, Germany, Austria, and Italy. In some cases attempts have been made to specify harmful and harmless colors. The National Confectioners' Association of the United States has compiled a useful classified list of injurious and harmless colors,† the classification being based largely on the results of experiments by Weyl and others, as well as on the Resolutions of the Association of Swiss Chemists, and on the French Ordinances regarding food colors. Eight years after the publication of this list Hesse‡ found that thirteen of the twenty-one organic dyes listed as harmful were on sale for coloring foods. The list is as follows, the names of the individual coal-tar dyes being those in Mathewson's

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\* Weyl, *Sanitary Relations of the Coal-tar Colors*, pp. 28-30.

† *Colors in Confectionery*. An Official Circular from the Executive Committee of the National Confectioners' Association of the U. S., 1899.

‡ *Coal-tar colors used in food products*, Washington, 1912.



table, (pages 868 and 875) and not in all cases the synonyms given the first place in the original list, and the numbers, those assigned in Green's English edition of Schultz and Julius' tables.\*

**Harmful Mineral Colors.**—Compounds of mercury, lead, copper, arsenic, antimony, tin, zinc, chromium, and barium, and preparations containing them.

**Harmful Organic Colors.**—*Red:* Biebrich Scarlet (163), Crocein Scarlet (160), New Coccin (106), Crocein Scarlet 8 B (169), Crocein Scarlet O extra (164), Saffranin (584). *Yellow:* Gum Gutta, Picric Acid (1), Martius Yellow (3), Resorcin Yellow (84), Victoria Yellow (2), Orange II (86), Metanil Yellow (95), Sudan I (11), Orange IV (88). *Green:* Naphthol Green B (398). *Blue:* Methylene Blue (650). *Brown:* Bismarck Brown (197), Bismarck Brown R (201), Fast Brown G (138), Chrysoidin (17, 18).

**Harmless Mineral Colors.**—*Green:* Ultramarine Green. *Blue:* Ultramarine Blue. *Violet:* Ultramarine Violet. *Brown:* Manganese Brown, Chocolate Brown and similar colors having as their basis natural or precipitated iron oxide free from arsenic.

**Harmless Organic Colors.**—*Red:* Cochineal Carmine, Carthamic Acid (from saffron), Redwood, Artificial Alizarin and Purpurin (534-537), Cherry and Beet Juices, Eosin (512), Erythrosin B (517), Rose Bengal (520), Phloxin (521), Ponceau 2 R (55), Ponceau R (55), Bordeaux B (65), Cerasin, Ponceau 2 G (15), Acid Magenta (462), Archil Substitute (28), Orange I (85), Congo Red (240), Azorubin S (103), Amaranth (107), Fast Red E (105), Crocein Orange (13) Fuchsin (448). *Yellow and Orange:* Annatto, Saffron, Safflower, Turmeric, Naphthol Yellow S (4), Brilliant Yellow (5), Crocein Orange (13), Acid Yellow G (8), Acid Yellow R (9), Azarin S (70), Orange I (85), Orange GT (43), mixtures of harmless red and yellow colors. *Green:* Spinach Green, Chinese Green, Malachite Green (427), Dinitrosoresorcin (394), mixtures of harmless blue and yellow colors. *Blue:* Indigo (689), Litmus, Archil Blue, Gentian Blue 6B (437), Coupier's Blue (600), in general such blues as are derived from triphenylrosanilin or from diphenylamin. *Violet:* Methyl Violet (451), Wool Black (166), Naphthol Black B (188), Azoblue (287), Mauvein (593), *Brown:* Caramel, Licorice, Chrysamin R (269).

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\* A Systematic Survey of the Organic Coloring Matters, founded on the German of Schultz and Julius. London, 1908.



## MINERAL COLORS.

It is impracticable to name all the mineral colors that might be added to food, as the list would include all known pigments. Even a list of the colors reported in the literature of the past generation as having been detected in foods would be of little value owing to changing conditions. Fortunately only a few comparatively harmless pigments, such as Prussian blue, ultramarine, and iron oxide, are now used to any considerable extent and these only in special classes of products.

The coloring of meat products and saccharine foods with pigments is taken up in Chapters VIII and XIV respectively, the facing of tea and coffee, in Chapter XI, and the greening of fruits and vegetables, in Chapter XXI.

**DETECTION OF MINERAL COLORS.**—Still more impracticable than to list the possible colors is to give adequate descriptions of methods for their detection and for the determination of the elements contained in them, as this would cover a wide field in qualitative and quantitative analysis.

In general it may be stated that the pigments appear as colored particles under the microscope and the chief elements occur in the ash prepared with special precautions. The pigments may be extracted from some foods by acids, alkali solutions, or other solvents, either directly or after evaporation.

Microscopic examination and microchemical tests of the sediment, obtained by shaking or dissolving the sample with water, are useful in the case of tea, coffee, sugar, confectionery, etc. Particles of the pigments making up the facing of tea may be found by examining the siftings from the sample under a lens.

Special methods for the detection of mineral colors are given in the chapters above mentioned; the following tests are for a few colors of common occurrence:

**Prussian Blue.**—This pigment is insoluble in water. It is decomposed and decolorized by treatment with potassium hydroxide. If the filtered alkaline solution of the coloring matter be treated with hydrochloric acid and ferric chloride, a precipitate of the original Prussian blue will be produced.

**Ultramarine Blue** is decolorized by hydrochloric acid with evolution of hydrogen sulphide, which blackens filter-paper moistened with lead acetate. Tests for the detection of both ultramarine and Prussian blue in tea are described on page 388 and in sugar on page 613.

**Chromate of Lead** has never been used to any extent in food products with the exception of confectionery. For its detection, see page 678.



## LAKES.

**Coal-tar Lakes.**—Berry\* has compiled lists of the coal-tar colors combined to form lakes, the mineral and organic substances used in their preparation, and the substances mixed with them to modify their color or properties. Over fifty dyes are listed but of these few are now used. Lakes of acid dyes are prepared chiefly with barium chloride, lead nitrate, lead acetate, zinc sulphate, aluminum sulphate, aluminum acetate, alums, tin chloride, antimony chloride, tartar emetic, double fluorides of antimony and sodium or potassium, calcium nitrate, and calcium acetate; those of basic dyes, with tannic acid, sodium phosphate, sodium arsenite, stannic and stannous acids and salts, antimony acids, resinic and various fatty acids. The principal materials used to modify the colors are barium sulphate, kaolin, calcium sulphate, infusorial earth, red lead, zinc oxide, lead sulphate, aluminum hydroxide, aluminum arsenite, barium phosphate, lead carbonate, calcium phosphate, lampblack and green earth.

**Lakes of Vegetable and Animal Dyes.**—The list given by Berry includes alum, ammonia, soda, and lime lakes of the following colors: buckthorn, Persian berries, yellow berries, quercitron, weld, gamboge, young and old fustic, barberry, annatto, turmeric, saffron, safflower, Indian yellow, Chinese yellow, cochineal (carmine), lac, dyewoods, indigo sulphonic acid, chlorophyl, lokao, and unripe Persian berries.

Alum lakes, particularly of cochineal, appear to be most used.

**DETECTION OF LAKES.**—Like inorganic pigments lakes are insoluble in water and therefore under the microscope appear as colored particles. The inorganic portion of a lake is tested for in the ash or charred mass, the organic portion whether of coal-tar, vegetable, or animal origin, by the usual tests after liberation by acid or alkali, according as the original color was acid or basic, and separation by dyeing or by immiscible solvents as described in subsequent sections.

## VEGETABLE AND ANIMAL COLORS.

These with a few mineral pigments were formerly almost exclusively used for coloring food products, and are still used to some extent.

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\* Coloring Matters for Foodstuffs and Methods for their Detection, U. S. Dept. of Agric., Bur. of Chem., Circ. 25, p. 7. Jennison, Manufacture of Lake Pigments, 1900.



**DETECTION OF VEGETABLE AND ANIMAL COLORS.**—Most of the soluble red colors of fruits and vegetables, according to L. Robin,\* react with ammonia to form a coloration, usually passing from violet to blue, then to a brownish green, when the ammonia is added little by little in excess to the color in solution while the yellow colors of such fruits as apples, peaches, plums, quinces, and apricots, according to Martin-Claude,† change to brown with ammonia.

**Dyeing Tests and Reactions on the Fiber.**—The natural colors of fruits and vegetables in an acid bath (page 841) impart scarcely any color to unmordanted wool or silk even by single dyeing. Most of the commercial vegetable dye stuffs also do not dye wool without a mordant, at least by the double dyeing method, while a few, notably the lichen colors (archil, cudbear, and litmus) impart a decided color although by no means of such a brilliant hue as many of the coal-tar dyes. Many of these colors dye cotton, previously mordanted by boiling in a solution of aluminum acetate or potassium bichromate, in a bath acidified with acetic acid.

Mathewson gives the reactions on the fiber of cochineal (page 855), azolitmin, the dyeing principle of litmus (page 855), and curcumin, the dyeing principle of turmeric (page 856). The reactions obtained by Loomis ‡ with twenty-one natural dyes fixed on wool or cotton appear in the table on page 819. In mordanting the fiber Loomis employs the following methods:

*Alum Mordanting.*—Dissolve 1 gram of crystallized aluminum sulphate and 1.2 grams of cream of tartar in 500 cc. of water. Stir 10 grams of fat-free wool in the solution for one hour, let stand two to three hours, wring, and dry at room temperature.

*Tin Mordanting.*—Dissolve 0.8 gram of tin crystals and 0.4 gram of oxalic acid in 500 cc. of water. Boil 10 grams of fat-free wool one and one-half hours in this solution.

*Chrom Mordanting.*—Heat to boiling 500 cc. of water containing 10 grams of fat-free wool, then add 0.2 gram potassium bichromate 0.35 gram of cream of tartar, and 0.1 cc. of concentrated sulphuric acid, and boil one and one-half hours. Dry at low temperature and keep from light.

**Extraction by Immiscible Solvent from Various Solutions.**—Mathewson, in connection with the table on page 868, makes the following statements:

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\* Girard et Dupre, *Analyse des Matières Alimentaires*, Paris, 1894, pp. 678, 679.

† Jour. pharm. chim., 13, 1901, p. 174.

‡ U. S. Dept. of Agric., Bur. of Chem., Circ. 63, pp. 47 and 48.



COLOR REACTIONS OF FIBERS DYED WITH NATURAL COLORS (LOOMIS).

Name of Color.	Kind of Fiber.	Reactions of Dyed Fiber with Reagents.			
		Concentrated Hydrochloric Acid.	Concentrated Sulphuric Acid.	10% Sodium Hydroxide Solution.	Ammonia Water, Sp. gr. 0.95.
Cochineal.....	Wool	No change	Brighter red	Magenta	Crimson
Cudbear.....	Wool	Deep pink	Dark gray	Violet	Violet
Archil.....	Wool	Deep pink	Blue-gray	Purple	Purple
Litmus.....	Wool	Pink	Brownish	Blue	Blue
Azolitmin.....	Wool	Pink	Brownish	Blue	Blue
Logwood.....	Cotton *	Red	Brown	Brown-black	Pale-brown
Brazil wood.....	Cotton *	Orange-red	Yellow-brown	Maroon	Purple
Barwood.....	Wool †	Salmon	Yellow-brown	Maroon	Blue-black
Catechu.....	Wool †	Brown	Dark brown	Brown	Brown
Spanish saffron.....	Wool †	Darker	Olive green, then maroon	No change	No change
Quercitron.....	Cotton †	No change	No change	No change	No change
Sumac.....	Wool †	No change	Yellow-brown	Brown-yellow	No change
Annatto.....	Wool †	Pale brown	Green	No change	No change
Turneric.....	Wool †	Crimson	Orange-brown	Orange	Orange
Persian berry extract.....	Wool †	Darker	Brown-yellow	Little darker	Darker
Fustic extract.....	Wool †	Orange-yellow	Yellow-brown	Orange-yellow	Orange-yellow
Weid extract.....	Wool †	No change	Brown-yellow	Little darker	Little darker
Buckthorn.....	Wool †	No change	Brown-yellow	No change	No change
Kalana.....	Wool †	No change	Darker	Brown-orange	No change
Poke berry.....	Wool †	Little change	Yellow-brown	Yellow	Yellow
Carthamin (safflower).....	Cotton	Orange	Brown	Yellow-brown	Salmon

\* Chrom-mordanted.

† Alum mordanted.



Colors of fustic, quercitron, Persian berries after hydrolysis, and alkanet are extracted in large part by amyl alcohol and amyl alcohol-gasoline (1 : 1) from N/64 acetic acid or N/64 to N hydrochloric acid, also by ether from N/64 hydrochloric acid, but not by amyl alcohol-gasoline or ether from N/64 sodium hydroxide solution. Annatto, not alkali treated, behaves similarly but is extracted in large part by amyl alcohol-gasoline from N/64 sodium hydroxide.

Colors of barwood, camwood and sandalwood resemble Nos. 483 and 510 in behavior, but are less soluble in aqueous solvents. They are extracted almost completely by amyl alcohol from salt solution and by amyl alcohol, amyl alcohol-gasoline, and ether from N/64 hydrochloric acid. They are not extracted by amyl alcohol-gasoline or ether from N/64 sodium hydroxide. Ether extracts the chief part from N but not from 4N hydrochloric acid. The color of Brazil wood is similar but more soluble in aqueous solvents. That of logwood is also similar but still more soluble. It is nearly all extracted by amyl alcohol from salt solution or N/64 hydrochloric acid, and the larger part by amyl alcohol-gasoline from N/64 hydrochloric acid. Only the smaller part is extracted by ether from N/64 and very little from 4N hydrochloric acid. A very small part is extracted by ether or amyl alcohol-gasoline from N/64 sodium hydroxide. The colors of archil, saffron, and cochineal also are easily extracted by amyl alcohol from slightly acid solutions but only in small amount by ether.

The colors of leaves, egg yolk, fats and oils, carrots, and tomatoes, all similar or identical, are taken up by ether from neutral solutions and not removed from this solvent by dilute alkali.

**Reactions in Aqueous Solution and with Sulphuric Acid.**—The table by Loomis\* on pages 822 and 823 gives the colors of a 0.1% solution of natural dyes as observed in a  $\frac{3}{4}$ -inch test-tube, the reactions of 10 cc. of the solution with 5 to 10 drops each of hydrochloric acid (sp.gr. 1.1), of 10% sodium hydroxide solution, and of ammonia water (sp.gr. 0.95), the reactions of 5 cc. of the solution with 0.2 gram of zinc dust and 10 drops of concentrated hydrochloric acid; also the colors obtained by shaking 0.05 gram of the dry color with 5 cc. of concentrated sulphuric acid and after dilution (cautiously with the first 20 cc.) until the change of color is merely in intensity.

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\* Loc. cit., pp. 59-61.



**SPECIAL TESTS FOR VEGETABLE COLORS.**—**Archil, Cudbear, and Litmus**, all derived from lichens, by the double dyeing method dye wool red in acid bath.\* The colored fiber is turned blue, purple, or violet by treatment with ammonia. For other reactions on the fiber see tables, pages 819 and 835.

*Robin's Test for Archil* in aqueous solution consists in shaking it with ether, which, if archil is present, is colored yellow. On treatment of the ether with ammonia, the yellow color is changed to blue, and, by adding acetic acid, goes over to a reddish violet. Other reactions of lichen colors are given on pages 820 and 822.

**Logwood**, according to Robin, in aqueous solution colors ether yellow, and on treating the ether with ammonia the color becomes red or faintly violet. Potassium bichromate gives a violet coloration, mingled with greenish yellow. If cotton is first mordanted by boiling with aluminum acetate, it is dyed violet when boiled in a solution of logwood. Reactions of chrom mordanted cotton dyed with logwood are given on page 819 and of the solution of the dye on page 822.

**Turmeric** is best extracted from a dry residue with alcohol, which it colors yellow. The color is transferred to a piece of filter-paper by soaking the paper in the alcoholic tincture, the paper is dried and dipped in a dilute solution of boric acid or borax slightly acidulated with hydrochloric acid. On again drying the paper, it will be of a cherry-red color if turmeric is present, and when touched with a drop of dilute alkali will turn dark olive. For solubilities of curcumin, the coloring principle of turmeric, see page 872, and for reactions on the fiber see page 856.

**Caramel.**—Care should be taken in testing for caramel not to subject the sample to long-continued heating, even on the water-bath. Indeed, caramel is sometimes developed spontaneously in saccharine food products during their process of manufacture when heat is used, by the charring of the sugar. If solutions are to be concentrated or brought to dryness before testing for caramel, this should be done in a vacuum desiccator over sulphuric acid, or at a temperature not exceeding 70°. For detection of caramel in milk, vinegar, and liquors, special tests are given elsewhere.

*Fradiss Test.*†—Extract the dried residue of the sample to be tested with warm, pure methyl alcohol, which, if caramel be present, is colored brown. Filter, and to the filtrate add amyl alcohol or chloroform. In

\* Tolman, Jour. Amer. Chem. Soc., 27, 1905, p. 213.

† Oestr. ungar. Zeits. Zucker Ind., 1899, 28, 229-231; Abs. Zeits. Unters. Nahr. Genussm., 2, 1899, p. 881.



APPEARANCE AND REACTIONS OF NATURAL COLORS IN AQUEOUS SOLUTION AND WITH CONCENTRATED SULPHURIC ACID (LOOMIS).

Name of Color.	Color of Aqueous Solution as Observed in $\frac{1}{4}$ " Test-tube.	Reactions of Aqueous Solution.				Dry Color + Concentrated Sulphuric Acid.	
		Hydrochloric Acid, Sp-gr. 1.10. (5-10 Drops.)	10% Sodium Hydroxide Solution. (5-10 Drops.)	Ammonia Water, Sp-gr. 0.95. (5-10 Drops.)	Zinc Dust + HCl. Exposed to Air on Filter Paper.	Before Dilution.	After Dilution.
Cochineal.....	Orange-red	Orange-yellow	Magenta	Magenta	Zinc dust and HCl. orange-yellow. Color not restored.	Pink	Yellow-pink, then straw
Cudbear.....	Lilac	Yellow-pink	Purple	Purple	Color restored	Purple	Red-brown
Archil.....	Deep lilac	Yellow-pink	Purple	Purple	Color restored	Purple	Red, red-brown, and red-orange
Litmus.....	Blue	Pink	No change	No change	.....	Purple	Orange-red, then pink
Azo litmin.....	Dark crimson	Orange-red	Purple	Purple	.....	Purple	Orange-red, then pink
Logwood.....	Brown-yellow	Orange	Dark brown	Light brown	Color not restored	Yellow-brown	Paler
Brazil wood.....	Red orange, slight fluorescence	Orange, not fluorescent	Crimson	Crimson	Color restored	Brown-yellow, fluorescent	Yellow, not fluorescent
Barwood.....	Insoluble	Color precipitated on acidifying alkaline solution	NaOH solution, deep brown-red	Ammonia solution, deep brown-red	NaOH solution + Zn dust, decolorized; on exposure, pinkish, then colorless	Orange-brown	Pink
Catechu (E and A)...	Yellow-brown	Paler, cloudy	Dark orange-brown	No change	.....	Brown-red	Paler
Spanish saffron.....	Yellow	No change	Paler	No change	Color not restored	Blue, then purple, maroon, and red-brown	Yellow, then nearly colorless



Name of Color.	Color of Aqueous Solution as Observed in 1" Test-tube.	Reactions of Aqueous Solution.				Dry Color + Concentrated Sulphuric Acid.	
		Hydrochloric Acid, Sp.gr. 1.10. (5-10 Drops.)	10% Sodium Hydroxide Solution. (5-10 Drops.)	Ammonia Water, Sp.gr. 0.95. (5-10 Drops.)	Zinc Dust + HCl, Exposed to Air on Filter Paper.	Before Dilution.	After Dilution.
Quercitron.....	Brown-yellow	Slightly redder	Orange-brown	Orange-brown	Not decolorized	Yellow	Yellow
Sumac.....	Dirty yellow	No change	Green-brown	Yellow-brown	NaOH solution + Zn dust, decolorized; on exposure, pinkish, then colorless. Zn + NaOH, not decolorized	Yellow	Yellow
Annatto.....	Yellow in alkaline solution	Paler	.....	.....	.....	Blue	Mauve
Turmeric.....	Yellow in alkaline solution	Paler	.....	.....	Zn + NaOH, not decolorized	Orange	Dirty yellow
Persian berry extract.	Yellow	No change	Orange	Deeper	Not decolorized	Yellow	Yellow
Fustic extract.....	Yellow	No change	Brown-orange	Orange	Not decolorized	Yellow	Yellow
Weld extract.....	Yellow	Paler	Deeper	Deeper	Not decolorized	Yellow	Yellow
Buckthorn.....	Yellow	No change	Red-brown	Yellow-brown	Not decolorized	Yellow	Yellow
Kamala.....	Yellow	Dilute alcohol solution of color paler and cloudy	Yellow-brown	Brown-yellow	NaOH solution of color, red; + zinc dust, orange Original color not restored	Orange	Nearly colorless solution and orange precipitate
Pokeberry.....	Crimson	Magenta	Orange-yellow	Magenta turning orange-brown	Color not restored	Orange-brown	Brown



presence of caramel, a brown flocculent precipitate is formed, which slowly settles to the bottom of the tube.

*Amthor Test.\**—Mix in a cylinder 10 cc. of the solution, 30 to 50 cc. of paraldehyde, and sufficient absolute alcohol to make the liquids miscible. After the brown caramel precipitate has settled decant off the liquid, wash with absolute alcohol, dissolve in a few cc. of hot water, and filter. Note the intensity of the color of the solution, then pour into a freshly prepared solution of two parts of phenylhydrazine hydrochloride, 3 parts of sodium acetate and 20 parts of water. A considerable amount of caramel will give a precipitate in the cold. Heating hastens the separation and long standing is essential if the amount is small.

Lasché's modification of this test is described on page 784.

**Indigo**, both natural and synthetic, is insoluble in alcohol and in water and therefore suited only for solid foods. It has been used for coloring confectionery and facing tea. With concentrated sulphuric acid the dry material becomes yellowish changing slowly to blue-green.

*Sulphonated Indigo*, also known as indigo carmine, indigo extract, indigotine, and indigo disulpho acid, being soluble in both alcohol and in water and allowed under federal ruling, is the basis of most blue food colors and also mixed with red and with yellow dyes of violet and green shades respectively. Its reactions in solution and on the fiber are given on pages 868 and 854.

**Cochineal.**—This animal dyestuff is used in ketchups, cordials, confections, and other food products.

*Robin Test.*—Acidulate the aqueous solution with hydrochloric acid, and shake out in a separatory funnel with amyl alcohol. Cochineal imparts to this solvent a yellowish color, the depth depending on the amount present. Wash the separated amyl alcohol with water till neutral, and divide into two portions. To one of these add a little water, and then drop by drop a solution of uranium acetate, shaking each time a drop is added. In presence of cochineal the water is colored a very characteristic emerald-green color. To the other portion add ammonia. If cochineal has been used, a violet coloration is produced.

#### COAL-TAR COLORS.

So many of the coal-tar dyes can be used in food products that it would be impossible to even name them all, especially in view of the fact that

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\* Zeits. Anal. Chem., 24, 1885, p. 30.



new colors are from time to time being added to the list. No attempt will be made in the present work to give the nature and composition of the dyes named, as such descriptions would lead beyond its scope. For detailed information along this line the reader is directed to the works of Schultz and Julius, Green, Mulliken, etc.

Green's list compiled in 1903 includes 688 coal-tar colors but does not claim to be complete. Mulliken described 1475 dyes found on the American market in 1909. Although some natural dyes and possibly a few mixtures are included the total of these is more than offset by definite individuals of coal-tar origin which are not enumerated because obsolete or for other reasons not available or have been discovered since the date of publication.

Various classifications of these colors are attempted, based on (1), their origin, as *anilin dyes*, *naphthalin dyes*, *anthracene dyes*, etc.; (2) their chemical composition, as *nitro*, *nitroso*, *azo*, *diazo*, *azin*, and other compounds; (3), their solubility in water and other solvents; and (4), their mode of application to the fiber, as *basic dyes*, *acid dyes*, *direct cotton dyes*, *mordant dyes*, etc.

These dyes are sold in the form of powder, and are readily made into solutions for food colors in the case of the water-soluble varieties, and into pastes in the case of the insoluble forms. Most of the coal-tar colors employed in foods are naturally of the soluble variety, especially such as are found in jellies, jams, fruit products, canned foods, ketchups, beverages, and milk. Pastes made from insoluble dyes are adapted mainly for exterior coatings of hard substances such as candies. Colors in the dry form are to be looked for in such spices as cayenne, mustard, and mace, but a commoner method of coloring these spices high in oil is to mix with them a solution of the color in oil (usually cottonseed). Oil solutions of coal-tar dyes are also employed for coloring butter and oleomargarine.

The chief concern of the food analyst, as regards artificial color is its recognition in food products. Coal-tar dyes may usually be identified as such, but it is not always possible to name the particular individual dye or combination of dyes employed, even though the class to which they belong may be determined. One reason for this is that not infrequently mixtures of two or more colors are employed.

**Coal-tar Colors Allowed under the Federal Law.\***—The use of any dye, harmless or otherwise, to color food in a manner whereby damage

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\* Food Inspection Decisions, Nos. 76, 77, 106, 117, 129, and 164.



or inferiority is concealed is in violation of Sec. 7 of the Food and Drugs Act of June 30, 1906. The addition of all mineral or metallic dyes, and all coal-tar dyes, other than those specially provided for, is also prohibited. Pending further investigation the following coal-tar colors are permitted in foods, provided they are certified to be true to name and to be free from mineral and metallic poisons, harmful organic constituents, and contaminations due to improper or incomplete manufacture:\*

*Red Shades.*—107. *Amaranth* [M.] [C.]. Synonyms: *Fast red D* [B.] *Bordeaux S* [A.], *azoacidrubine 2B* [D.], *fast red EB* [B.].

56. *Ponceau 3R* [A.] [B.] [M.]. Synonyms: *Ponceau 4R* [A.], *cumidin red*, *cumidin ponceau*.

517. *Erythrosin* [B.] [M.] [B.S.S.]. Synonyms: *Erythrosin D* [C.], *erythrosin B* [A.], *pyrosin B* [Mo.], *iodosin B*, *eosin bluish*, *eosin J* [B.].

*Orange Shade.*—85. *Orange I*. Synonyms: *Alphanaphthol orange*, *naphthol orange* [A.], *tropæolin 000 No. 1*, *orange B* [L.].

*Yellow Shades.*—4. *Naphthol yellow S* [B.]. Synonyms: *Naphthol yellow*, *acid yellow S*, *citronin A* (L.).

94. *Tartrazin* [B.] [I.] [H.]. Synonym: *Hydrazin yellow* [O.].

*Green Shade.*—435. *Light green SF yellowish* [B.]. Synonyms: *Acid green* [By.] [M.] [T.M.] [O.], *acid green extra conc.* [C.].

*Blue Shade.*—692. *Indigo disulphoacid*. Synonyms: *Indigo carmine*, *indigo extract*, *indigotine* [B.], *sulphonated indigo*.

None of these colors is patented, hence their manufacture is not likely to become a monopoly. They may be used in combinations, thus securing any desired shade. For example, violet may be obtained by mixing indigo disulphoacid and one of the red colors, a blue-green by mixing indigo-disulphoacid with naphthol yellow S or light-green SF and so on.

**EXAMINATION OF COAL-TAR FOOD COLORS.**—The testing of synthetic colors designed for foods differs from the task confronted in the dyeing industry in that the number of dyes is more limited and the presence of injurious substances, especially metallic by-products, is of paramount importance. A knowledge of the probable and possible dyes is naturally a great aid in examining samples; with this information the various analyti-

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\* The numbers preceding the dyes are those given by Green; the letters in brackets represent the manufacturers who originated the names.



cal schemes and tables covering the whole field can be used to best advantage. These same data are also of value in detecting colors in foods, and on the other hand, the tables of solubilities and reactions, as well as general methods, designed especially for the examination of foods for foreign colors, apply also to the food colors themselves.

**Analytical Schemes.**—Witt,\* the pioneer in the identification of dyes, devised a scheme employing the reactions and color tests with acids, alkalies, and other reagents, reduction with zinc dust and subsequent oxidation by exposure to air, as well as dyeing tests, and spectroscopic examination. As a means of learning whether or not a dye was a mixture, he devised the very useful test of dusting the powder over concentrated sulphuric acid, noting the color as the individual particles dissolved.

Weingärtner † revised Witt's scheme, adding new dyes and employing tannin solution to differentiate acid and basic colors. In testing for mixtures he sprinkled the powder over a filter paper moistened with water.

Green ‡ introduced chromic acid as an oxidizing agent, following reduction with zinc dust, but later, with his associates Yoeman and Jones, § rejected both reagents in favor of sodium hydrosulphite ("T blankite") and potassium persulphates, both of which are colorless.

Rota in his scheme (pages 827 to 832) departs quite radically from class reactions developed by Witt and others of his school.

Of special value are the tables of Green || (based on Schultz and Julius), and Mulliken. ¶ The latter has the advantage of being more recent, more distinctly analytical, and broader in its application owing to the greater number of dyes included and the wider variety of tests (including spectroscopic) employed.

**Rota's Analytical Scheme** \*\* is based on the structure of the dyes as shown by their reactions with certain reagents. The colors are divided into two main groups, according to whether or not they are reducible by stannous chloride. These two groups are each further subdivided into

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\* Zeits. anal. Chem., 26, 1887, p. 100.

† Ibid., 27, 1888, p. 232.

‡ Jour. Soc. Chem. Ind., 12, 1893, p. 3.

§ Jour. Soc. Dyers, Colorists, 9, 1905, p. 236.

|| A Systematic Survey of the Organic Coloring Matters, London, 1904.

¶ Identification of Pure Organic Compounds, Vol. VI, New York, 1917.

\*\* Chem. Ztg., 22, 1898, p. 437.



two classes, the reducible colors being classed according to whether the color remains unchanged, or is restored by treatment with ferric chloride, and the non-reducible colors according to their action with potassium hydroxide.

The tests are carried out on a dilute aqueous or alcoholic solution of the coloring matter, the strength being about 1 in 10,000. Treat about 5 cc. of this solution with 4 or 5 drops of concentrated hydrochloric acid and about as much 10% stannous chloride solution, shake the mixture, and heat if necessary to boiling. With some colors the process of decolorization is a slow one, especially if the solution is too concentrated, and it is well to repeat the experiment, if in doubt, diluting the original sample still further with water. Tin in solution in concentrated hydrochloric acid may be employed instead of stannous chloride, if desired.

Here, as in all cases of color testing, it is well to make comparative tests with known colors.

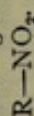
#### CLASSIFICATION OF ORGANIC COLORING MATTERS.

[A portion of the aqueous or alcoholic solution is treated with HCl and  $\text{SnCl}_2$ .]

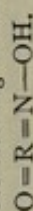
Complete decolorization. Reducible coloring matters. Colorless solution is treated with $\text{Fe}_2\text{Cl}_6$ or shaken with exposure to air.		The color changed no further than with HCl alone. Nonreducible colors. A part of original solution is mixed with 20% KOH and warmed.	
The liquid remains unchanged. Coloring matters not re-oxidizable.	The original color restored. Reoxidizable coloring matters.	Decolorization, or a precipitate. Imido-carbo-quinone coloring matters.	No precipitation. Liquid becomes more colored. Oxy-carbo-quinone coloring matters.
CLASS I.	CLASS II.	CLASS III.	CLASS IV.
Nitro, nitroso, and azo colors, including oxyazo and hydrazo colors. Picric acid, naphthol yellow, ponceau, Bordeaux, and Congo red.	Indogenide and imido-quinone coloring matters, methylene blue, safranin, indigo carmine.	Amido-derivatives of di and triphenyl methane, auramins, acridins, quinolins, and color derivatives of thio benzenil. Fuchsin, rosanilin, auramin.	Nonamide diphenyl methane, oxy-ketone, and most of natural organic coloring matters. Eosins, aurin, alizarin.



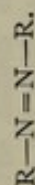
## CHARACTERISTICS OF ORGANIC COLORING MATTERS.

CLASS I.—REDUCED BY HCl+SnCl<sub>2</sub> AND NOT REOXIDIZABLE.*Types.**Nitro-coloring matters.*

Yellow or orange, soluble in water. Wool and silk dyed directly, but not cotton. The aqueous solution shows tendency to decolorization with HCl. With HCl+SnCl<sub>2</sub> partially reduced, giving red nitro-amido derivatives (nitramines), or nitro-phenols turning red in KOH.

*Nitroso-coloring matters.*

Brown or green, usually insoluble in water; indirect for fibers; with H<sub>2</sub>SO<sub>4</sub>+CHOH give blue color (Liebermann's reaction).

*Azo-coloring matters.*

Their aqueous solution decomposed with KOH and extracted with ether gives an ethereal extract with annexed characteristics.

Nitramines; soluble in ether in the presence of KOH.	$-N=R-N\begin{smallmatrix} O \\ \diagup \\ OH \end{smallmatrix}$	{ e.g., Aurantia.
Nitro-phenols; insoluble in ether in the presence of KOH.	Nonsulphonated; soluble in ether in { $O=R-N\begin{smallmatrix} O \\ \diagup \\ H \end{smallmatrix}$ } presence of acetic acid.	{ Victoria yellow.
Sulphonated; insoluble in ether.		Naphthol yellow S.
Nonsulphonated; insoluble in water; soluble in alcohol; soluble in ether in presence of acetic acid.		{ Dioxin (L).
Sulphonated; soluble in water; insoluble in ether.		Naphthol green.
Colored; shaken with dilute acetic acid yields to it the original color. Basic coloring matters.	{ Nonsulphonated amido-azo coloring matters.	{ $-N=R=N-NHR_1$ . Bismarck brown.
Colored solution; not yielding its color to dilute acetic acid. Neutral coloring matters.	{ Oxyazo coloring matter, without carboxyl.	{ $O=R=N-HR_1$ . Sudan 1 (A).
Nor sulphonated; extracted by ether from dilute solution in acetic acid.	{ Oxyazo coloring matters, with carboxyl group.	{ Indirect for cotton wool. Direct for cotton wool. } Diamond yellow (By) Chrysamin.
Colorless solution; yields nothing to acetic acid. Acid coloring matters.	{ Sulphonated; not extracted by ether from solution in dilute acetic acid.	{ Indirect for cotton wool. Direct for cotton wool. } Bordeaux B (A). Azo blue (A).
{ Sulphonated; not extracted by ether from solution in dilute acetic acid.	{ Nonamido compounds; unaltered by HNO <sub>3</sub> . } Amido compounds; changed by HNO <sub>3</sub> .	{ Indirect for cotton wool. Direct for cotton wool. } Solid yellow N (P). Congo red (A).



CLASS II.—REDUCED BY  $\text{HCl} + \text{SnCl}_2$  AND REOXIDIZABLE.

The aqueous or alcoholic solution is treated with KOH and extracted with ether. The ethereal solution washed with water has the annexed characteristics.		Types.		
<p>The ethereal solution is colored or colorless, and yields the original color to 5% acetic acid.</p> <p>Basic coloring matters fixed on wool in alkaline bath.</p>	<p>The solution is readily reduced by HCl + SnCl<sub>2</sub> in the cold.</p>	<p>Oxyazines (no sulphur).</p>	<p>Nile blue A (B).</p>	
		<p>Thiazins (sulphur).</p>		
		<p>Indulins; blue color with concentrated H<sub>2</sub>SO<sub>4</sub>. Blue on dilution.</p>	<p>Indulin soluble in alcohol.</p>	
<p>Colored; does not yield the color to acetic acid.</p> <p>Neutral coloring matters. Insoluble in water. Soluble in alcohol. Fixed on fibers in bath.</p>	<p>Blue coloring matters changed by HCl on warming.</p>	<p>Safranins; green color with H<sub>2</sub>SO<sub>4</sub>. On dilution blue, then violet.</p>	<p>Safranin T extra (A).</p>	
		<p>Indophenols.</p>		
		<p>Red or blue coloring matters. Unaltered by HCl. With HNO<sub>3</sub> yield isatin.</p>	<p>Indogenides.</p>	
<p>Uncolored; yields nothing to acetic acid.</p> <p>Acid coloring matters. Soluble in water. Fixed on wool in acid bath.</p>	<p>Nonsulphonated. Soluble in ether in presence of acetic acid.</p>	<p>Oxazones.</p>	<p>Fluorescent blue; orcein.</p>	
		<p>Sulphonated. Insoluble in ether under all circumstances.</p>	<p>Reduced by SnCl<sub>2</sub> + HCl.</p>	<p>Sulphonated indogenides. Indigo carmine. Thiocarmine R (C).</p>
		<p>Not reduced by SnCl<sub>2</sub> + HCl.</p>	<p>Sulphonated indulins.</p>	<p>Soluble nigrosin.</p>



CHARACTERISTICS OF ORGANIC COLORING MATTERS—(Continued).  
 CLASS III.—COLORING MATTERS NOT REDUCED BY  $\text{SnCl}_2 + \text{HCl}$ . CONTAINING THE IMIDO-QUINONE CARBON CHROMOPHORE— $\text{N}=\text{R}=\text{C}=\text{}$ .  
*Types.*

<p>The ethereal solution is colorless or colored. The color is yielded to 5% acetic acid.</p> <p>Basic coloring matters. Fixed on wool in alkaline bath (<math>\text{NH}_3</math>).</p>	Colorless, nonfluorescent ethereal solution. Yellow color yielded to acetic acid nonfluorescent. The aqueous solution is decolorized by KOH and decomposed by HCl.	Auramins.	$\left\{ \begin{array}{c} \text{R} \\ \text{C}=\text{N}- \\ \text{R} \end{array} \right\}$	E.g. Auramin O (B).
	Colorless, ethereal solution. Green fluorescence. Aqueous solution precipitated by KOH, hardly altered by HCl. Turns red with $\text{HNO}_3$ .	Acridins.	$\left\{ \begin{array}{c} \text{R} \\ \text{C} \\ \text{R} \end{array} \right\}$	Phosphin.
	Colorless, or colored ethereal solution. Nonfluorescent. Color yielded to acetic acid—reddish violet, blue, and green without fluorescence. Aqueous solution usually decolorized on warming with KOH, and colored yellow by HCl (excepting fuchsin).	Fuchsins (nonsulphonated).	$\left\{ \begin{array}{c} \text{R} \\ \text{C}-\text{R} \\ \text{R}=\text{N}- \end{array} \right\}$	Fuchsin.
	Ethereal solution colorless and nonfluorescent. Acetic acid colored rose and fluoresces.	Pyronins (colored yellow by HCl. Direct for cotton wool).	$\left\{ \begin{array}{c} \text{R} \\ \text{C} \\ \text{R} \end{array} \right\}$	Pyronin (G).
	Aqueous solution decolorized with KOH.	Rhodamins (non-sulphonated. Unaltered by HCl).		Rhodamin S (By).
<p>The colored ethereal solution does not yield its color to acetic acid.</p> <p>Neutral coloring matters. Insoluble in water. Soluble in alcohol.</p> <p>Ethereal solution colorless. Yields nothing to acetic acid.</p> <p>Acid coloring matters. Soluble in water. Fixed on wool in acid bath (HCl).</p>	The ethereal solution is yellow, and nonfluorescent. Alcoholic solution yellow, nonfluorescent, and unaltered by aqueous acids and alkalis.	Quinone-phthalones.	$\left\{ \begin{array}{c} \text{R} \\ \text{C} \\ \text{R}=\text{N} \end{array} \right\}$	Quinolin yellow A (soluble in alcohol).
	Yellow coloring matters. No fluorescence in water. Unaltered by aqueous acids and alkalis.	Sulphonated quinone-phthalones.		Quinolin yellow A (soluble in water).
	Reddish violet, blue, or green coloring matters. Usually decolorized by KOH, little changed by HCl.	Sulphonated fuchsins.		Fuchsin S (B).
	Red or violet coloring matters. Soluble in water with fluorescence. Precipitated by HCl. Changed but little, or not at all, by KOH.	Sulphonated rhodamins.		Violamin R (M).
	Brownish yellow or orange coloring matters. Aqueous solution $\pm$ fluorescent. Fixed directly on silk, wool, and cotton.	Thiazoles.	$\left\{ \begin{array}{c} \text{C}=\text{N} \\   \\ \text{S}-\text{R} \end{array} \right\}$	Primulin (B).

The aqueous or alcoholic solution treated with KOH and extracted with ether.



CLASS IV.—COLORING MATTERS NOT REDUCED BY $\text{SnCl}_2 + \text{HCl}$ CONTAINING THE OXY-QUINONE CARBON CHROMOPHORE $\text{O}=\text{R}-\text{C}=\text{O}$ .			
		Types.	
Remains unaltered. Nonamido triphenylmethane coloring matters. Usually soluble in water and direct for wool.	The coloring matter is dissolved or suspended in boiling water.	Aurins.	$\begin{array}{c} \text{R}_1 \\ \diagup \\ \text{C}-\text{R}_1 \\ \diagdown \\ \text{R}=\text{O} \end{array}$ Aurin.
		Phthaleins.	$\begin{array}{c} \text{R}_1 \\ \diagup \\ \text{C}-\text{R}_1 \\ \diagdown \\ \text{R}=\text{O} \end{array}$ Eosin.
Changes to green or olive green. Oxy-ketone coloring matters. Most of them insoluble in water and indirect for fibers.	Dissolves with yellow or reddish yellow color. Monoketones.	Benzophenones.	$\begin{array}{c} \text{R} \\ \diagup \\ \text{CO} \\ \diagdown \\ \text{R} \end{array}$ Alizarin yellow A (B).
		Flavones.	$\begin{array}{c} \text{R} \\ \diagup \\ \text{CO} \\ \diagdown \\ \text{C}=\text{C} \\   \\ \text{I} \end{array}$ Quercetin.
The original coloring matter is treated with faintly alkaline water (KOH, 1 per cent). Dissolves with red, reddish violet, green, or blue color. Diketones (quinones).	The alkaline solution treated with excess of HCl. The free coloring matter precipitated. Usually soluble in ether, and indirect for fibers.	Nonsulphonated anthraquinones.	$\begin{array}{c} \text{R} \\ \diagup \\ \text{CO} \\ \diagdown \\ \text{CO} \\ \diagup \\ \text{R} \end{array}$ Alizarin.
		Sulphonated anthraquinones.	Sulphonated alizarin (alizarin red).

The alcoholic solution of the coloring matter treated with a few drops of a dilute solution of  $\text{Fe}_2\text{Cl}_6$  (1:1000).



**Separation and Identification of Allowed Colors.**—Price\* devised a method for the identification of the seven colors allowed under Food Inspection Decision 76, 1907. The addition of tartrazin to the list necessitated a modification of the method.

*Price-Estes Method.*†—The scheme is given on page 834.

*Price-Ingersoll Method.*‡—Ingersoll states with regard to the Price-Estes method that when small amounts of the dye are taken out of the original mixture on extraction with the ammonium sulphate reagent the separation is difficult or impossible, furthermore, that while some tartrazin, like naphthol yellow S, is soluble in that reagent, the larger part is not, following Price's directions. He accordingly proposed the following modification of the Price method:

Rub from 0.1 to 0.2 gram of the dye sample, depending upon the amount of foreign salts in the mixture, with 25 cc. of saturated ammonium sulphate solution in a mortar and filter through a dry filter. If the filtrate comes through red, wash the color residue in the mortar and on the filter with successive 10 to 15 cc. portions of the ammonium sulphate solution until the washings are no longer colored red. The filtrate and washings contain the greater part of the amaranth together with some naphthol yellow S and also some tartrazin. Combine the filtrate and washings and shake with successive portions of acetic ether until the acetic ether is no longer colored yellow. The acetic ether removes that portion of the naphthol yellow S which was dissolved by the ammonium sulphate solution and may be discarded, since the greater part of this dye is recovered later in the scheme. Shake the ammonium sulphate solution containing amaranth and some tartrazin with acetone to remove these colors; discard the ammonium sulphate solution, dilute the acetone portion with an equal volume of water, and drive off the acetone on a steam bath. Saturate with sodium chloride, add 10 cc. of alumina cream, agitate, warm, settle, filter, and wash with warm saturated sodium chloride solution until the washings are no longer colored yellow. To recover the amaranth, suspend the alumina cream precipitate in saturated ammonium sulphate solution and shake with acetone.

The filtrate contains tartrazin and is to be discarded, or, when dealing with small amounts, if desired, can be saved and the tartrazin identified with the greater portion of this color separated further in the scheme.

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\* U. S. Dept. of Agric., Bur. of Anim. Ind., Circ. 180, 1911.

† Jour. Ind. Eng. Chem., 8, 1916, p. 1123.

‡ Ibid., 9, 1917, p. 955.



## PRICE-ESTES SCHEME FOR SEPARATING THE COAL-TAR COLORS ALLOWED BY U. S. RULINGS.

Rub approximately 0.2 gram of sample in a mortar with supersaturated  $(\text{NH}_4)_2\text{SO}_4$  solution,\* filter and wash with the  $(\text{NH}_4)_2\text{SO}_4$  solution until washings are no longer colored red.

<p>Filtrate contains Tartrazin, Amaranth, and small amount Naphthol Yellow S. Shake with acetic ether to remove Naphthol Yellow S and discard this solution. Remove the Tartrazin and Amaranth from the <math>(\text{NH}_4)_2\text{SO}_4</math> solution by shaking with acetone, dilute with <math>\text{H}_2\text{O}</math>, evaporate off the acetone on the water-bath, supersaturate with NaCl, filter, and wash with supersaturated NaCl solution until the filtrate is no longer yellow.</p>	<p>Residue contains Erythrosin, Naphthol Yellow S, Light Green S F Yellowish, Orange 1, Ponceau 3 R, and Indigo Disulfo Acid. Dissolve in <math>\text{H}_2\text{O}</math>, acidify with 5% acetic acid, and shake with ether.</p>	<p>Ether contains Erythrosin. Wash with <math>\text{H}_2\text{O}</math>, and then shake with dilute <math>\text{NH}_4\text{OH}</math>, 1:100, which removes the Erythrosin. The excess of <math>\text{NH}_3</math> is then driven off.</p>	<p><math>\text{H}_2\text{O}</math> solution is freed of ether and saturated with NaCl on steam bath, cooled, filtered, and washed with supersaturated NaCl solution * until washings are colorless.</p>	<p>Filtrate contains Light Green S F Yellowish and Naphthol Yellow S. Shake with acetone. Separate acetone and salt solution.</p>	<p>Filtrate contains Tartrazin, Add excess of conc. HCl or acetic acid and shake with acetone. Dilute the acetone solution with <math>\text{H}_2\text{O}</math> and evaporate off the acetone.</p>	<p>Residue contains Amaranth.</p>	<p>Acetone removes Naphthol Yellow S. Wash with supersaturated NaCl solution. Remove excess of acetone, add <math>\text{H}_2\text{O}</math> to salt by evaporating the acetone, and taking up with alcohol and test for on steam bath. Acidify with acetic acid; shake with a methyl alcohol which removes any traces of Orange 1 present, and then test the acidified water solution for Naphthol Yellow S.</p>	<p>NaCl solution contains Light Green S F Yellowish. Remove excess of saturated NaCl solution by evaporating and then taking up with <math>\text{H}_2\text{O}</math> solution and drive off alcohol which takes out the Orange 1.</p>	<p>Acetic ether contains Orange 1. Wash with supersaturated NaCl solution and then take out the blue color is destroyed. Filter.</p>	
<p>Tartrazin.</p>	<p>Erythrosin.</p>	<p>Naphthol Yellow S.</p>	<p>Light Green S F Yellowish.</p>	<p>Orange 1.</p>	<p>Filtrate contains Indigo Disulpho Acid. It is treated with 3% <math>\text{H}_2\text{O}_2</math>, which should give a deep blue color if Indigo Disulpho Acid is present.</p>	<p>The precipitate contains Ponceau 3 R. It is washed with <math>\text{CaCl}_2</math> solution until free from blue color. Dissolve in dilute <math>\text{NH}_4\text{OH}</math>. The excess of <math>\text{NH}_3</math> is then driven off.</p>	<p>Ponceau 3 R</p>			

\* Prepared by agitating an excess of the salt in water on a steam bath for several hours and cooling to room temperature. The solution should always contain undissolved crystals and be agitated previous to use.

† Dissolve 30 grams of granulated tin, free from iron, in 125 c.c. conc. HCl, dilute to 250 c.c., and filter through asbestos. Dilute with 250 c.c. HCl (sp.gr. 1.124) and 500 c.c.  $\text{H}_2\text{O}$ .



Dissolve in water the portion of the original sample not dissolved by ammonium sulphate, acidify with acetic acid, and shake with successive portions of ethyl ether until the ether is no longer colored. The ether contains erythrosine, which it is very essential to remove completely from the other dyes before proceeding further. Wash the ether solution several times with water and finally extract the erythrosine from the ether with dilute ammonia solution. Remove the ammonia by evaporation on the steam bath and observe if this solution, when very dilute, has any fluorescence which might indicate the presence of prohibited colors having similar reactions. Remove the ether from the acetic acid aqueous solution by warming on a steam bath, saturate with sodium chloride at steam bath temperature, and add sodium chloride in excess; cool and filter through a dry filter. Wash with saturated sodium chloride solution until the washings are colorless. When a bulky precipitate is obtained here, which is difficult to wash, it may be time-saving to redissolve the precipitate and excess salt in water and repeat the salting and washing process, adding the filtrate and washings to those of the first saturation. The combined filtrate and washings contain light green SF yellowish, naphthol yellow S, tartrazin, traces of orange I, and possibly amaranth, since the latter dye may not be entirely removed by the first extraction of the dry sample with the ammonium sulphate reagent.

To separate the naphthol yellow S, extract with successive portions of acetone until the acetone fails to remove any more color. Combine the acetone extracts and wash with several portions of saturated sodium chloride solution to remove traces of tartrazin and light green SF yellowish from the acetone. Add to the acetone solution an equal volume of water and drive off acetone on the steam bath. Acidify the water solution and shake with amyl alcohol to remove traces of orange I that may be present; discard this amyl alcohol solution. Drive off all amyl alcohol mechanically held in the aqueous solution by warming on the steam bath and test this solution for naphthol yellow S.

To separate the light green SF yellowish from the tartrazin, remove the acetone from the aqueous salt solution by heating on the steam bath, and add fuller's earth in the proportion of 0.5 gram to each 10 cc. of warm dye solution. After mixing well and heating, allow to settle; then filter and wash with water. The light green SF yellowish remains on the filter and can be dissolved in strong, hot acetic acid and further identified. If tartrazin was present in the original mixture, the filtrate from the precipitation of the light green SF yellowish will be yellow or golden yellow, not



decolorized by hydrochloric acid. Imperfect removal of naphthol yellow S, previously, would result in a yellow filtrate here which could be decolorized by hydrochloric acid. The tartrazin can be further isolated from a possible trace of amaranth by adding 10 cc. of alumina cream to each 100 cc. of solution, mixing, warming, and filtering, when the tartrazin will be found in the sodium chloride filtrate. To isolate from the salt, evaporate and redissolve in alcohol.

Dissolve the precipitate containing orange I, ponceau 3R and indigo disulfo acid, together with excess sodium chloride on the filter paper, in water and extract with three successive portions of acetic ether. Orange I is taken up by acetic ether. Combine the acetic ether extracts and wash with saturated sodium chloride solution, until no more color is removed. Extract the acetic ether solution with water to obtain the Orange I in an aqueous solution and free from acetic ether by warming on the steam bath.

Warm the water solution containing ponceau 3R and indigo disulfo acid, from which the greater part of the Orange I has been removed on the steam bath until free from acetic ether, cool, add 10 grams of granulated calcium chloride, allow to stand fifteen minutes, and then add 15 cc. of a freshly prepared stannous chloride solution containing the equivalent of 3% metallic tin and 12% of hydrochloric acid (sp.gr. 1.19). Mix well and allow to stand until the solution shows no blue color. If ponceau 3R is present, it will be precipitated. Filter immediately, wash the precipitate twice with 25% calcium chloride solution to remove all the reduced indigo disulfo acid, dissolve the remaining residue in dilute ammonia solution and test for ponceau 3R.

To the filtrate, which should be practically colorless, add 3% hydrogen peroxide solution. A deep blue coloration indicates the presence of indigo disulfo acid.

**Quantitative Separation of Acid Coal-tar Colors.**—*Mathewson Method.*\*—This process, like the preceding, is for the colors themselves, but may be adapted for the detection of the colors in food products after separation by means of solvents or less satisfactorily by dyeing. Mathewson's table is given on pages 837 and 838.

In applying the data given in the table proceed essentially as follows: Treat the solution containing 0.2 to 0.4 gram of color (depending on the nature of the dyes) with sufficient water and hydrochloric acid to bring

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\* U. S. Dept. of Agric. Bur. of Chem., Circ. 89.



its volume to about 50 cc. and its acid concentration to that point for which the difference in percentage of color extracted for the two dyes is near its maximum. Shake the solution with the immiscible solvent, passing it in succession through three or four separatory funnels each containing 50 cc. of the latter. Wash the portions of the solvent with 50 cc. of hydrochloric acid of the same normality as the solution, passing it

MATHEWSON'S TABLE SHOWING PERCENTAGE OF COLOR IN THE WATER SOLUTION AFTER SHAKING WITH AN EQUAL VOLUME OF IMMISCIBLE SOLVENT.

SOLVENT: AMYL ALCOHOL.

Colors.	Normality of Hydrochloric Acid in Water Layer before Shaking							
	4	2	1	$\frac{1}{2}$	$\frac{1}{4}$	$\frac{1}{8}$	$\frac{1}{16}$	$\frac{1}{32}$
	Percentage of Color in Water Solution after Shaking.							
Naphthol Yellow S No. 4.....			4	7	11	17	27	43
Orange I No. 85.....					0.5	1	2	4
Ponceau 3 R No. 56.....		1	3	7	21	43	64	78
Amaranth No. 107.....	5	15	52	82	93			
Light Green S F No. 435.....	90	95	97	99	99			
Erythrosin No. 517.....								0
Indigo Carmin No. 692.....	34	51	89	96	99			
Fast Yellow No. 8.....	36		61					
Crocein Orange G No. 13.....							1	2
Orange G No. 14.....			18		58			
Ponceau 2 R No. 55.....		2		14				
Crystal Ponceau No. 64.....					8		39	
Fast Red B No. 65.....			2		16		62	
Resorcin Yellow No. 84.....					5		11	17
Orange II No. 86.....							2	3
Brilliant Yellow S No. 89.....			73		90			
Tartrazin No. 94.....			47		88			
Metanil Yellow No. 95.....							1	1
Fast Red A No. 102.....							0.2	1
Fast Red C No. 103.....			1		4			
Fast Red E No. 105.....			1		17		68	
New Coccin No. 106.....		15			75			
Scarlet 6 R No. 108.....	41	80	95					
Resorcin Brown No. 137.....					8		20	
Cotton Scarlet 3 B No. 146.....					2		10	
Congo Red No. 240 *.....								
Azo Blue No. 287 †.....								
Chrysophenin No. 329.....							1	2
Guinea Green B No. 433.....			48		32		25	
Acid Magenta No. 462.....	75		93					

\* Color acid nearly insoluble in both layers.

† Similar to Congo Red but color acid more soluble in alcohol.



MATHEWSON'S TABLE SHOWING PERCENTAGE OF COLOR IN THE WATER SOLUTION AFTER SHAKING WITH AN EQUAL VOLUME OF IMMISCIBLE SOLVENT—(Continued).

SOLVENT: DICHLORHYDRIN.

Colors.	Normality of Hydrochloric Acid in Water Layer before Shaking.				
	4	2	1	$\frac{1}{2}$	$\frac{1}{4}$
	Percentage of Color in Water Solution after Shaking.				
Naphthol Yellow S No. 4.....			15		
Ponceau 3 R No. 56.....			37		
Orange I No. 85.....			4		
Amaranth No. 107.....			95		
Light Green S F No. 435.....			15	.....	17
Indigo Carmin No. 692.....			91		
Acid Magenta No. 462.....			86		

SOLVENT: AMYL ACETATE.

Naphthol Yellow S No. 4.....	22	33	48	
Ponceau 3 R No. 56.....	95	96	97	

SOLVENT: ETHER.

Naphthol Yellow S No. 4.....	.....	94	97	
Orange I No. 86.....	.....	97	97	

successively through the separatory funnels in the same order as was the original solution, and repeat this operation with one or two fresh amounts of the hydrochloric acid. The dye relatively more soluble in water is determined in the combined washings and extracted solution. Remove the second dye from the solvent by shaking with water, very dilute caustic soda, or, more quickly, with dilute caustic soda after the addition of some gasoline, or similar substance in which the color is insoluble.

The table given above shows the percentage of the color in the water solution after shaking with an equal volume of the immiscible solvent.

Assuming the distribution ratios to remain constant, this procedure using four funnels and making three washings gives for a pair of colors whose "distribution numbers" (as the percentage numbers given in the



table may be called) are 80 and 20, respectively, a separation of 98.30 per cent for each color. With distribution numbers 90 and 10 four funnels and three washings give a calculated separation of 99.73%, and the same is obtained with distribution numbers 81.8 and 5.3 if the solvent in which the dyes are relatively more soluble be taken in portions one-half the volume of those of the other liquid. If the second, third, and fourth funnels be given a fifth washing, the third and fourth funnels a sixth, and the last funnel a seventh washing, the calculated loss for the color more soluble in the solvent layer is 0.76%, while the percentage of the other dye removed is relatively much increased (to 99.99%). In most mixtures the progress of the separation is always apparent.

In practice, because of incomplete extraction and separation, and especially on account of uncertainty due to small amounts of subsidiary dyes always present, it is necessary to increase the number of successive extractions. The formation of esters of the color acids is a possible source of difficulty, but is not believed to take place. With amyl alcohol as solvent it is usually desirable to make the original solution more strongly acid than is indicated by the distribution data and use relatively more portions of the washing liquid.

Of the permitted colors, Naphthol Yellow S is best separated from Orange I by washing the amyl alcohol solution of the color acids with strong salt solution, care being taken that not too much color is present. With a solution containing 20 grams of salt and 0.04 gram Naphthol Yellow S per 100 cc. and shaken with an equal volume amyl alcohol, 97% of the color is retained by the water. With a similar solution containing 0.07 gram Orange I, the water layer contains 1.5% of the total color. With higher concentrations some color may be salted out in solid form, but this does not interfere if the amount is small. Erythrosin being quantitatively removed from slightly acid solutions by amyl acetate, ether, or amyl alcohol, its separation from sulphonated colors presents no difficulty.

**Analysis of Food Colors.**—Seeker and his co-workers have devised methods for the analysis of the seven coal-tar colors allowed by federal decision in the United States. The methods are for the determination of the ultimate constituents and for impurities, including arsenic and other heavy metals. The reader is referred to Hesse's report \* for details of these processes.

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\* Loc. cit., pp. 210-226.



Loomis\* has prepared a table giving the solubility of food colors in various solvents, and another table showing the relative amounts extracted from neutral, alkaline, and acid solutions, shaking with amyl alcohol, ethyl acetate and acetone, the aqueous solution in the latter case being saturated with salt.

**Spectroscopic Examination.**—The absorption spectra of dyes, both of coal-tar and vegetable origin, in various solvents, have been described by Vogel† and by Formánek‡ on the Continent, by Sorby§ in England, and by Mulliken|| in the United States. These data, although specially designed for the dye chemist, are none the less valuable for the food analyst. Unfortunately, few chemists are equipped with suitable spectroscopic apparatus or are acquainted with the details of manipulation.

**DETECTION OF COAL-TAR COLORS IN FOODS.**—The examination of foods for foreign colors involves usually at least two distinct steps: First the extraction of the dye from the product, and second the identification of the dye thus removed; where more than one color is present a third step or series of steps is necessary to separate these colors previous to identification.

*Extraction of Colors from Foods.*—There are various methods for the separation of coloring matters from food products, and these may be divided into three general classes: First, dyeing silk or wool with the color by boiling the fiber in a solution of the sample to be examined; second, extracting the color from a solution of the sample by the use of an immiscible solvent; and third, extracting the color from the dried residue of the sample by means of a suitable solvent. Of these the method of dyeing wool lends itself most readily to the analyst's use, by reason of its simplicity, and from the fact that the coal-tar dyes adapted for food colors, with few exceptions (i.e., auramin), being substantive dyes are readily taken up by wool, whereas the natural colors of foods are left in the solution. Some vegetable and animal dyes, such as lichen colors and cochineal, also dye wool, but these are readily distinguished from coal-tar colors by special tests.

*The Separation of Colors* is best carried out by fractioning between

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\* Loc. cit., pp. 8-21.

† Praktische Spektralanalyse iridischer Stoffe, 1889.

‡ Spektralanalytischer Nachweis künstlicher organischen Farbstoffe, 1900; Qualitative Spektralanalyse anorganischer und organischer Körper 1905; (with Grandmougin) Untersuchung und Nachweis organischer Farbstoffe auf spektroskopischem Wege.

§ Proc. Royal Soc., 92, p. 1867.

|| Loc. cit.



aqueous solutions and immiscible solvents according to Mathewson's method, page 859.

*Identification of Colors.*—Three types of methods are mostly used: First, spot tests, that is the application of reagents directly to the dyed fiber or dry color (pages 854–858); second, reactions in the solution of the color (pages 868–875); and third spectroscopic examination. Most analysts are limited to the first two; those equipped with spectroscopic apparatus are referred to the work named on page 840.

**Basic and Acid Dyes.**—The soluble coal-tar dyes are either basic or acid. Basic dyes are precipitated from their aqueous solution by tannin. Acid dyes are not so precipitated. Theoretically, all the basic colors are taken up by wool from a faintly alkaline or neutral bath, while the acid colors are left in solution. Thus if a dilute solution of the color be made faintly alkaline with ammonia and boiled with the wool, only basic colors will be taken up. If both acid and basic dyes are present in the same solution, the basic color should first be exhausted by the use of fresh pieces of wool in the ammoniacal solution, till they no longer take out color, after which the solution should be made slightly acid with hydrochloric acid and again boiled with wool, which under these conditions takes out any acid colors. Comparatively few basic colors are employed in foods. Basic colors can be removed from the fiber by boiling with 5% acetic acid. Acid colors are removed therefrom by boiling with 5% ammonia. Having dissolved the dye from the fiber by the appropriate solvent as above, the decolorized fiber may be removed, and the solution evaporated to dryness on the water-bath. The residue consists chiefly of the dyestuffs, and may be put through various reactions for identification.

**Methods of Dyeing Wool from Food Products.**—The wool employed should be white worsted, or strips of white cloth, such as nun's veiling or albatross cloth. Care should be taken that the color is pure white and not the mere common cream white. The woolen material should be freed from grease by boiling first in 0.1% sodium hydroxide solution and finally in water. Strips of the woolen cloth, or pieces of the worsted thus cleansed, are boiled in diluted unfiltered solutions of jams, jellies, ketchup, and other solid or semi-solid preparations, or in undiluted fruit juices, carbonated beverages, and of wines, the liquid, previous to the boiling being slightly acidified as described below.

*Arata* \* was the first to employ dyeing tests in an acid bath in food

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\* Zeits. anal. Chem., 28, 1889, p. 639.



examination, but limited his observations to wines. Winton \* later found that the method was well adapted to the detection of coal-tar colors in various foods. The method consists in boiling the wool in a dilute solution of the food material to which potassium bisulphate has been added, using 10 cc. of a 10% solution of the bisulphate to 100 cc. of the solution to be tested. If the color solution is neutral, the wool may first be boiled in this before acidifying, to separate out any basic dyes. The dyed wool, after removal from the solution, is boiled first in water, and afterwards preferably in an alkali-free soap solution. It is then washed and dried. The dried fiber may then be subjected to the various reactions given in the table, pages 854-858, for recognition of the dye. This method of identifying colors by means of reactions on the dyed fiber is one of the most convenient—in fact Arata's test, supplemented by reactions on the fiber, suffices in many cases of suspected coloring.

Some of the vegetable dyes (including lichen colors), also cochineal, dye wool directly, and these may be identified by special reactions. Other vegetable colors, and the natural colors of fruits nearly always give a slight dull coloration or stain to the wool, but this is not, as a rule, to be mistaken for the vivid hues of the coal-tar dyes. Moreover, most of the vegetable colors on the fiber turn green when treated with ammonia. Care should be taken to thoroughly wash the wool after the dyeing, so that colored particles simply held thereon mechanically may be removed.

*Sostegni and Carpentieri* † recommend a method of double dyeing, applicable when acid dyes are employed. The wool is boiled in a solution of the food sample acidulated with hydrochloric acid, after which the fiber is removed and boiled, first in very dilute hydrochloric acid solution, and then in water, till free from acid. The color is next dissolved from the fiber by boiling the latter in a weak ammoniacal solution, some of the colors being more readily dissolved than others. The fiber is then removed from the solution, the latter is acidified with hydrochloric acid, and the color fixed on a fresh piece of wool by boiling therein. The second dyeing fixes coal-tar and lichen colors on the fiber, but fruit colors and most others of vegetable origin remain in solution after this treatment. Any color left on the first fiber, after treatment with ammonia, is probably due to the natural vegetable color of the sample, and is usually no more than a dull stain.

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\* Jour. Amer. Chem. Soc., 22, 1900, p. 582.

† Zeits. anal. Chem., 35, 1896, p. 397.



**Extraction of Colors from their Solution by Amyl Alcohol.**—Methods based on this principle have for years been used in examining wines in the municipal laboratory at Paris\* and were found by Winton† to be adapted for various foods. Sanglé-Ferrière uses the following method: 50 cc. of the wine or solution to be tested for color are rendered slightly alkaline by ammonia, and cautiously shaken with about 15 cc. of amyl alcohol. If acid dyes are present, they will be dissolved, and will impart to the amyl alcohol a distinct color.‡ Basic dyes also are dissolved, but when they are present the amyl alcohol solution is colorless. Remove the amyl alcohol by means of a separatory funnel, wash with water, and finally, if the alcohol is colored, dilute with about an equal volume of distilled water and evaporate on the water-bath with a piece of white wool. The wool should be kept in the solution till the odor of the amyl alcohol has disappeared, and, if not then colored, for a short time longer, as with some colors the wool will dye more readily in the aqueous solution than in the amyl alcohol. Remove the wool, and evaporate the solution to dryness. Test for color in the dried residue, and on the fiber also.

*Archil* and other lichen colors, like the acid colors, is extracted by the amyl alcohol under the above conditions, the color being a light violet.

If the amyl alcohol extract after separation, washing, and filtering is colorless, acidify with acetic acid; if a basic color is present, it will be indicated by a coloration at this stage; if there is no coloration on the addition of acetic acid, no basic color is present excepting fuchsin, which is separately tested for. In case a basic dye is indicated, add distilled water and evaporate with wool as before. Test the dried residue with pure concentrated sulphuric acid.

*Fuchsin* is indicated by a yellow-brown color with sulphuric acid, which by dilution with water becomes rose; *safranin*, by a green color becoming first blue, then red, when diluted with water, and *magdala red* by a dark blue color, turning red on the addition of water.

Many coal-tar colors are extracted by amyl alcohol in acid solution, but some fruit colors as well as cochineal are also dissolved under these conditions. The coal-tar dyes thus dissolved will, however, dye wool and the fruit colors will not. The test for cochineal in the amyl alcohol solution

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\* Girard, *Analyse des Matières Alimentaires*, pp. 183, 681.

† Loc. cit.

‡ Acid fuchsin forms an exception to this rule by dissolving colorless like basic dyes. Special tests are given on p. 845.



is described on page 824. Fruit colors are not extracted from acid or alkaline solution by ether, nor from alkaline solution by amyl alcohol.

**Extraction of Colors from their Solution by Acetic Ether.**—Basic colors are extracted readily, according to Robin, by making the solution to be tested alkaline with sodium hydroxide, and shaking with acetic ether. The solvent is removed, washed, and evaporated with wool (on which the tests are to be made), or the evaporation is carried to dryness and the tests made on the residue.

**Separation of Acid and Basic Colors with Ether.\***—*Rota's Method.*—To 100 cc. of the aqueous solution containing the color add 1 cc. of 20% potassium hydroxide and shake in a separatory funnel with several portions of ether. Basic dyes are dissolved by the ether, leaving behind as a rule the acid colors.† Wash the ether extract with faintly alkaline water, and shake out with 5% acetic acid. Some colors remain in the ether, others are dissolved in the acid. Separate the two solvents, and evaporate each to dryness on the water-bath.

The acid colors left in the slightly alkaline, aqueous solution after removal of the basic colors by ether as above, may, if desired, be separated into several groups by successive extraction, as follows: first slightly acidulate with acetic acid and extract with ether, then acidify with hydrochloric acid and again extract, and finally examine the residual solution for colors that are insoluble in ether. Thus erythrosin and eosin are soluble in ether when shaken with their aqueous solution made acid with hydrochloric acid, while acid fuchsin is insoluble.

**Separation of Colors from Dried Food Residues by Solvents.**—This method is rarely employed, excepting in the case of colors insoluble in water, but soluble in ether or alcohol. The dried pulp of canned vegetables, ketchups, etc., may be acidified with hydrochloric acid, and the color extracted therefrom directly with alcohol. In this case, however, there is no obvious advantage over the previous methods of dyeing the fiber directly in the acid solution of the sample.

**Robin's Test for Acid Colors.**—Add to the liquid to be tested an excess of calcined magnesia, and a little 20% mercuric acetate solution, boil, and filter. If the filtrate is colored, or if by the addition of acetic acid to the colorless filtrate a color is developed, a coal-tar dye is indicated.

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\* Analyst, 24, p. 45.

† A few acid dyes are exceptional in being soluble in ether with alkali, as for example, quinolin yellow and the sudans.



**Girard's Test for Acid Fuchsin.\***—Acid 2 cc. of 5% potassium hydroxide to 10 cc. of the wine or other solution to be tested, or enough of the alkali to neutralize the acid. Then add 4 cc. of 10% acetate of mercury and filter. The filtrate should be alkaline and colorless. If the solution remains uncolored after acidifying with dilute sulphuric acid, no acid fuchsin is present. If, however, there is produced a red to violet coloration, and no other coal-tar colors have been found by the amyl alcohol extraction, the presence of acid fuchsin is shown.

**Bellier's Test for Acid Fuchsin.**—Presence of acid fuchsin is indicated by adding to 20 cc. of wine or other solution to be tested about 4 grams of freshly precipitated yellow oxide of mercury, boiling and filtering. The filtrate, if acid fuchsin is present, is colored red, tinged with violet.

According to Blarez, all red coal-tar colors, with the exception of acid fuchsin, and all red vegetable colors are completely decolorized by acidulating their aqueous solution with tartaric acid, and digesting with lead peroxide.†

**Loomis' Scheme for Preliminary Identification of Colors in Foods.‡**—This scheme covers certain coal-tar, animal, and vegetable dyes commonly used in foods. The strength of the aqueous solution should be approximately 0.01% for coal-tar colors and 0.1% for animal and vegetable ("natural") colors.

The following reagents are required:

**Weingartner's Tannin Reagent.**—A solution of 10 grams each of tannic acid and sodium acetate in 100 cc. of water.

**Hydrochloric Acid.**—Equal volumes of concentrated acid and water.

**Sodium Hydroxide Solution.**—Ten grams in 100 cc. of water.

**Ammonia Water.**—Approximately 10%  $\text{NH}_3$ .

**Lead Subacetate Solution.**—See p. 610.

**Normal Lead Acetate Solution.**—Ten grams in 100 cc. of water.

Reactions in aqueous or alcoholic solution are carried out with 10 cc. of color solution and 5 to 10 drops of reagent; unless otherwise noted, each test is made on a part of the original solution. To determine whether a large or small amount is extracted by an immiscible solvent from an aqueous solution, separate the former, filter, evaporate (with addition of water if necessary), take up in water, add a little acid or alkali to correspond

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\* Analyse des Substances Alimentaires, p. 185.

† Allen, Commercial Org. Analysis, 4 Ed., Vol. V, p. 250.

‡ Loc. cit., pp. 62-69.



## ANALYTICAL SCHEME FOR THE PRELIMINARY IDENTIFICATION OF COLORS IN FOODS (LOOMIS).

(Confirm by tests given in Tables on pages 819, 854, and 868. Numbers are those in Green's table.)

## I. GREEN COAL-TAR COLORS.

[Mixed blue and yellow generally detected by spot test on filter paper or by fractional dyeing.]

## I. To aqueous 0.01 per cent solution of color add tannin reagent.

A. No PRECIPITATE. Add 10 per cent NaOH solution to aqueous color solution.

a. Gradually decolorized.....*Acid green* (435)b. Dark blue.....*Cyanole green 6 G*c. No change.....*Naphthol green B* (398)

B. PRECIPITATE. Acidify aqueous solution with HCl and extract with equal volume of ethyl acetate.

a. Color all extracted.....*Ethyl green*b. About one-half color extracted.....*Malachite green* (427)

## II. BLUE OR VIOLET COLORS.

## I. Insoluble in cold water. Treat with 50 per cent alcohol.

A. INSOLUBLE.....(*Indigo* 689)B. SOLUBLE.....*Litmus*

## II. Soluble in cold water. Add tannin reagent to 0.01 per cent aqueous solution.

A. No PRECIPITATE. Treat with zinc dust + HCl till decolorized, then filter.

a. Color of filtrate quickly restored.....*Indigo disulpho-acid* (692)

b. Color not soon restored in filtrate. To aqueous solution add NaOH solution.

1. Solution blue green by transmitted light, pink by reflected light.....*Cyanole F F* (439)2. No change.....*Tetracyanole S F* (440)3. Pale magenta.....*Azo blue* (287)

B. PRECIPITATE. Aqueous solution + 10 per cent NaOH.

a. Solution magenta.

1. Aqueous solution + HCl = yellow.....*Methyl violet* (451)2. Aqueous solution + HCl = green.....*Methylene violet* (585)b. Solution; no change.....*Methylene blue* (650)







## III. ORANGE AND YELLOW COLORS—Continued.

## I. Soluble in cold water—Continued.

## A. NO PRECIPITATE—Continued.

6. Crimson or red. To aqueous solution add NaOH solution.
- α. No change in color; pale glistening precipitate. Dry color + concentrated  $H_2SO_4$ .  
 Purple or violet solution.....Orange IV (88)  
 Brown solution.....Orange III (87)  
 Orange I (85)
- β. Much redder.....
- γ. Little or no change and no precipitate. Dry color + concentrated  $H_2SO_4$ .  
 1. Brown solution.....Orange III (87)  
 2. Crimson or magenta solution.....Brilliant yellow S (89)  
 3. Purple or violet solution. To 10 cc. aqueous solution add about 20 drops NaOH solution.  
 α. Colorless glistening precipitate at once or on standing a few minutes.....Orange IV (88)  
 β. No change.....Metanil yellow (95)
- b. Wool not dyed. To aqueous solution add dilute  $NH_4OH$ .  
 1. No change. Dry color + concentrated  $H_2SO_4$  = blue or greenish blue.....Safron  
 2. Darker. Shake aqueous solution with equal volume of acetic ether.  
 α. No color extracted. Aqueous solution + 10 per cent  $FeCl_3$  solution, yellow brown solution, no precipitate.....Weld (696)  
 β. Considerable color extracted by acetic ether. To aqueous solution add 10 per cent solution normal lead acetate.  
 1. Solution turns bright yellow, no precipitate.....Buckthorn (700)  
 2. Brownish precipitate. Add to aqueous solution 10 per cent alum solution.  
 α. Solution yellow; no precipitate.....Quercitron (699)  
 β. Turbidity which is cleared up by a few drops of acetic acid and solution then has decided green fluorescence.....Fustic (698).
- B. COLORED PRECIPITATE. To color solution add NaOH solution.
- a. Solution decolorized and white precipitate of base. On shaking alkaline solution with ether, solution becomes clear and ether layer turns yellow on adding drop of acetic acid.....Auramine (425 or 426)
- b. Yellow or orange precipitate. On shaking alkaline solution with ether, solution becomes clear and color passes mostly into ether layer, which is orange yellow.....Chrysoidine (18?)
- c. No change.....Thioflavin



**II. Insoluble in cold water.** Treat with 95 per cent alcohol.**A. SOLUBLE.** To alcoholic solution add HCl.

1. Crimson or pink.....*Butter yellow* (16)
2. No change or paler. Treat alcoholic solution with zinc dust and a few drops of acetic acid and shake for about half a minute and filter.

**α.** Filtrate colorless. Dry color and concentrated  $H_2SO_4$ .

Reddish brown.....*Sudan G* (10)  
 Bright red.....*Sudan I* (11)

**β.** Not decolorized or only partially so. (Distinguish and identify by usual tests).....*Turneric or Annatto***B. INSOLUBLE.** Treat with boiling water.

Soluble.....*Catechu* (703)

Insoluble.....*Color lakes*  
 (Identify metallic base by qualitative analysis of ash. Identify coloring matter by dissolving lake in acid and extracting with immiscible solvent or by fixing on fiber.)

**IV. RED COLORS.****I. Soluble in cold water.****A. DILUTE AQUEOUS SOLUTION FLUORESCENT.** Apply double dyeing test of Sostegni and Carpentieri (p. 842).

*a.* Fiber not dyed.....*Brazil wood* (701)

*b.* Fiber dyed. Add to aqueous solution a few drops tannin reagent.

1. Colored precipitate.....*Rhodamine* (504)

2. No precipitate. Prepare a dilute solution of color in 95 per cent alcohol.

*α.* Solution has green fluorescence.....*Eosin A* (512)

*β.* Solution has yellow or orange fluorescence.....*Phloxine* (518 or 521)

**B. DILUTE AQUEOUS SOLUTION NOT FLUORESCENT.** Add to solution of color a few drops of tannin reagent.

*a.* No precipitate of color. Apply double dyeing test of Sostegni and Carpentieri. [If, in course of test, fiber or solution becomes purple on addition of  $NH_4OH$ , test directly for *cochineal* by Robin's tests (p. 824) or *lichen colors* by Tolman's method (p. 821).]

1. Fiber dyed. Make 0.01 per cent aqueous solution alkaline with  $NH_4OH$  and extract with amyl alcohol.

*α.* Considerable color extracted by amyl alcohol, which is colored red. Shake color solution, acidified with HCl, with ether.







- b.* Almost no color extracted. Treat dyed fiber with concentrated HCl.
- 1a.* Crimson or scarlet. To dry color add concentrated  $H_2SO_4$ .
- 1b.* Purple or violet. Acidify aqueous color solution with HCl and shake with amyl alcohol.
- 1c.* No color extracted. To aqueous solution add NaOH solution.
- 2c.* Dirty yellow solution—Dry color is yellow-brown. *Ponceau 6R* (108)
- 2d.* Darker red solution—Dry color is brownish-red. *Amaranth* (107)
- 2c.* Much color extracted—Amyl alcohol layer scarlet. *Fast red C* (103)
- 2b.* Crimson or scarlet. Note color of neutral aqueous solution (0.01 per cent).
- 1c.* Orange red. To 0.01 per cent aqueous solution add 10 per cent  $BaCl_2$  solution.
- 1d.* Crimson precipitate. To aqueous solution add 10 per cent  $FeCl_3$  solution.
- Orange precipitate. *Ponceau 3R* (56)
- No precipitate. *Ponceau 2R* (55)
- 2d.* No precipitate. Saturate aqueous solution with salt and extract with equal volume of neutral acetone.
- Acetone extracts almost no color. *Cochineal red A* (106)
- Acetone layer extracts considerable color and is orange. *Brilliant cochineal 2R* (53)
- 2c.* Crimson or magenta. *Lanafuchsine*
- 2a.* Nearly or quite decolorized. *Acid magenta* (462)
- (See also p. 845.)
- 3a.* Purple. *Fast red B* (65)
- 4a.* Blue. To dry color add concentrated  $H_2SO_4$ .
- 1b.* Magenta solution. *Biebrich brilliant crocein scarlet* (146)
- 2b.* Blue solution. To dyed fiber add NaOH solution.
- 1c.* Fiber purple. *Biebrich fast scarlet* (150)
- 2c.* Fiber brown. To aqueous solution add lead subacetate solution.
- 1d.* Yellow solution; orange precipitate. *Crocein scarlet 3B* (166)
- 2d.* Pink solution; red precipitate. *Crocein scarlet 7B* (169)
- 2.* Fiber not dyed. Dye wool, mordanted with alum, in aqueous color solution.



## IV. RED COLORS—Continued.

## I. Soluble in cold water—Continued.

## B. DILUTE AQUEOUS SOLUTION NOT FLUORESCENT—Continued.

## b. Almost no color extracted—Continued.

## 2. Fiber not dyed—Continued.

α. Wool colored bluish or grayish violet.....Logwood (702)

β. Wool colored bright red.....Brazil wood (701)

## c. Colored precipitate. Dissolve color in ethyl or amyl alcohol and also in acetone.

1. Solutions fluorescent. To dyed fiber add 10 per cent NaOH solution = crimson.....Safranine (584)

2. Solutions not fluorescent. To dyed fiber add 10 per cent NaOH solution = decolorized.....Magenta (448)

## II. Insoluble in cold water.

A. To alcoholic solution add  $\text{NH}_4\text{OH}$  = blue.....Alcannin

B. To dry color add concentrated  $\text{H}_2\text{SO}_4$ .

1. Greenish-blue solution.....Sudan III (143)

2. Crimson solution.....Sudan II (49)

3. Fluorescent brown-yellow solution.....Brazil wood (701)

C. Color dissolves in alkalis, forming orange solution (cotton immersed in alkaline solution is dyed red on acidifying).....Carthamine

D. Nearly insoluble even in hot water, soluble in dilute alkali, with brown-red color; completely precipitated, on acidifying, in red-brown flakes.....Barwood or sandalwood (705)



with the aqueous layer, and compare with the latter. Dyeing tests may be made on the solution thus obtained. When the solution is decolorized by acid or alkali during shaking with an immiscible solvent, separate both layers and neutralize both in order to find the relative portions of color in the two.

**Direct Identification of Colors.**—In identifying the colors commonly used in food, it is frequently possible to ascertain the color or group of colors present by making direct tests with various reagents, either on the dyed fiber or on the dry coloring matter, or in a solution containing it.

Many tables for this purpose are prepared, but they are never complete by reason of the many new dyestuffs constantly introduced. Such tables are to be found in the works of Allen, Schultz and Julius, and Mulliken. While it is true that the limitation of the dyes suitable for purposes of food coloring imposes a somewhat lighter task on the food analyst than that on the chemist who has to deal with all varieties of commercial colors, yet it is obviously impossible to make a complete list covering even the restricted field of food colors. Doubtless there are colors long well known that would serve admirably for this purpose, but have never yet been tried.

**Reactions of Dry Colors or Dyed Fibers.**—For the purposes of the food analyst the table of Mathewson\* (pages 854 to 858) is well adapted as it includes colors which actually have been found in foods, being the same colors as are also included in the table on pages 868 to 875.

The analyst should be provided, for standards, with as complete a collection of known purity dyestuffs as possible covering the colors he is likely to meet with in foods, and should make comparative tests, if the slightest doubt exists. If the unknown color is apparently not found in the table, and the more exhaustive tables are unavailable, it is still possible to locate the dye, by making similar tests on other standard colors suggested by the behavior of the unknown color, and carefully comparing them.

Most difficulty is encountered when the coloring matters are mixtures instead of simple dyes. In this case it is recommended to resort to systematic separation by immiscible solvents as elaborated by Mathewson (page 859).

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\* Separation and Identification of Food-coloring Substances, U. S. Dept. of Agric., Bul. 148, pp. 37-45.



## BEHAVIOR OF DRY COLORS OR OF DYED FIBERS WITH REAGENTS (MATHEWSON).

Numbers of dyes permitted by federal rulings are in boldfaced type; numbers of natural colors are in italics. Dyes most common in foods are designated by \*\*\*, others found less often by \*\* and \*. Names of colors are given on pages 868-875.

No. (Green).	Color of Sulphuric Acid Solution.	Color of Dyed Wool.	Reactions of Dyed Wool with Reagents.			
			Concentrated Hydrochloric Acid.	Concentrated Sulphuric Acid.	10% Sodium Hydroxide Solution.	Ammonia Water, Sp.gr., 0.95.
***462	Yellow	Violet-red	Nearly decolorized	Yellow	Decolorized	Decolorized
434	Yellow	Green	Yellow	Orange or pale brown	Decolorized	Decolorized
<b>435</b>	Yellow	Green	Yellow	Orange or pale brown	Decolorized	Decolorized
*436	Yellow	Green-blue	Yellow	Orange or pale brown	Slightly darker	Slightly darker
439	Yellow	Green-blue	Yellow	Orange or pale brown	Yellow-olive	Redder
491	Yellow	Green	Yellow	Orange or pale brown	Little change	Little change
440	Yellow	Green-blue	Yellow	Orange or pale brown	Little change	Little change
**602	Deep blue	Blue-gray to black	Dull bluish	Dull greenish	Pale brown-red	Pale reddish
**108	Violet	Red	Blue-red	Violet	Brown	Orange-red
***8	Brown-yellow	Yellow	Red	Orange	Little change	No change
9	Brown-yellow	Yellow	Red	Orange	Little change	No change
**89	Violet-red	Yellow	Blue-red	Blue-red	Little change	Little change
<b>692</b>	Violet-blue	Blue	Slightly darker	Slightly darker	Green-yellow	Green-blue
399	Red-brown	Orange-yellow	Yellowish, dull	Red-brown	Orange	Orange
***106	Red-violet	Scarlet	Red	Violet-red	Yellow-brown	Orange-red
<b>107</b>	Violet	Red	Slightly darker	Violet to brownish	Dull brownish	Little change
<b>94</b>	Yellow	Yellow	Slightly darker	Slightly darker	Little change	Little change
*398	Yellow-brown	Yellow-green	Yellowish	Brown-yellow	No change	No change
605	Green	Red	Little change	Dark green	Little change	Little change
604	Green	Red	Dull brown	Dark green	Little change	Little change
188	Green-black	Blue-black	Green-blue	Olive green	Black	No change
***14	Orange-yellow	Orange-yellow	Little change	Orange	Dull brown-red	No change
21	Red	Violet-red	Orange-red	Rrown-red	Brown-red	Little change
318	Blue-green	Blue	Little change	Green-blue	Red	Redder
20	Réd	Red	Little change	Darker	Red-brown	Brown
93	Violet	Violet red	Little change	Violet	Slightly bluer	Orange-red
***480	Brown	Blue	Paler	Brown	Pale reddish	Almost decolorized
*53	Violet red	Scarlet	Darker	Violet-red	Brown-yellow	No change



## BEHAVIOR OF DRY COLORS OR OF DYED FIBERS WITH REAGENTS—Continued.

No. (Green).	Color of Sulphuric Acid Solution.	Color of Dyed Wool.	Reactions of Dyed Wool with Reagents.			
			Concentrated Hydrochloric Acid.	Concentrated Sulphuric Acid.	10% Sodium Hydroxide Solution.	Ammonia Water, Sp.gr., 0.95.
*55	Red	Scarlet	Little change	Little change	Brown-yellow	No change
105	Violet	Red	Slightly bluer	Red-violet	Dull brown-red	Almost unchanged
***706	Pale yellow	Yellow	Nearly decolorized	Pale dull brown	No change	No change
56	Red-orange	Dull orange-red	Little change	Little change	Violet-red	Violet-red
*62	Red	Red	Little change	Little change	Dull orange	Little change
**64	Blue	Violet-red	Darker	Violet	Dull brown	Little change
*65	Violet	Scarlet	Violet-red	Violet	Dull brown	Little change
**103	Blue	Violet-red	Violet	Blue	Brown	Little change
139	Violet	Violet-red	Little change	Violet	Scarlet	Scarlet
164	Violet	Brown	.....	Dark violet	Darker	Little change
*667	Blue	Scarlet	.....	.....	.....	.....
*169	Orange-red	Yellow	Slightly darker	Brown-yellow	Slightly paler	Little change
163	Blue	Red	Blue	Blue	Dull violet red	Little change
170	Green	Red	Violet-blue	Green	Dull violet	Little change
84	Blue	Red	.....	.....	.....	.....
146	Yellow	Orange-yellow	Orange	Orange	Dull orange-red	No change
287	Red-violet	Red	Blue	Violet	Brown	No change
78	Deep blue	Red-blue	Darker	Green-blue	Red	Violet
***710	Red	Red	Brown	Violet-red	Little change	Little change
*546	Violet-red	Red	Red	Brown, dull	Violet	Violet
1	Violet	Red	Yellow	Orange	Violet	Violet
507	Orange	Orange-yellow	Decolorized	Pale brownish	Slightly darker	No change
328	Almost colorless	Yellow	Slightly yellower	Orange	Red	Red
606	Red-violet	Red-violet	Dull violet	Brown-violet	Orange-red	Orange-red
154	Dull green	Orange	Yellow-brown	Dark green	Little change	Little change
85	Violet blue	Red	Blue	Blue	Brown-violet	Little change
**13	Violet	Orange	Violet	Violet	Red, dark	Red, dark
***86	Orange-yellow	Orange	Orange red	Orange	Slightly darker	No change
97	Red	Orange	Red	Red	Dull orange-red	No change
54	Red	Orange	Red	Red	Browner	Browner
	Orange-red	Orange	Scarlet	Scarlet	Little change	No change



BEHAVIOR OF DRY COLORS OR OF DYED FIBERS WITH REAGENTS—Continued.

No. (Green).	Color of Sulphuric Acid Solution.	Color of Dyed Wool.	Reactions of Dyed Wool with Reagents.			
			Concentrated Hydrochloric Acid.	Concentrated Sulphuric Acid.	10% Sodium Hydroxide Solution.	Ammonia Water, Sp.gr., 0.95.
329	Red-violet	Yellow	Blue	Red-violet	Orange-yellow	No change
137	Brown	Brown	.....	Darker	Darker	.....
157	Blue-green	Red	Blue-violet	Green	Brown-violet	Little change
*95	Violet	Orange-yellow	Violet-red	Violet	No change	No change
88	Violet	Orange-yellow	Violet-red	Violet	No change	No change
*92	Violet-red	Yellow	Violet-red	Violet-red	Dull brown	Little change
101	Blue	Brown	Red-blue	Blue	Violet-red	Violet-red
102	Violet	Red	Red	Violet	Dull orange-red	No change
483	Brown-yellow	Orange red	Orange-yellow	Dull orange-yellow	Little change	Little change
**510	Yellow	Yellow	Little change	Little change	Slightly redder, green fluorescence	Little change, green fluorescence
26	Orange-yellow	Orange-yellow	Orange	Orange	Orange-red	Orange
220	Red-violet	Yellow	Brown	Violet-red	Red-orange	Orange-yellow
269	Red-violet	Yellow	Brown	Violet-red	Red-orange	Orange-yellow
512	Yellow	Red	Yellow-orange	Yellow-orange	No change	No change
515	Brown-yellow	Violet-red	Yellow	Brownish	Scarlet	Little change
516	Brown-yellow	Orange-red	Orange-yellow	Orange-yellow	No change	No change
517	Brown-yellow	Violet red	Orange-yellow	Orange-yellow	No change	No change
518	Brown-yellow	Violet-red	Yellow	Orange	No change	No change
520	Orange	Violet-red	Yellowish	Orange	No change	No change
521	Orange	Violet-red	Yellowish	Orange	No change	No change
*523	Orange	Red violet	Almost decolorized	Orange	No change	No change
2	Pale yellow	Orange-yellow	Almost decolorized	Pale brown-yellow	No change	No change
*3	Pale yellow	Yellow	Almost decolorized	Pale brown-yellow	No change	No change
6	Very pale yellow	Orange	Almost decolorized	Pale brown-yellow	Little change	Little change
534	Orange-red	Orange	Yellower	Redder	Dull orange-red	Slightly redder
**707	Orange	Yellow	Red	Red-brown	Violet	Violet
*10	Yellow Orange	(Silk.) Orange yellow	(Silk.) Orange-yellow	(Silk.) Brown-yellow	Orange	Orange
*468	Orange-yellow	Violet	Pale orange-yellow	Pale dull orange	(Silk.) Orange-yellow	(Silk.) No change
464	Yellow	Blue-violet	Pale orange-yellow	Pale dull orange	Decolorized	Decolorized
438	Yellow-brown	Green	Orange-yellow	Brown-yellow	Decolorized	Paler



## BEHAVIOR OF DRY COLORS OR OF DYED FIBERS WITH REAGENTS—Continued.

No. (Green).	Color of Sulphuric Acid Solution.	Color of Dyed Wool.	Reactions of Dyed Wool with Reagents.			
			Concentrated Hydrochloric Acid.	Concentrated Sulphuric Acid.	10% Sodium Hydroxide Solution.	Ammonia Water, Sp.gr., 0.95.
**433	Yellow	Green	Pale orange-yellow	Pale dull yellow	Decolorized	Decolorized
442	Yellow	Green-blue	Pale orange-yellow	Pale dull yellow	Little change	Little change
476	Red-brown	Blue	Little change	Red-brown	Dull brown	Little change
240	Blue	Red-brown	Blue	Blue	No change	No change
277	Blue	Brown-red	Blue	Green-blue	Orange-red	Orange-red
562	Brown-red	Violet-blue	Orange-red	Brown-red	Green	Blue
658	Colorless	Yellow	Decolorized	Almost decolorized	Decolorized	Paler
496	Orange	Red	Orange-yellow	Yellow	Paler	No change
650	Yellow-green	Blue	Almost decolorized	Greenish, paler	Pale violet	No change
639	Dark green	Blue	Violet-blue	Dull greenish	Pale dull violet-red	Pale dull violet-red
*584	Green	Red	Green-blue	Green	Red	Red
**448	Yellow-brown	Violet-red	Yellow-brown	Dull brown	Decolorized	Paler
***425	Colorless	Yellow	Decolorized	Nearly decolorized	Decolorized	Paler
426	Colorless	Yellow	Decolorized	Nearly decolorized	Decolorized	Paler
***451	Yellow	Violet	Yellowish	Yellowish	Decolorized	Almost decolorized
452	Yellow	Violet	Yellowish	Yellowish	Decolorized	Almost decolorized
**427	Yellow	Blue-green	Almost decolorized	Almost decolorized	Decolorized	Decolorized
428	Yellow	Green	Almost decolorized	Almost decolorized	Decolorized	Decolorized
*107	Brown	Red-brown	Redder, darker	Browner	Yellow	Yellow
*201	Brown	Red-brown	Redder, darker	Browner	Yellow	Yellow
17	Orange-yellow	Orange-yellow	Orange-red	Brown-red	Slightly duller	Yellow
18	Orange-yellow	Orange-yellow	Red	Brown	Slightly duller	Yellow
505	Yellow	Violet-red	Orange	Yellow	Slightly bluer	Slightly bluer
*499	Yellow	Blue-red	Orange	Yellow, pale	Slightly bluer	Slightly bluer
***504	Yellow-brown	Blue-red	Orange	Yellow	Bluer	Bluer
502	Yellow-brown	Blue-red	Orange	Yellow	Slightly bluer	Slightly bluer



## BEHAVIOR OF DRY COLORS OR OF DYED FIBERS WITH REAGENTS—Continued.

Coloring Matter.	Color of Sulphuric Acid Solution.	Color of Dyed Silk.	Reactions of Dyed Silk with Reagents.			
			Concentrated Hydrochloric Acid.	Concentrated Sulphuric Acid.	10% Sodium Hydroxide Solution.	Ammonia Water. Sp. Gr., 0.95.
**16. Dimethylaminoazobenzene.	Yellow.	Orange-yellow.	Red, bluish.	Orange-yellow.	No change.	No change.
7. Aminoazobenzene.	Yellow.	Orange-yellow.	Brown-red.	Orange-yellow.	Little change.	No change.
*. Ortho-aminoazotoluene.	Orange-yellow.	Orange-yellow.	Dull orange.	Orange-yellow.	Little change.	No change.
Benzeneazo- $\beta$ -naphthylamin.	Red-violet.	Yellow-brown.	Red.	Violet.	Little change.	No change.
Ortho-tolueneazo- $\beta$ -naphthylamin.	Violet.	Yellow-brown.	Red.	Violet.	Little change.	No change.
666. Quinophthalon.	Red-brown.	Yellow.	Orange-yellow.	Brown-yellow.	Orange-yellow.	Little change.
Benzeneazo- $\alpha$ -naphthol.	Violet.	Orange.	Violet-red.	Violet.	Deep red.	Deep red.
59. Naphthaleneazo- $\alpha$ -naphthol.	Green-blue.	Brown.	Blue-violet.	Green-blue.	Blue-red.	Blue-red.
Ortho-tolueneazo-o-tolueneazo- $\alpha$ -naphthol.	Green.	Red.	Blue.	Green.	Red-blue.	Red-blue.
*11. Benzeneazo- $\beta$ -naphthol.	Red.	Orange.	Orange-red.	Red.	Redder.	Little change.
**49. Meta-xylenazo- $\beta$ -naphthol.	Violet-red.	Orange-red.	Red.	Violet-red.	Little change.	No change.
60. $\alpha$ Naphthaleneazo- $\beta$ -naphthol.	Blue-violet.	Blue-red.	Slightly bluer.	Dull blue.	Brown and paler.	Slightly paler.
*143. Benzeneazobenzeneazo- $\beta$ -naphthol.	Green.	Red.	Violet, then brown.	Green.	Violet-red.	Little change.
*. Ortho-tolueneazo-o-tolueneazo- $\beta$ -naphthol.	Green.	Violet-red.	Violet-blue.	Blue-green.	Violet-red.	Little change.
31. Para-nitrobenzeneazo- $\beta$ -naphthol.	Violet-red.	Brown-red.	Orange-red.	Violet-red.	Violet-blue.	No change.

With concentrated solution of stannous chloride in hydrochloric acid the fiber is decolorized in all cases except that of No. 666.



**Separation of Colors by Immiscible Solvents and their Identification.**—*Mathewson's Method.*\*—(a) *Preparation of the Solution.*—Make a 0.05 to 0.01% solution of commercial colors. Take up candies, syrups and other saccharine products in hot water. Evaporate or dilute wines and liquors to less than 10% alcohol content. Extract fruit products, flesh foods, and similar products with 80% alcohol containing a very little acetic acid to remove basic colors, cochineal, etc., and the residue with 65 to 80% alcohol containing 3 to 5% of ammonia. Boil both extracts until the alcohol is reduced to less than 10% and ammonia is nearly removed from the second, then combine. It is often preferable to add strong HCl directly to strongly colored jams, sausage, etc., extract with amyl alcohol, and shake with salt solution and other aqueous solvents. For procedure with cereal pastes see pages 366 to 369 and with butter and other fats, pages 557 to 560.

Whatever the method of solution treat the aqueous liquid with sodium carbonate if acid, or with acetic acid if alkaline, until neutral or slightly acid. Suspended matter, other than precipitated dye, should not be present in considerable amount. Too great color concentration (over 0.1%) of food extracts is rarely encountered. Large amounts of sugar, glycerol, etc., affect somewhat the solubilities.

(b) *Separation.*—Since most coal-tar dyes are accompanied by subsidiary dyes those present in largest amount should be first carried through until identified. Colors of fruits and flowers being usually unstable, especially in alkaline solution, are best tested for in a separate portion. The table on pages 868 to 875, based on 0.01% concentration, is more useful than an analytical key. The following outline is for complicated mixtures. Ordinarily the analyst will vary the procedure according to probabilities. A preliminary dyeing test is advisable.

1. Add to the solution NaCl equivalent to 5 to 6% and shake with 20 cc. or more of amyl alcohol, repeating the treatment once or twice if considerable color is extracted. Wash the combined extracts, if colored, once or twice with small portions of 5% NaCl and add the washings, if also colored, together with any suspended matter, to the extracted solution.

2. Add to the extracted NaCl solution half its volume of concentrated HCl and shake with amyl alcohol as before. If the extract is colored wash once with 4N HCl (1 : 2) and proceed according to sec. 6; if colorless, even after adding  $\text{NH}_4\text{OH}$ , reject as most strongly sulphonated azo colors are absent. For procedure when naphthol green is present see sec. 18.

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\* Loc. cit., pp. 8-53.



3. Make the extracted acid NaCl solution, which may be nearly colorless, slightly alkaline with  $\text{NH}_4\text{OH}$ , then slightly acid with acetic acid. If then colorless reject as strongly sulphonated triphenylmethane green and blue dyes are absent. If green or blue shake with enough dichlorhydrin to give a lower layer after separation of 20 cc. or less. If a blue color is extracted repeat once or twice, wash the combined extracts with a little NaCl solution and examine according to sec. 5.

4. The solution after the preceding extraction may still contain natural colors (fruits, etc.), acid magenta (No. 462), and large fractions of acid yellows (Nos. 8 and 9) although these latter are chiefly extracted from the acid solution (sec. 2). To detect No. 462 apply the nitrous acid test, dye test, etc.; to separate it add HCl until over  $\text{N}/4$ , allowing for ammonium acetate, shake with anilin, wash the extract with 5 to 6% NaCl in  $\text{N}/4$  HCl, and remove the dye with water. Before testing make alkaline and remove the dissolved anilin with several portions of carbon tetrachloride or benzene. Commercial acid magenta contains various sulphonates and yields derivatives of greater solubility in organic solvents. If the color of the extracted acid salt solution is entirely due to Nos. 8 or 9 it will be orange red, becoming yellow on neutralization. Nitrous acid, etc., give characteristic reactions.

5. Dilute the dichlorhydrin extract (sec. 3) with 3 to 4 volumes of carbon tetrachloride and remove the color with a few small portions of water. Shake the combined washings once with carbon tetrachloride to remove dissolved dichlorhydrin. The washings may contain higher sulphonated triphenylmethane dyes or sulphonated indulin, with large amounts of subsidiary products. Their solubilities cannot be definitely established. Compare their properties as given in the tables.

6. Wash the amyl alcohol extract of the acid salt solution (sec. 2), if colored, 4 to 5 times with  $\text{N}/4$  HCl, keeping the washings separately and reserving the washed solvent for treatment according to sec. 7 or 8. Nos. 108 and 692 predominate in the first washings; which owing to HCl dissolved in the amyl alcohol, is high in acidity; Nos. 106, 107, and 94 come out chiefly in the third. Vary the procedure according to probabilities. The color of the washings will usually show if more than one dye is present in considerable amount. Separate Nos. 106, 107, and 94 from No. 108 by 2N HCl and amyl alcohol and from Nos. 692 and 8 by 8N  $\text{H}_2\text{SO}_4$  and amyl alcohol-gasoline (1 : 1). Wash Nos. 106, 107, and 94 out of the latter solvent with a little water, add at least half the volume of concentrated HCl, re-extract with amyl alcohol, remove  $\text{H}_2\text{SO}_4$  with a few portions of 4 to 6



N HCl, and finally wash out the dye with water and evaporate to dryness on a water-bath. Test with cyanide, etc. Separate the dyes in the  $\text{H}_2\text{SO}_4$  solution with anilin (page 868). Use anilin and 5 to 6% NaCl in N/4 HCl to separate No. 94 from Nos. 106 and 107, removing the anilin from the solutions, after making faintly alkaline, with carbon tetrachloride. No. 692, which like No. 8 is of indefinite composition, may be separated from azo dyes by cautiously adding powdered sodium hydrosulphite ( $\text{Na}_2\text{S}_2\text{O}_4$ ) to the acid solution, then shaking with air to restore the blue color. Reduction by this reagent in an ammoniacal solution, avoiding an excess, destroys Nos. 106 and 107 while No. 8 is merely converted into the hydrazo compound and may be restored by shaking with air. No. 692 is destroyed by warming in an acid solution containing a little urea and a drop of 7% sodium nitrite solution, while Nos. 106, 107, and 108 are scarcely attacked. Dyes of this group are much used in foods.

7. Wash the amyl alcohol extract with N/16 HCl same as previously with N/4 HCl. Omit if Nos. 14 and 188 appear to be absent.

8. Dilute the amyl alcohol with an equal volume of gasoline (sp.gr. 0.65) then wash successively 2 or 3 times with N/4 HCl, N/16 HCl, N/64 HCl, N/64  $\text{C}_2\text{H}_4\text{O}_2$  and N/64 NaOH. Wash with the alkaline solution even if the preceding appear to remove all the color as some weakly acid dyes (mostly of other groups) are nearly colorless in neutral or acid solvents. Study the solubilities as given on pages 869-872 inclusive. When the fractions appear to contain more than one dye, refraction until pure. Test for No. 4 (nearly colorless in acid solution) by adding HCl to the first strongly acid washings until 2N, shaking with washed ethyl acetate, and treating the latter with alkali. If a yellow color is obtained treat in like manner the remainder of the fractions containing it. Reserve the N/64  $\text{C}_2\text{H}_4\text{O}_2$  and NaOH washings until the neutral NaCl amyl alcohol extract has been tested, as this will contain the bulk of the dyes, or mix these washings with the corresponding ones of secs. 11 and 12, or omit these washings entirely and mix the amyl alcohol-gasoline extract after washing with N/64 HCl with the similar mixture of sec. 10.

9. Chemical methods of separating closely related dyes: (1) cyanide test (page 866) for separating R-salt derivatives (Nos. 55, 56, 65, 15) from mixtures with isomers; (2) reduction and subsequent oxidation methods for destruction of azo and nitro colors in presence of most other colors (see pages 866 and 867); (3) cautious reduction in  $\text{Na}_2\text{CO}_3$  or  $\text{NH}_4\text{OH}$  solution whereby oxyazo dyes tend to be attacked more rapidly than aminoazo dyes, allowing for new dyes formed by partial reduction of polyazo or



nitroazo derivatives; (4) bromine oxidation (page 864), halogenated fluorescin derivatives (fully substituted) being more resistant than most other colors in acid solution and No. 4 than most azo dyes in alkaline solution, but the test is seldom so satisfactory as extraction with ethyl or amyl acetate and fails when Nos. 62, 64, 65, and 188 are present owing to blue substances formed.

10. Dilute the amyl alcohol extract of the NaCl solution (sec. 1), which contains practically all the basic dyes and most acid dyes of low sulphur content, with an equal volume of gasoline, wash a few times with N/64 HCl, and treat the washings, if colored, according to sec. 11. Wash the extract with N/64  $C_2H_4O_2$  and treat the washings according to sec. 12; then, to remove eosins and (in general) unsulphonated, water soluble, acid (phenolic) dyes, wash with a few portions of N/64 NaOH and treat the washings according to sec. 13. Finally wash once with very dilute  $C_2H_4O_2$  and, if still appreciably colored, evaporate to dryness on the water-bath, and examine the residue according to sec. 14.

11. Make a small portion of the N/64 HCl extract (sec. 10) alkaline with NaOH, shake with ether, and treat the usually colorless ether solution with dilute  $C_2H_4O_2$ .\* If a color appears, indicating basic dyes, extract the alkaline portion once or twice more to learn if acid dyes are also present. In the presence of both, add NaOH to the main portion until of normal alkalinity, shake with ether, and remove the basic dyes from the combined ether extracts first with N/64  $C_2H_4O_2$  and finally with dilute HCl. Omit this treatment if acid dyes are absent, since most basic dyes (especially auramin) decompose in alkaline solution. The basic dyes may be further fractionated from amyl alcohol with dilute HCl, from ether with dilute alkali, etc. After removal of the basic dyes with ether add to the alkaline solution HCl to normal strength and shake with amyl alcohol-gasoline. If a color is extracted it will probably be a minor portion of one obtained according to sec. 8 and may be further fractionated with the main portion or separately. Reduce the N HCl to N/4 by adding NaOH and shake with carbon tetrachloride-dichlorhydrin (3 : 1) to extract lower sulphonated triphenylmethane dyes, then add more tetrachloride and wash out these dyes with water.

12. Fraction any monosulphonated monazo dyes in the acetic acid solutions of sec. 10 (chief part) and sec. 8 (small part) with amyl alcohol and N  $NaCO_3$  or with ether and dilute HCl.

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\* Witt, Zeits. anal. Chem., 26, 1887, p. 100; Weingärtner, *ibid.*, 27, 1888, p. 232.



13. The main part of the eosins and unsulphonated water-soluble dyes are found in the N/64 NaOH of sec. 10. Fraction the eosins between N NaOH and amyl alcohol or amyl alcohol-gasoline (3 : 1). Acid dyes with basic tendencies (Nos. 510, 95, etc.), differ from the others in being extracted in smaller amount from strongly than from weakly acid solutions (pages 871 and 872). The amyl alcohol-gasoline solution (sec. 8) may also contain these dyes although usually in small amount. Most natural colors appear in the N/64 NaOH solution.

14. Moisten the amyl alcohol-gasoline residue (sec. 10) with a small drop of alcohol, add ether, and N/64 HCl, and shake. If the acid layer is colored (rhodamins, possibly basic dyes) wash the ether with further portions. If the ether is colored, wash a few times with 4N HCl, neutralize the washings, and treat according to sec. 15. Finally wash the acid out of the ether with water, evaporate to dryness on the water-bath, and treat the residue according to sec. 16.

15. Dissolve the oil soluble dyes from the neutralized solution (sec. 14) in gasoline and fraction with 70 to 90% methyl alcohol (page 875). Ortho-tolueneazo- $\beta$ -naphthylamine, although decomposing rather rapidly in strongly acid solutions, like its lower benzene homologue, is extracted slowly by acid from an ether solution. Probably the dyes undergo rearrangement before forming water-soluble salts, both forms decomposing by prolonged standing with acid.

16. Add to the ether residue (sec. 14) methyl alcohol, water, and NaOH solution, sufficient to make the alcohol strength 80% and the alkalinity N/4, and shake with gasoline. No. 666 and  $\alpha$ -naphthol derivatives remain chiefly in the alkaline liquid. Treat the gasoline again, if necessary, then according to sec. 17.

17. Separate sudans further with gasoline and 90% methyl-alcohol. Shake the gasoline solution with 85% phosphoric acid to which has been added 10 to 20% of  $H_2SO_4$ , thus separating from oily impurities although with probable destruction of some of the dye. These colors, like those of secs. 15 and 16, may be almost quantitatively removed from gasoline by 90% phenol and purified by redissolving in alkali and again taking up with ether or gasoline.

18. To avoid decomposition of No. 398 in acid solution, extract the neutral NaCl solution with dichlorhydrin, wash once with benzene to remove the dissolved solvent, make N/2 with HCl, and shake with anilin, adding the latter first. Fraction from the anilin solution with N/4 to N/64 HCl containing 5 to 6% NaCl.



(c) *Bromine Test*.—This is useful in examining the fractions for azo and azin dyes in the presence of natural colors. To 5 cc. of the solution (0.005 to 0.01%) add drop by drop slightly more 1% bromine water than is required to destroy the dye, then a few drops of 3% hydrazin sulphate solution. To half of the solution add a few drops of freshly prepared 10 to 20% alcoholic  $\alpha$ -naphthol and an excess of  $\text{Na}_2\text{CO}_3$ ; to the other half only  $\text{Na}_2\text{CO}_3$ . With azo compounds sodium formate may be substituted for the hydrazin salt. The reactions belong in classes as follows:

A. Azo dyes which with bromine in acid or neutral solution become colorless, pale yellow or orange and with hydrazin sulphate are colorless, pale brown, or pink, the color being more marked when nearly neutral. With  $\alpha$ -naphthol and  $\text{Na}_2\text{CO}_3$  a pronounced color appears but with  $\text{Na}_2\text{CO}_3$  alone no marked coloration. If the first component of the original dye was an unsulphonated amin the new color (*e*) may be removed by ether from the alkaline solution imparting usually an orange color changing with a large excess of strong HCl in most cases to violet or blue; if the new dye (*w*) is sulphonated it will not be extracted by ether. The new dye may be fixed on wool from a suitable solvent and identified by spot tests. The following dyes belong in this class. A (*e*): 14, 21, 20, 53, 55, 56, 146, 154, 13, 54, 157, 26, 10. A or C (*e*): 17, 18. A (*w*): 108, 8, 9, 89, 399, 106, 94, 105, 164, 169, 163, 170, 84, 328, 85, 86, 97, 139, 95, 88, 92, 102. A or AA (*w*): 107, 93, 103, 139, 101. A or C: 197, 201. A (imperfectly) 318, 287, 220, 269, 240. Of the oil soluble colors in the tables all belong in class A but quinophthalon which in 60%  $\text{C}_2\text{H}_4\text{O}_2$  gives reactions of class B. Class A also includes 277 and 78, the latter requiring addition of some alcohol before  $\alpha$ -naphthol.

AA. Azo dyes reacting like those of class A in HCl (N/2 or above) solution. In neutral solution the reaction is less trustworthy as other oxidations take place, blue and other colors being produced with  $\text{Na}_2\text{CO}_3$  alone which, excepting 62, 64, and 65, is less intense than those of the original dyes. Bromine bleaches 62, 64, and 65 in N/4  $\text{Na}_2\text{CO}_3$  but an intense blue appears on adding hydrazin sulphate. AA (*e*) includes 62, 64, and 65, AA (*w*), 188.

B. Azin derivatives, etc., bleached by bromine in neutral or acid solution, the color being restored by hydrazin sulphate. With typical members the color may be again bleached and restored.  $\text{Na}_2\text{CO}_3$ , also  $\alpha$ -naphthol and  $\text{Na}_2\text{CO}_3$  give no change other than that shown by the original dye with alkali. Class B includes 604, 605, 667, 606, 534, and 562. 546 reacts imperfectly and 584 belongs to class B or C.



C. Dyes giving precipitates at dilutions as high as 0.01%. This class includes most basic dyes.

D. Dyes giving marked color changes in neutral or faintly acid solution. As the color usually appears with a trace of bromine and is destroyed by an excess the reactions are unsatisfactory. Hydrazin sulphate produces no color change except that due to removal of excess of bromine. The color with  $\alpha$ -naphthol plus  $\text{Na}_2\text{CO}_3$  is the same as with  $\text{Na}_2\text{CO}_3$  alone. Most yellow colors become brownish, other colors usually ill-defined. In acid solution the dye, as a rule, is merely destroyed by bromine same as with class E. Class D includes 434, 435, 436, 480, 507, 438, and 433; class D or C, 496, 427, 428, 505, 499, 504, and 502.

E. Dyes similar to those of class D but showing with bromine no change other than bleaching. Class E includes 439, 440, 602, 692, 398, 4, 706, 710, 1, 329, 483, 512, 515, 516, 517, 518, 520, 521, 523, 2, 3, 6, 707, 468, 464, 442, 476, and 658, class E or C, 650, 425, 426, 451, 452; class E or D, 491, 462, 639, and 448.

F. Halogenated fluorescin derivatives and similar dyes very resistant to bromine. Non-fluorescent iodine compounds tend to become yellower and develop a green fluorescence. No. 510 gives eosin.

(d) *Nitrous Acid Test*.—Most common coal-tar dyes in dilute solution do not react readily, but some show marked changes due to diazotization of free amino groups, formation of nitroso compounds, or direct oxidation. To the water solution add 2 to 3 drops of strong HCl and 1 to 2 drops of 7% sodium nitrite solution. Add 1 cc. or an excess of 3% hydrazin sulphate solution, after standing a few minutes in case of blue and green dyes. To half of the solution, after  $\frac{1}{2}$  to 1 minute, add a few drops of alcoholic  $\alpha$ -naphthol solution and  $\text{Na}_2\text{CO}_3$  to strong alkaline reaction; to the other half for a check add only  $\text{Na}_2\text{CO}_3$ . The following gives reactions; others in the table pages 868 to 875 show no color changes except those due to the acid or alkali. 462:  $\text{NaNO}_2$ , blue, then colorless;  $\alpha$ -naphthol— $\text{Na}_2\text{CO}_3$ , orange. 439:  $\text{NaNO}_2$ , yellow. 491:  $\text{NaNO}_2$ , violet (slowly). 8:  $\text{NaNO}_2$ , much paler;  $\alpha$ -naphthol— $\text{Na}_2\text{CO}_3$ , deep blue (Hesse). 9:  $\text{NaNO}_2$ , much paler;  $\alpha$ -naphthol— $\text{Na}_2\text{CO}_3$ , red (Hesse). 89: red solution;  $\text{NaNO}_2$ , yellow; hydrazin sulphate, red again. 692:  $\text{NaNO}_2$ , slowly oxidized to yellow isatin derivative. 398:  $\text{NaNO}_2$ , brown. 21:  $\text{NaNO}_2$ , slightly darker;  $\alpha$ -naphthol— $\text{Na}_2\text{CO}_3$ , dull green black. 318:  $\text{NaNO}_2$ , pale and redder. 480:  $\text{NaNO}_2$ , slowly attacked. 84:  $\text{NaNO}_2$ , redder. 507:  $\text{NaNO}_2$ , bluer. 85:  $\text{NaNO}_2$ , paler. 95 and 88: HCl, crimson;  $\text{NaNO}_2$ , yellow; hydrazin sulphate, crimson. 101:  $\text{NaNO}_2$ , paler. 220 and 229:



$\text{NaNO}_2$ ; slightly paler. 562: scarcely attacked; in 50% acetic acid  $\text{NaNO}_2$  gives same change as bromine. 584:  $\text{NaNO}_2$ , blue; hydrazin sulphate, red. 448:  $\alpha$ -naphthol- $\text{Na}_2\text{CO}_3$ , wine red; in acetic acid solution,  $\text{NaNO}_2$ , first blue, then colorless. 427:  $\text{NaNO}_2$ , reddish. 17 and 18:  $\text{NaNO}_2$ , paler;  $\alpha$ -naphthol- $\text{Na}_2\text{CO}_3$ , redder. 505, 499, 504, 502: may appear bluer with  $\alpha$ -naphthol- $\text{Na}_2\text{CO}_3$ . 16:  $\text{NaNO}_2$ , slowly destroyed. 7 and aminoazotoluene:  $\text{NaNO}_2$ , paler; other reagents red.

(e) *Cyanide Test*.—This is based on the reaction, discovered by Lange, of the second component of certain ortho-azo dyes with KCN the 3-sulphonic group being replaced by cyanogen. Heat 10 cc. of the color solution, 1 cc. of 20% KCN solution, 1 cc. of 20%  $\text{NH}_4\text{Cl}$  solution in a test-tube in a boiling water-bath for 5 to 8 minutes. Carry along tests with known dyes at the same time. The common nitro dyes become brownish or reddish, certain azo dyes react as follows: 108: warmed 8 minutes dye nearly all changed to orange or yellow substances; warmed until dark red (1 to 2 minutes), strongly acidified, and washed with 2 N HCl, practically no color removed; subsequent washing with N/4 HCl, blue red dye removed. Azorubin S. G.: apparently unchanged by cyanide. 106: solubility unchanged but much color destroyed on long heating; 10 cc. amyl alcohol extracts the color from the cyanide mixture acidified with 5 cc. conc. HCl and gives it up to N/4 HCl. 107: cyanide mixture pale brown; treated same as 106, color remains largely in amyl alcohol; color destroyed on long heating. 14, 20, 21, 52, 53, 62, 64: dye unchanged; cyanide mixture acidified with 1 cc. glacial  $\text{C}_2\text{H}_4\text{O}_2$  gives up little color to 5 to 10 cc. amyl alcohol. 15, 55, 56, 65: cyanide mixture pale brown; treated like preceding dyes, gives up most of the color to amyl alcohol.

(f) *Reduction and Reoxidation*.—The reagents are those introduced by Green, Yeomans, and Jones. Drop into the neutral solution a few particles of powdered sodium hydrosulphite ( $\text{Na}_2\text{S}_2\text{O}_4$ ). If no color change appears at once, warm and add more reagent, avoiding an excess. If reduction and consequent decolorization takes place shake thoroughly with air and, if no color reappears, warm and allow to stand a few minutes. If still practically colorless drop in a little potassium persulphate. Disregard slight yellowish or brownish tints produced by air or persulphate. The reactions with most of the dyes on pages 868–875 belong in three classes:

A. Decolorized (or nearly) with hydrosulphite; remain colorless (or nearly) with air and persulphate: 108, 8, 9, 89, 106, 107, 94, 398, 198, 14, 20, 93, 53, 55, 105, 4, 56, 62, 64, 65, 103, 139, 164, 163, 84, 1, 328, 85,



13, 86, 97, 54, 329, 139, 157, 95, 88, 92, 101, 102, 26, 220, 269, 2, 3, 10, 240, 197, 201, 17, 18; slowly decolorized, 21, 287, 78; bluer, then decolorized, 318, 169, 170, 146, 154; browner then decolorized, 277.

B. Decolorized with hydrosulphite but original color restored by air or persulphate: 440, 692, 483, 468, 464, 650, 639, 584, 448, 451, 452, 427, 428. The following are nearly decolorized with hydrosulphite or change to the colors noted and the original color is restored with persulphate: nearly decolorized, 462, 710, 438; slowly paler, 439, 442; much paler, 480, 510; much paler (with excess), 512, 515, 516, 517, 518, 520, 521, 523; pale olive, 602; pale orange, 604, 605; pale yellow, 606. The following are nearly decolorized with hydrosulphite and the color is partly restored with persulphate: 434, 435, 491, 496.

C. No change with hydrosulphite: 399, 667, 706, 546, 507, 707, 658, 425, 426, 505, 499, 504, 502. No. 476 is not readily reduced.

Certain dyes do not belong in any of the preceding classes: 436 becomes very slowly paler with hydrosulphite; 6 becomes dark, then pale and with persulphate pale reddish; 433 is paler with hydrosulphite and greener with persulphate; 534 and 562 in alkaline solution become red (slowly) and yellow respectively with hydrosulphite and the colors are restored by persulphate.



## EXTRACTION OF COLORS BY IMMISCIBLE SOLVENTS FROM VARIOUS SOLUTIONS (MATHEWSON).

Numbers of coal-tar dyes permitted by Federal rulings are in bold-faced type; numbers of natural colors are in italics. Dyes most common in foods are designated by \*\*\*, others found less often by \*\* and \*.

No. (Green).	Name of Color.	Color of Water Solution.	HCl to Water Solution.	Relative Amounts Extracted.					Other Solvents.
				Amyl Alcohol from 5-6% NaCl Solution.	Amyl Alcohol from HCl Solutions.	Amyl Alcohol- Gasoline (1:1) from HCl Solutions.	Ether.	Dichlor- hydrin from N NaCl Solution.	
***462	Acid fuchsin	Crimson	Little change	Little or none	4N or less, lit- tle or none	N or less, little or none	Acid, alkali, neutral solutions, little or none	Little	
434	Light green SF, bluish	Green	Strongly acid solution					5-20% solu- tion, larger part or nearly all	Washed dichlorhydrin-carbon tetrachlorid (1:3) from N/64 C <sub>2</sub> H <sub>5</sub> O <sub>2</sub> : Nos. 435 and 439, very little; No. 491, little; No. 440, half or more
435	Light green SF, yellowish	Green	low to col- orless ac- cording to dye concen- tration						
*436	Erioglaucin A	Green-blue							
439	Cyanol, extra	Blue							
491	Wool green S	Green-blue							
440	Patent blue	Blue		Less than half					
**602	Nigrosin, soluble	Blue-black	Little change	Little					
**108	Ponceau 6R	Magenta		Little or none	4N, larger part; N, little			Little or none	
***8	Acid yellow G	Yellow			4N, larger part; N, half or less; N/4 and be- low, little or none				Amyl alcohol-gasoline from 8N H <sub>2</sub> SO <sub>4</sub> : No. 8, little; No. 692, smaller part; No. 89, more than half; Nos. 107, 106 and 94, larger part. Aniline from 5% NaCl and HCl of different strength: N/2, Nos. 692, 107, and 94, almost all; N/4, No. 94, less than half; Nos. 8, 89, 602, 107, 106, and 398, nearly all; N/16, No. 94, little; No. 89, less than half; No. 602, about half; Nos. 106 and 107, more than half; No. 398, larger part; N/64, Nos. 107, 106, 94, and 398, little or none
9	Past yellow R	Yellow	Red						
**89	Brilliant yellow S	Yellow							
692	Indigo carmine	Blue	Little change						
399	Sun yellow	Yellow			4N, almost all, N, larger part				
***106	New coccin	Scarlet			4N and below, little or none				
107	Amaranth	Magenta							

Most of the dyes described on this page are usually accompanied by considerable amounts of subsidiary products, especially those similar chemically, but of a different degree of sulphonation.

Caramel and the colors (glucosides) of most red fruits resemble No. 462 in being only slightly extracted by amyl alcohol or dichlorhydrin.



EXTRACTION OF COLORS BY IMMISCIBLE SOLVENTS FROM VARIOUS SOLUTIONS—Continued.

No. (Green).	Name of Color.	Color of Water Solution.	HCl to Water Solution.	Amyl Alcohol from 5-6% NaCl Solution.	Amyl Alcohol from HCl Solutions.	Amyl Alcohol- Gasoline (1 : 1) from HCl Solutions.	Ether.	Dichloro- hydrin- Carbon Tetrachlorid (1 : 1 and 1 : 3).	Washed Ethyl Acetate from 2N HCl Solution.
94	Tartrazin	Yellow	No change	Little or none	N/4 and below, little or none		Neutral, acid, or alkaline solutions, little or none	N/4 HCl or N/64 C <sub>2</sub> H <sub>4</sub> - O, little or or none	Very little or none
*398	Naphthol green B	Green	Yellow, gradually	Similar to 94 but rap- idly decomposed					
605	Azocarmine B	Red	Little change	4N and N, larger part N/4, less than half					
604	Azocarmine G	Red							
188	Naphthol black B	Violet	Bluer	N and above, nearly all; N/4, about half; N/16 and below, lit- tle		4N, larger part; N and below, little or none			
***14	Orange G	Orange	No change			4N, smaller part; N, little			
21	Fast acid fuchsin B	Magenta	Slightly yellow	4N and N, larger part; N/4 over half; N/16, less than half					
318	Chicago blue 6B	Blue	Little change	N and above, nearly all; N/4, larger part; N/64, less than half		Same as 188			
20	Chromotrope 2R	Magenta	No change			4N, nearly all; N, smaller part; N/16 and below, little or none			
93	Azofuchsin G	Blue-red	Yellow			4N, less than half; N and below, little			
***480	Soluble blue	Blue	Slightly paler						
*53	Palatine scarlet	Scarlet	Little change	N/4 and above, nearly all; N/64, half or more		N, half or less; N/16 and below, little or none			
*55	Ponceau 2R	Scarlet							
105	Fast red E	Scarlet							
4	Naphthol yellow S	Yellow	Nearly colorless	Same as above but larger part at N/64					Larger part
***706	Cochineal	Orange-red	Little change						Little



## EXTRACTION OF COLORS BY IMMISCIBLE SOLVENTS FROM VARIOUS SOLUTIONS—Continued.

No. (Green).	Name of Color.	Color of Water Solution.	HCl to Water Solution.	Relative Amounts Extracted.						
				Amyl Alcohol from 5-6% NaCl Solution.	Amyl Alcohol from HCl Solutions.	Amyl Alcohol- Gasoline (1 : 1) from HCl Solutions.	Ether.	Dichloro- hydrin- Carbon Tetra- chlorid (1 : 3) from N/64, C <sub>2</sub> H <sub>4</sub> O <sub>2</sub> . Solution.	Washed Ethyl Acetate from 2N HCl Solution.	Amyl Alcohol- Gasoline (1 : 1) from N/64, NaOH Solution.
56	Ponceau 3R	Cherry	No change	Little or none	N/16 and above, almost all; N/64, larger part	N, more than half; N/16 and below, little or none	Neutral, acid, or alkaline so- lutions, little or none	Little or none	Little	Little or none
*62	Palatite red	Blue-red	Little change							
**64	Crystal Ponceau	Scarlet	Slightly darker							
*65	Bordeaux B	Magenta	Little change							
**103	Azorubin	Magenta	Little change							
139	Fast brown	Brown	Little change							
164	Crocein scarlet O, extra	Orange-red	Darker, fi- nally violet							
**667	Quinoline yellow water soluble	Yellow	No change	Intermediate between pre- ceding and succeeding groups. Of most of the dyes over half extracted	N/64 and above, nearly all	N, larger part; N/16, smaller part; N/64, little				
*169	Crocein scarlet 8B	Scarlet	Same as 164							
163	Biebrich scarlet	Orange-red	Darker							
170	Bordeaux G	Red	Darker, fi- nally blue- violet							
84	Resorcin yellow	Orange-yellow	Little change							
146	Brilliant crocein	Cherry	Same as 170							
287	Azo blue	Violet	Little change							
78	Erika B	Red								
**710	Azolitmin	Violet								

Nos. 169-710, especially the last three which may be partly precipitated, are difficultly soluble in both layers with most acid mixtures.



## EXTRACTION OF COLORS BY IMMISCIBLE SOLVENTS FROM VARIOUS SOLUTIONS—Continued.

Relative Amounts Extracted.											
No. (Green).	Name of Color.	Color of Water Solution.	Amyl Alcohol from 5-6% NaCl Solution.	Amyl Alcohol from HCl Solution.	Amyl Alcohol- Gasoline (1 : 1) from HCl Solutions.	Amyl Alcohol- Gasoline (1 : 1) from N/64 C <sub>2</sub> H <sub>5</sub> O <sub>2</sub> Solution.	Ether from HCl Solutions.	Dichlor- hydrin- Carbon Tet'rochlorid (1 : 3) from N/64 C <sub>2</sub> H <sub>5</sub> O <sub>2</sub> Solution.	Amyl Alcohol from N Na <sub>2</sub> CO <sub>3</sub> Solution.	Amyl Alcohol- Gasoline from N/64 NaOH Solution.	Ether from N/64 NaOH Solution.
*546 1	Alizarin red S Picric acid	Orange-yellow Yellow	Intermediate between pre- ceding and succeeding groups	N/64 and above, almost all	Same as 163			Little		Little or none	Little or none
507 328	Violamin R Brilliant yellow	Violet-red Yellow			N, more than half; N/16, half or less; N/64, smaller part						
606	Rosindulin 2G	Scarlet	Larger part								
154	Cloth red B	Magenta			N/16, half or more;	Little			Smaller part		
85	Orange I	Orange			N/64, less than half				Almost all		
**13 ***86 97	Crocein orange Orange 2 Orange R	Orange Orange Orange	Almost all		N/16 and above, nearly all; N/64, larg- er part			Smaller part			
54 329	Scarlet GR Chrysophenin	Red-orange Orange-yellow									
137 157 *95 88	Resorcin brown Bordeaux BX Metanil yellow Orange IV	Brown Brown-red Orange-yellow Orange-yellow				157, more than half; others less than half	4N, very lit- tle; N, less than half; N/64, little	Half or more		Less than half	

The dyes on this page are rather difficultly soluble with acid mixtures.



## EXTRACTION OF COLORS BY IMMISCIBLE SOLVENTS FROM VARIOUS SOLUTIONS—Continued.

No. (Green).	Name of Color.	Color of Water Solution.	Relative Amounts Extracted.						
			Amyl Alcohol from 5-6% NaCl Solution.	Amyl Alcohol from HCl Solutions.	Amyl-Alcohol Gasoline (1 : 1) from N/64 C <sub>2</sub> H <sub>5</sub> O <sub>2</sub> Solution.	Ether from HCl Solutions.	Amyl-Alcohol- Gasoline (1 : 1) from N/64 NaOH Solution.	Ether from NaOH Solutions.	Amyl Alcohol from N NaOH Solution.
*92 101	Azoflavin Fast brown N	Yellow Brown	Almost all	N/64 and above, near- ly all	Less than half	4N and N, larger part; N/64, little	Smaller part	N to N/64, little or none	Amyl Alcohol- Gasoline (3 : 1) from N NaOH Solution.
102	Fast red A	Red							
483	Rosolic acid	Red							
**310	Uranin	Yellow							
26	Metachrome orange R	Orange-yellow		N/64, nearly all	Nearly all	4N, very lit- tle; N, lit- tle; N/64, nearly all	No. 6, small- er part; others, lit- tles		
220	Chrysamin G	Brown-yellow							
269	Chrysamin R	Brown-yellow							
512	Eosin	Red							
515	Saffrosin	Red							
516	Erythrosin G	Red							
517	Erythrosin B	Red							
518	Phloxin	Red							
520	Rose bengale	Blue-red							
521	Eosin 10B	Blue-red							
*523	Rose bengale 3B	Blue-red	Nearly all						
2	Victoria yellow	Orange-yellow							
*3	Martius yellow	Yellow							
6	Aurantia	Orange-yellow							
534 ***707 *10	Alizarin Curcumin <sup>1</sup> Sudan G	Violet <sup>2</sup> Red-brown <sup>2</sup> Orange <sup>2</sup>							

<sup>1</sup> Principle of turmeric. For properties of other natural colors see p. 818.<sup>2</sup> Alkaline solution.



## EXTRACTION OF COLORS BY IMMISCIBLE SOLVENTS FROM VARIOUS SOLUTIONS—Continued.

No. (Green).	Name of Color.	Color of Water Solution.	Relative Amounts Extracted.							Ether from N/64 HCl or C <sub>2</sub> H <sub>5</sub> O <sub>2</sub> Solution.	Ether from N/64 NaOH Solution.	Dichloro- hydrin-Carbon Tetrachlorid from N/64 C <sub>2</sub> H <sub>5</sub> O <sub>2</sub> Solution.
			Amyl Alcohol from 5-6% NaCl Solution.	Amyl Alcohol from HCl Solutions.	AmylAlcohol- Gasoline (1 : 1) from N/64 HCl Solution.	AmylAlcohol- Gasoline (1 : 1) from N/64 C <sub>2</sub> H <sub>5</sub> O <sub>2</sub> Solution.	AmylAlcohol- Gasoline (1 : 1) from N/64 NaOH Solution.	AmylAlcohol- Gasoline (1 : 1) from N/64 NaOH Solution.	AmylAlcohol- Gasoline (1 : 1) from N/64 NaOH Solution.			
*468	Formyl violet S 4B	Violet	Nearly all	4N very little; N/64, nearly all	Little	Little	Little	Little	Little or none	Little or none	1 : 1, nearly all; 1 : 3, half or more	
464	Acid violet N	Violet			Over half	Over half	Larger part					
438	Night green 2B	Green		N/64, nearly all	Smaller part	Smaller part	Smaller part					
**433	Guinea green B	Green		Partly								
442	Patent blue A	Blue		N/64, nearly all	Some extd.; rest pptd.				Little or none; pptd. by HCl			
476	Methyl alkali blue	Blue			Pptd.	Pptd.	None					
240	Congo red	Red	Extd. from neutral or slightly alk. sol.	Pptd.								
277	Benzopurpurin 4B	Brown-red	Nearly all pptd.	Nearly all pptd.; rest extd.								
562	Alizarin blue	Blue to green <sup>1</sup>			Very little extd.							
638	Thioflavin T	Yellow	Nearly all extd.						Little or none			
496	Rhodamin S	Red										
650	Methylene blue	Blue										
639	New blue	Violet										
*584	Safranin	Red										
**448	Fuchsin	Crimson										

<sup>1</sup> Alkaline solution.







## EXTRACTION OF COLORS BY IMMISCIBLE SOLVENTS FROM VARIOUS SOLUTIONS—Concluded.

Relative Amounts Extracted.									
No. (Green).	Name of Color.	Color of Ether Solution.	Amyl Alcohol from Various Aqueous Solutions.	Amyl Alcohol- Gasoline (1 : 1).	Ether from HCl Solutions. <sup>1</sup>	Gasoline from Methyl Alcohol of Different Volume Percentages.			
						90%	80%	70%	80% with N/4 NaOH.
**16	Butter yellow	Orange-yellow	Oil soluble colors insol- uble in water extracted from aqueous solvents al- most com- pletely	Water, N/64 C <sub>2</sub> H <sub>5</sub> O <sub>2</sub> , or N/64 HCl, extracted	4N, very little; N/64, nearly all	Smaller part	Less than half	Nearly all	About half
7	Aniline yellow	Orange-yellow				Very little	Very little	Very little	Very little
*	Yellow fat color	Orange-yellow				Little	Smaller part	Smaller part	Smaller part
	Benzeneazo-β-naphthyl- anilin	Orange-yellow				Smaller part	Much	Chief part	Much
	Ortho-tolueneazo-β- naphthylamine	Orange-yellow				Smaller part	Over half	Over half	Over half
666	Quinolin yellow, spirit soluble	Yellow			4N, nearly all; N/64, all	Little	Smaller part	Smaller part	Very little
59	Benzeneazo-α-naphthol  Sudan brown	Brown  Brown				Very little	Very little	Very little	
	Ortho-tolueneazo-o- tolueneazo-α-naphthol Sudan I	Brown-red  Orange			4N, larger part; N and below, all	Smaller part	Larger part	Larger part	
*11	Sudan II	Orange-red			4N and below, practically all	Larger part	Nearly all	Nearly all	Little
**49	Carminaph garnet	Red							Half or more
60	Sudan III	Red							Larger part
*143	Sudan IV	Red							Nearly all
31	Para red	Orange				Smaller part	Larger part	Larger part	Little

<sup>1</sup> Colors are completely extracted by ether from N to N/64 C<sub>2</sub>H<sub>5</sub>O<sub>2</sub>.



## CHAPTER XVIII.

### FOOD PRESERVATIVES.

**Preservation of Food.**—Various processes have from ancient times been known and used for arresting the fermentative changes which food products in their natural state undergo on long standing. These processes include pickling with vinegar, drying, smoking, salting, preserving with sugar, and finally in the employment of heat in sterilizing and pasteurizing, and of low temperature as in cold storage. All of them are still in use, and are universally regarded as unobjectionable. In addition to these old and well-known methods of food preservation is the comparatively modern practice of arresting fermentation by the use of such antiseptic chemical agents as formaldehyde, beta-naphthol, boric, salicylic, benzoic, and sulphurous acids or salts of these acids, etc., in regard to the wholesomeness of which there is considerable difference of opinion. These substances depend for their efficiency on the more or less complete inhibition of bacterial growth. Nearly all exert a powerful antiseptic influence, to such an extent that to accomplish their object only small quantities need be used in food.

Apart from their toxic effects, a marked difference naturally exists between the employment of such substances as salt, sugar, and vinegar for food preservation, all of which are in themselves foods, and in the use of chemical agents that have no food value. The advocates of the use of chemical antiseptics claim that there are no authentic instances on record of injury from the use of such small quantities of these substances as are necessary to arrest decay, while there are many cases of injury arising from the use of foods which, while apparently wholesome, have undergone such fermentation as to develop ptomaines or other harmful toxins, and that because antiseptics prevent such spoiling of the food, their use is decidedly beneficial; that there is, besides, no more reason why a prejudice should exist against the employment of these



newer chemicals than against saltpeter, which has long been used in the corning of meat, or against the cresols and phenols left as a product of smoking.

The opponents to their use assert, that the addition to food of such antiseptic substances as prevent its decay also serves to retard the digestive processes when the food is eaten; that many of these substances are drugs, and as such cannot fail even in small quantities to exercise a toxic effect of some sort on the system; that finally their use is objectionable, as allowing the employment in certain foods of old materials that have in some cases already undergone incipient decomposition before the addition of the antiseptic, and are thus unwholesome.

**Regulation of Antiseptics in Food.**—In the absence of legislation directly prohibiting the use of any of the above-named antiseptics, and in view of the difference of opinion regarding their toxic effects when present in small quantities, it is difficult to maintain a complaint under the general food laws as they exist in most states, basing the complaint solely on their harmfulness. In some localities certain antiseptics are specifically allowed and others are prohibited. Some of the states, as, for example, Massachusetts, have special laws under which it is required that in the case of all foods thus treated, the name and percentage of such antiseptics as are used must appear plainly on labels of the packages or containers thereof, such a provision being based on the assumption that the general public should be informed of what they are buying, where any doubt exists as to the wholesomeness of any ingredient present. Where such laws as these are in force, the chemist's task is comparatively easy, in that conviction in court is not dependent on his individual opinion regarding the toxic effects of the antiseptic employed.

Physiological experiments for testing the toxicity of these chemical preservatives were formerly confined to the lower animals, but no satisfactory results could be thus obtained. Later, metabolism experiments were made on human beings treated with varying amounts of the preservatives under carefully controlled conditions, but the results of these, though made by experts of unquestioned ability, do not agree. Even if any of these substances as used in food appear to have little or no effect on people in good health, they cannot be assumed to be equally harmless to those who are inclined to be delicate or sickly. Even though pronounced harmless in themselves, there is still the objection that the chemical preservatives may readily conceal unclean methods or materials. If perishable foods are free from preservatives and are sweet and



untainted, the consumer has reason to believe that clean and wholesome materials and sanitary processes were employed throughout in their manufacture.

**Commercial Food Preservatives.**—A large number of commercial preparations are sold for purposes of preserving specific articles of food and are put out under trade names that usually convey no suggestion of their true character. Some of these consist of a single antiseptic substance, such as salicylic acid, ammonium fluoride, calcium sulphate, borax, or benzoic acid, while others are mixtures of several antiseptics, of which the following are typical examples, showing their composition as found, together with the amount of the mixture to be employed.

**A.** For preserving sausage meat, using 8 ounces per 100 pounds of meat:

Borax.....	36%
Salt.....	46%
Saltpeter.....	18%
(Colored with an anilin dye.)	

**B.** For preserving cider and ketchup.

A 34% solution of beta-naphthol in alcohol, using 2 fluid ounces to 45 gallons of cider, or 1½ ounces to 10 gallons of ketchup.

**C.** For preserving beer, using 1½ ounces per barrel of beer:

Salt.....	45%
Salicylic acid.....	27%
Sodium carbonate and salicylate.....	28%

**D.** For preserving chopped meats, using 1 ounce to 50 pounds of meat:

Sodium sulphite.....	65%
Borax.....	35%

**E.** Effective for curing beef, hams, tongues, bacon, pig's feet, etc.:

Borax.....	28%
Boric acid.....	12%
Sodium chloride.....	35%
Potassium nitrate.....	25%

**F.** For preserving milk and cream:

Boric acid.....	75%
Borax.....	25%



*G.* For preserving jellies, jams, preserves, mince-meat, and syrups, using from 1 to 2 ounces of preservative to 100 pounds of product:

Sodium benzoate. . . . .	50%
Boric acid. . . . .	40%
Sodium chloride. . . . .	5%
Sodium bicarbonate. . . . .	5%

*H.* For preserving ketchup and tomato pulp, using from 6 to 8 ounces to 45 gallons of the product:

Sodium benzoate. . . . .	50%
Sodium chloride. . . . .	40%
Sodium sulphite. . . . .	10%

*I.* Effective for keeping butter from becoming tainted or rancid, also for salt codfish, using 8 to 12 ounces per 100 pounds butter:

Boric acid. . . . .	25%
Borax. . . . .	50%
Sodium chloride. . . . .	25%

*J.* For preserving eggs (surface application). A saturated solution of salicylic acid in 3 quarts of water, 1 quart strong alcohol and 7 ounces of glycerin.

#### FORMALDEHYDE.

**Formaldehyde** (HCHO) is a gas formed by the action of a red-hot spiral of platinum wire on vaporized methyl alcohol. It is also produced by the dry distillation of calcium formate. In the market it commonly appears in the form of a 40% solution of the gas in water under the name of formalin, and for use as a food preservative dilute solutions of from 2% to 5% strength are usually employed. Its use as a food preservative is comparatively modern.

The prompt and direct action of formaldehyde in checking or preventing the growth of lactic acid bacteria renders it especially desirable for use as a milk and cream preservative, from the standpoint of the dairyman who does not concern himself as to whether or not its use is injurious or illegal. The common proportion of 1 part of formaldehyde to 20,000 parts of milk will keep milk sweet for four days in Summer weather.

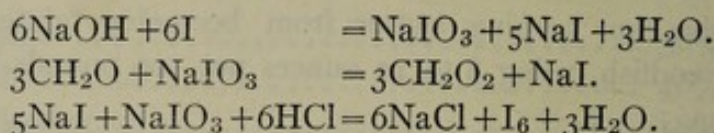
Small amounts of formaldehyde occur naturally in certain foods. For



example, Kawahata and Namba \* have detected it in smoked meats and Ishida † in crab meat, especially in preserved crab meat kept several months.

**Determination of Formaldehyde in the Commercial Preservative.—**

(1) *Iodometric Method.*‡—Mix 10 cc. of the aldehyde solution (diluted if necessary to a strength not exceeding 3% of formaldehyde) with 25 cc. of tenth-normal iodine solution, and add drop by drop a solution of sodium hydroxide, till the color of the liquid becomes clear yellow. The solution is set aside for at least ten minutes, after which hydrochloric acid is added to set free the uncombined iodine, and the latter is titrated back with tenth-normal thiosulphate. Two atoms of iodine are equivalent to one molecule of formaldehyde, in accordance with the following reactions:



(2) *Method of Blank and Finkenbeiner.*§—Three grams of the solution are weighed into a tall Erlenmeyer flask, to which is then added from 25 to 30 cc. of twice-normal sodium hydroxide. Fifty cc. of pure 2.5% to 3% hydrogen peroxide solution are next gradually run in during a space of from three to ten minutes, through a funnel placed in the neck of the flask to prevent spurring, and the solution is allowed to stand for two or three minutes, after which the funnel is washed with water.

Finally the unused sodium hydroxide is titrated with twice-normal sulphuric acid, using litmus as an indicator. The less formaldehyde in the sample, the longer the mixture should stand after addition of the hydrogen peroxide, to complete the reaction. When less than 30% is present, it should stand at least ten minutes.

Ascertain the percentage of formaldehyde, by multiplying by 2 the number of cubic centimeters of soda solution used, when 3 grams of the sample are taken.

(3) *Ammonia Method.*||—Weigh 10 grams of the formaldehyde solution into a flask, and treat with an excess of ammonia. Cork the flask

\* Jour. Pharm. Soc., Japan, 432, 1918, p. 95.

† Ibid., 422, 1917, p. 300.

‡ Zeits. anal. Chem., 1897, 36, pp. 18-24.

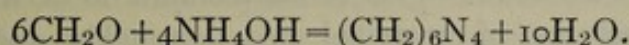
§ Ber., 31 (17), 2979.

|| Conn. Exp. Sta., Annual Report, 1899, p. 143.



and shake frequently during several days. The formaldehyde is by this process converted into hexamethylamine.

Transfer the solution to a tared platinum dish, and evaporate nearly to dryness on the top of a closed water-bath. Finally the dish is transferred to a desiccator, and the drying continued over sulphuric acid to constant weight. The per cent of formaldehyde is calculated from the weight of the hexamethylamine, making a correction for the residue left by the formaldehyde itself by direct evaporation:



Or an excess of a standardized ammonia solution may be added in the first place, the excess of ammonia being distilled off and titrated with standard acid, calculating the per cent of formaldehyde by the amount of ammonia absorbed.

**Detection of Formaldehyde.**—Methods have previously been given for the detection of formaldehyde in milk. For other materials acidify a portion of the sample with phosphoric, sulphuric, or citric acid, subject to distillation, and test the first few cubic centimeters of the distillate as follows:

*Leach Test.*—Add a few drops of the suspected distillate to about 10 cc. of pure milk (previously proved free from formaldehyde) in a porcelain casserole, and carry out the test as described on page 165.

*Hehner Test.*—Apply the test as described on page 165 to 10 cc. of pure milk to which a few drops of the suspected distillate have been added.

*Rimini Test.\**—Mix 20 cc. of the distillate with 1 cc. of phenylhydrazine hydrochloride solution (4 : 100) and 4 drops of freshly prepared sodium nitroprusside solution (1 : 200) and finally add concentrated sodium hydroxide solution drop by drop to the mixture. Formaldehyde is indicated by the appearance of a blue or, in dilute solutions, a green coloration which changes to red on standing. When formaldehyde is absent, only the red color appears.

*Arnold and Mentzel†* shake 5 grams of meat or melted fat with 10 cc. of alcohol, or 10 cc. each of milk and alcohol, and filter, then add to 5 cc. of the filtrate 0.03 gram phenylhydrazine hydrochloride, 4 to 5 drops of 1% ferric chloride solution, and, with agitation in a bath of cold

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\* Anal. farm., 1898, p. 97.

† Zeits. Unters. Nahr. Genussm., 5, 1902, p. 353.



water, 10 to 12 drops of concentrated sulphuric acid. A red color indicates formaldehyde.

*Barbier and Jandrier Test.*\*—According to Williams and Sherman † this test is especially trustworthy. Mix 5 cc. of the distillate with 0.2 to 0.3 cc. of a saturated alcoholic solution of gallic acid and pour the mixture into 3 to 5 cc. concentrated sulphuric acid in a test-tube. A green zone slowly changing to blue at the juncture of the liquids indicates formaldehyde.

*Lebbin Test.*—To about 10 cc. of the distillate to be tested, add a few drops of a 1% solution of resorcinol, mix thoroughly, and carefully pour the liquid down the side of a test-tube containing concentrated sulphuric acid. In the presence of formaldehyde, a rose-red zone is formed at the junction of the two liquids, sensitive to 1 part in 200,000. If formaldehyde be present to an extent exceeding 1 part in 100,000, a white turbidity or precipitate is formed above the colored zone.

*Schiff's Reagent* (one gram of fuchsin dissolved in water, 20 cc. saturated sodium hydrogen sulphite solution, and 10 cc. concentrated hydrochloric acid, made up to 1 liter) gives a pink coloration when a drop is added to a few drops of the distillate containing any aldehyde and is therefore a group reaction and not characteristic of formaldehyde.

*Fincke* ‡ states that employing the Grosse-Bohle reagent (25 grams crystallized sodium sulphite dissolved in a solution of 1 gram of rosanilin hydrochloride or acetate in 500 cc. of water, treated with 15 cc. 25% hydrochloric acid, diluted to 1 liter, and allowed to stand several hours) a test is obtained with which ordinary amounts of other aldehydes do not interfere, although hexamethylenetetramine reacts in a similar manner. He proceeds as follows: Mix 10 cc. of the solution to be tested with 1 to 2 cc. of 25% hydrochloric acid and decolorize by shaking or warming with purified animal charcoal or with the addition of mercuric chloride in the case of meat products or mercuric acetate in the case of fruit products. Filter and shake the filtrate with 1 cc. of the reagent. The blue or blue-violet color indicative of formaldehyde should appear within twelve hours.

To detect hexamethylenetetramine, heat the solution to be tested, after mixing with the hydrochloric acid, in a water-bath for ten minutes, cool, and then add the reagent or else test the distillate obtained in the usual manner.

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\* Ann. chim. anal. app., 1, 1896, p. 325.

† Jour. Amer. Chem. Soc., 27, 1905, p. 1497.

‡ Zeits. Unters. Nahr. Genussm., 27, 1914, p. 246.



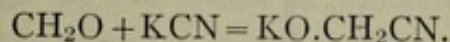
**Quantitative Determination of Formaldehyde**, especially in the case of milk (page 165) and other products containing proteins, is unsatisfactory. Results by the following method should therefore be reported as recoverable formaldehyde.

*Romijn Method.\**—Treat 10 cc. of tenth-normal silver nitrate with 6 drops of 50% nitric acid in a 50-cc. flask, add 10 cc. of a solution of potassium cyanide containing 3.1 grams of KCN in 500 cc. of water, and make up to the 50-cc. mark. Shake, filter, and titrate 25 cc. of the filtrate with tenth-normal ammonium sulphocyanate, using ferric chloride as an indicator.

Acidify another portion of 10 cc. of tenth-normal silver nitrate with nitric acid, add 10 cc. of the potassium cyanide solution to which the above 20 cc. of the formaldehyde distillate has been added. Make up the whole to 50 cc., filter and titrate as before—25 cc. of the filtrate with tenth-normal ammonium sulphocyanate for the excess of silver.

The amount of potassium cyanide used up by the formaldehyde, in terms of tenth-normal ammonium sulphocyanate, is found by multiplying by 2 the difference between the two results, and the total formaldehyde is calculated by multiplying by 3 the amount found in the 20 cc. of distillate.

The reaction that takes place between the formaldehyde and the potassium cyanide probably results in the formation of an addition product as follows:



#### BORIC ACID.

Boric or boracic acid is commonly obtained in impure form from lagoons or fumaroles of volcanic origin in Tuscany. It is afterwards purified by recrystallization. It is weakly acid, and readily soluble in water and in alcohol. Its alcoholic solution, even when the acid is present in small quantity, burns with a characteristic green flame. The acid is quite volatile with steam.

*Borax*, the most commonly known salt of boric acid, is found native in Italy, California, and elsewhere, and is also made from boric acid. It is mildly alkaline, and readily soluble in water.

Boric acid and borax, either used separately or mixed, have long been used as preservatives, especially in animal foods. A mixture of 3 parts

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\* Zeits. anal. Chem., 36, 1897, p. 18.



boric acid and 1 part borax has been found very effective as a milk and butter preservative, as well as for meat products. It also has been used in fruit products, wines, beer, and temperance beverages.

Boric acid is quite widely distributed in nature. In small amounts it is a normal constituent of fruits including the grape, and consequently wines. It occurs in minute quantities in vegetables, meat, fish, eggs, and even milk. Mediterranean Sea water, according to Bertrand and Agulhon,\* contains 56.3 mg. of boric acid per liter. The amounts naturally present in foods are ordinarily too small to give decisive reactions with the turmeric tests employing the usual quantities.

**Determination of Boric Anhydride in Commercial Preservatives.—***Gladding Method.*†—A 150-cc. flask, Fig. 117, is arranged with a doubly

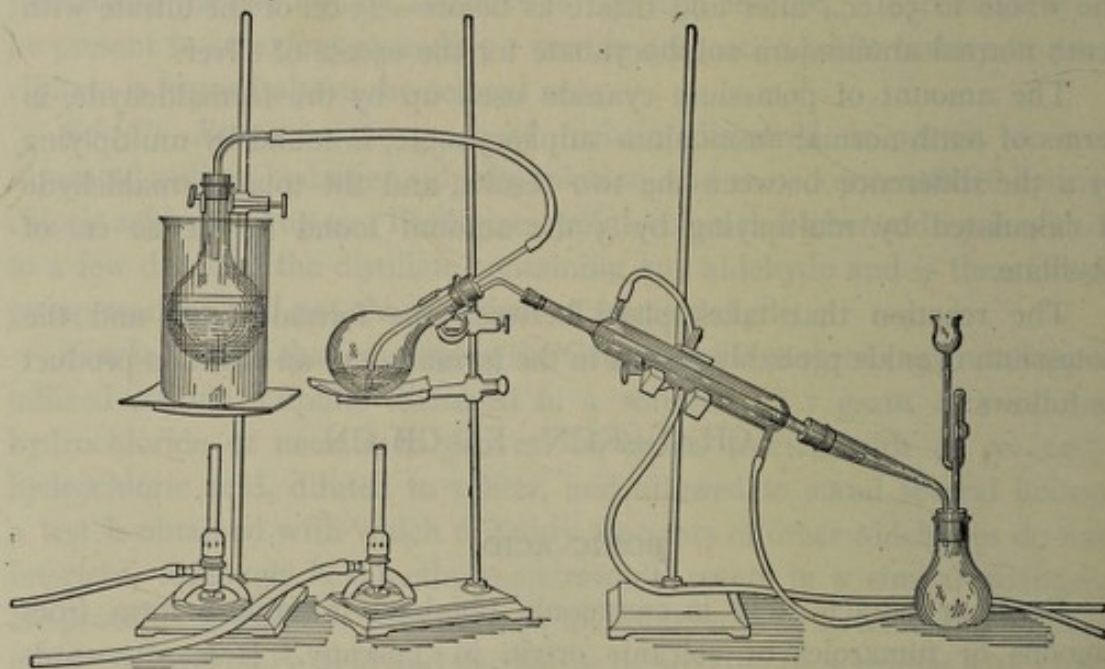


FIG. 117.—Apparatus for Determining Boric Acid According to Gladding.

perforated stopper having two tubes, one of which, the inlet-tube reaching nearly to the bottom, connects it with a larger flask, while the other or outlet-tube communicates with a Liebig condenser, which in turn delivers into a receiving-flask. In the 150-cc. flask, 1 gram of the powdered sample is placed, with about 20 cc. of 95% methyl alcohol and 5 cc. of 85% phosphoric acid. The larger flask is then filled two-thirds full of methyl alcohol, and heated on the water-bath after the apparatus has been con-

\* Bol. soc. chim., 15, 1914, p. 292.

† Jour. Amer. Chem. Soc., 20, 1898, p. 288.



nected up. Heat is also applied to the 150-cc. flask, the whole arrangement being such that a continuous current of methyl alcohol vapor bubbles through the liquid in the smaller flask, the heat being so regulated that from 15 to 25 cc. of methyl alcohol remains in the 150-cc. flask, while about 100 cc. of distillate passes into the receiving-flask in half an hour. Continue the distillation till all the acid has passed over, which is usually accomplished by distilling 100 cc. By a gentle aspiration upon the receiving-flask, loss by leaking may be avoided.

Prepare a mixture of 40 cc. of glycerin and 100 cc. of water, and carefully neutralize, using phenolphthalein as an indicator. Add this mixture to the distillate, and titrate the whole with tenth-normal sodium hydroxide. Run a blank with the reagents alone, deducting any acidity. For the factors for calculation see page 887.

**Detection of Boric Acid and Borates.**—These are tested for in the aqueous extract of the material itself or of the ash, the quantity to be used for the test depending largely on the case in hand. With meat products and canned goods, about 25 grams are either boiled up with water or first made distinctly alkaline with lime water, dried over the water-bath, and burned. The ash is boiled with from 10 to 15 cc. of water, and tests made on the solution. With such products as salt codfish, which is preserved by brushing or coating with boric mixture, portions of the coating may be scraped off and boiled in water, the tests being made on the aqueous solutions.

*The Turmeric-paper Test.*—The most delicate test for boric acid, free or combined, is made by the aid of turmeric-paper, prepared by soaking a smooth, thin grade of filter-paper in an alcoholic tincture of powdered turmeric. The paper is afterwards dried and cut into strips, which are kept for convenience in a wide-mouthed bottle in a dark place.

Acidulate the aqueous extract of the material or the ash with concentrated hydrochloric acid, equivalent to about 5 drops per 10 cc. in excess of what is necessary for neutralization. Then dissolve the ash in a few drops of water and thoroughly saturate a strip of the turmeric-paper in the solution. On drying the paper, if boric acid either free or combined be present, a cherry-red coloration will be imparted to the paper, the depth of color depending on the amount present. As a confirmatory test, apply a drop of dilute alkali to the reddened paper, and a dark-olive color will be due to boric acid, sharply to be distinguished from the deep-red color produced when an alkaline solution is applied to ordinary turmeric-paper. The turmeric-paper reaction is delicate to 1 part in 8000.



*Tincture of Turmeric Test.*—To the solution to be tested, slightly acidified with hydrochloric acid, add an equal volume of saturated tincture of turmeric in an evaporating-dish, and heat for a minute or two. A red color, light or dark, depending on the amount of the preservative, is produced if boric acid be present, changed to an olive color by the addition of dilute alkali, after cooling.

*The Flame Test.*—A few cubic centimeters of alcohol are added to the dish containing the slightly acidulated ash of the sample to be tested, or to the acidulated dried residue from the evaporation of the aqueous solution of the suspected preservative, and after mixing by the aid of a stirring-rod, the alcohol is ignited. In the presence of any considerable portion of free or combined boric acid, a greenish tinge will be observed in the flame of the burning alcohol, especially at the first flash due to the boric ether formed. This test is by no means as delicate as the turmeric paper test.

**Determination of Boric Acid in Foods.**—*Thompson Method.\**—Add 1 or 2 grams of sodium hydroxide to 100 grams of the sample, and evaporate to dryness in a platinum dish. Char the residue thoroughly, and boil with 20 cc. of water, adding hydrochloric acid drop by drop till all but the carbon is dissolved. In burning, avoid too high a heat, simply charring sufficiently to insure a clear solution with water. Transfer by washing to a 100-cc. graduated flask, taking care that the volume does not exceed 50 or 60 cc. Add half a gram of dry calcium chloride, then a few drops of phenolphthalein solution, and next a 10% solution of sodium hydroxide, till a permanent pink color persists. Finally add 25 cc. of lime-water. By this means all phosphoric acid is precipitated in the form of calcium phosphate. Make up to the 100-cc. mark with water, shake, and pour upon a dry filter. To 50 cc. of the filtrate add sufficient normal sulphuric acid to remove the pink color. Then add a few drops of methyl orange, and continue the addition of sulphuric acid till the yellow is just turned to pink. Tenth-normal sodium hydroxide is then added † till the liquid takes on a faint yellow, excess of alkali being avoided. The salts of the acids present at this time are all neutral to phenolphthalein except boric acid and carbon dioxide. Boil the solution to expel the carbon dioxide, cool, add a little more phenolphthalein, and a quantity of glycerin equal

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\* Jour. Soc. Chem. Ind., 12, 1893, p. 432.

† If the value of the standard alkali solution is not absolutely certain, it had best be restandardized against pure crystallized boric acid, 0.31 gram of which should neutralize 50 cc. of tenth-normal alkali.



in volume to the solution. Finally titrate with tenth-normal sodium hydroxide to a permanent pink color. Each cubic centimeter of tenth-normal sodium hydroxide equals 0.0062 gram crystallized boric acid,  $\text{H}_3\text{BO}_3$ , or 0.0035 gram boric anhydride,  $\text{B}_2\text{O}_3$ , or 0.00955 gram crystallized borax,  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$ .

*Gooch Method.*—Mix 400 to 500 grams of the substance with 10 grams of calcium hydrate, evaporate to dryness over a water-bath in a platinum dish and burn cautiously to an ash. Dissolve the residue in cold nitric acid, and add an excess of silver nitrate to precipitate the chlorine. Filter, make up to 500 cc. with water, shake, and measure out 25 cc. into a 200-cc. flask fitted with a stopper provided with an outlet-tube, and with a separatory funnel forming virtually a thistle-tube, capable of being closed with a glass stop-cock. Through the outlet-tube connect the flask with a Liebig condenser provided with an adapter which can dip below the liquid in the receiver. As a receiver, use a 150-cc. tared platinum dish, which contains a weighed quantity of ignited lime in water.

Add through the thistle-tube 10 cc. of methyl alcohol to the contents of the flask, close the stop-cock therein, and distil the contents in a paraffin-bath at a temperature of  $140^\circ \text{C}$ ., constantly stirring the liquid in the receiver to keep it alkaline during the distillation. Add five successive portions of methyl alcohol of 12 cc. each to the distilling flask, and continue the distillation till all the alcohol has passed over. Finally evaporate to dryness the contents of the platinum dish, and ignite over a blast-lamp to constant weight. Multiply the increased weight due to boric oxide by 2.728 to give the equivalent in borax.

#### SALICYLIC ACID.

Salicylic acid ( $\text{HC}_7\text{H}_5\text{O}_3$ ) is a white, crystalline, strongly acid powder, made synthetically by treatment of carbolic acid with sodium hydroxide and carbon dioxide, or naturally from methyl salicylate (which occurs in oil of wintergreen to the extent of about 90%) by treatment of the wintergreen oil with strong potash lye. Most of the commercial salicylic acid is of the synthetic variety. Pure salicylic acid crystallizes from alcoholic solutions in 4-sided prisms, and from aqueous solution in long, slender needles. It melts at  $155^\circ$  to  $156^\circ \text{C}$ . It is slightly soluble in cold water (1 part in 450), and much more so in hot water. It is readily soluble in ether, alcohol, and chloroform.

It is frequently found on the market as a food preservative in the form



of the much more soluble sodium salt, sodium salicylate ( $\text{NaC}_7\text{H}_5\text{O}_3$ ), which is, however, converted into salicylic acid when added to acid-fruit preparations, condiments, and liquors.

Sodium salicylate is a white, amorphous powder, soluble in 0.9 part water and in 6 parts alcohol. It is prepared by treating salicylic acid with a strong, aqueous solution of sodium carbonate, and afterwards purifying. If a known weight of the powdered preservative be ignited, and a solution of the ash titrated with tenth-normal sulphuric acid, using cochineal as an indicator, each cubic centimeter of the acid is equivalent to 0.0160 gram of sodium salicylate.

Salicylic acid is largely used as a preservative of jellies, jams, and fruit preparations, canned vegetables, ketchups, table sauces, wines, beer, and cider. It is rarely used in milk and milk products, or in meats.

Bucholz has shown that 0.15% of salicylic acid is sufficient to prevent bacteria from developing in ordinary organic substances, while as small a quantity as 0.04% produces a marked restraining influence.

Small amounts of salicylic acid occur naturally in grapes, strawberries, and other fruits, but the amounts are too small to give distinct color reactions when only 50 grams of the fruit products are used for tests.

**Detection of Salicylic Acid.**—If the sample to be tested is of a similar nature to jelly, jam, ketchup, cider, etc., or capable of getting into aqueous solution, slightly acidify the liquid or pasty material, diluted, if necessary, with weak sulphuric (if not already acid) and shake directly with an equal bulk of ether, petroleum ether, or chloroform, in a corked flask, or in a separatory funnel. If the sample be too thick in consistency to shake directly, macerate in a mortar with alkaline water, and strain through cloth. Acidify the filtrate with dilute sulphuric acid, and then proceed to shake with the immiscible solvent as above. Separate by decantation or otherwise the immiscible solvent containing the preservative, if present, and allow it to evaporate in an open shallow dish, either at room temperature or at a low heat. In case an emulsion forms on shaking, which is quite apt to happen, especially with ether for a solvent, divide the whole mixture between two tubes of a centrifuge of the form shown in Fig. 11, and whirl for three minutes at a high rate of speed. This usually serves to break up the most obstinate emulsion, so that it is easy to separate by decantation. If a considerable amount of salicylic acid be present, it will sometimes appear in the residue in the form of fibrous crystals.

**Ferric Chloride Test.**—To a portion of the dry residue obtained as above add a drop of ferric chloride solution. A deep purple or violet color indi-



cates salicylic acid. If doubt exists as to the color, dilute with water, which often serves to bring out a distinctive purple coloration otherwise unobservable.

Leach, instead of evaporating the ether solution of the salicylic acid to dryness, prefers to shake out the salicylic acid from the ether with dilute ammonia, evaporate the solution of ammonium salicylate nearly to dryness, and apply the tests given above to the concentrated solution. In this case the ether may be recovered.

Maltol ( $C_6H_6O_3$ ), occurring in caramelized products or products containing caramel, such as dark beer, also other substances named by Sherman and Gross,\* give a similar violet color, but the following test, recommended by Sherman† and Sherman and Gross, is characteristic only of salicylic acid.

*Jorissen Test.*‡—Add to the solution, obtained as above, in a test-tube 4 to 5 drops of 10% sodium or potassium nitrite, 4 to 5 drops of 50% acetic acid, and 1 drop of 1% copper sulphate solution, shaking after adding each reagent.

Heat in a boiling water-bath with liquid completely immersed for forty-five minutes, cool, and compare the red color indicative of salicylic acid with a blank test against a white background. Both Allen and Sherman and Gross have shown that benzoic, cinnamic, and tartaric acids do not respond to the test.

Schott,§ in the examination of milk, first removes interfering substances by adding to 25 cc. of the sample 10 cc. of Fehling copper sulphate solution and then sodium hydroxide solution until only faintly acid, and filtering.

*Methyl Salicylate Test.*—Another portion of the residue may be heated with methyl alcohol and sulphuric acid. If salicylic acid be present, the well-known odor of methyl salicylate will be produced.

*Ammonium Picrate Test.*—A portion of the dry ether extract is warmed gently with a drop of concentrated nitric acid, and 2 or 3 drops of ammonia are added. Yellow ammonium picrate will be formed if a considerable quantity of salicylic acid be present, and a thread of wool free from fat may be dyed by soaking therein. This test is by no means as delicate as the ferric chloride color test.

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\* Jour. Ind. Eng. Chem., 3, 1911, p. 492.

† Ibid., 2, 1910, p. 24.

‡ Bul. acad. roy. sci. let. beaux. Belg. [3] 3, 1882, p. 259.

§ Zeits. Unters. Nahr. Genussm., 22, 1911, p. 727.



**Determination of Salicylic Acid.**—*Dubois Method.*\*—In the case of catsups and similar pulped materials place 50 grams in a graduated 200-cc. flask, make slightly alkaline with ammonia, add 15 cc. of milk of lime (200 grams of quicklime in 2000 cc. water), complete the volume, shake and filter. Transfer 150 cc. of the filtrate to a separatory funnel, acidify with hydrochloric acid, and extract with four portions of 75 to 100 cc. of ether. Wash the combined extract twice with 25 cc. of water, and distil off the ether slowly, allowing the last 20 to 25 cc. to evaporate spontaneously. Dissolve the residue in a small amount of hot water, make up to a definite volume with water, and add to an aliquot portion a few drops of a 2% solution of ferric alum to develop the color. Estimate the amount of salicylic acid by matching the color thus obtained with that produced in a solution containing 1 mg. of salicylic in 50 cc., using either a colorimeter or Nessler tubes for making the comparison.

In the case of semi-solid materials, such as mince meat, jams, etc., macerate 50 grams with water in a mortar previous to treatment as above described.

Liquids and solutions of jellies and other materials free from pulp may be extracted with ether directly after acidifying.

#### BENZOIC ACID.

*Benzoic Acid* ( $\text{HC}_7\text{H}_5\text{O}_2$ ) is produced by the oxidation of a large number of organic substances, particularly toluene. It is also extracted by sublimation from gum benzoin, which exudes from the bark of the *Styrax benzoin*, a tree growing in Java, Sumatra, Borneo, and Siam. Most of the commercial benzoic acid is made from toluene by treatment with chlorine and subsequent oxidation.

Benzoic acid crystallizes in leaflets, having a silky luster. It is odorless when cold, is soluble in 200 parts of cold, and 25 parts of boiling water, and readily dissolves in alcohol, ether, and chloroform. Its melting-point is  $120^\circ$ , and it sublimes at a slightly higher temperature. It occurs naturally in the cranberry and other berries of the *Ericaceæ*.

*Sodium Benzoate* ( $\text{NaC}_7\text{H}_5\text{O}_2$ ) is the salt most largely used in commercial preservatives, being much more soluble than the acid itself, into which, however, it is converted when put into acid fruit preparations. Sodium benzoate is prepared by adding benzoic acid to a concentrated

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\* Jour. Amer. Chem. Soc., 28, 1916, p. 1616.



hot solution of sodium carbonate till there is no longer effervescence, and then cooling, and allowing the sodium benzoate to crystallize out. In titrating solutions of ignited sodium benzoate with tenth-normal sulphuric acid, each cubic centimeter of the standard acid is equivalent to 0.0144 gram of the benzoate.

Sodium benzoate is a white amorphous powder, having a sweetish, astringent taste, and is soluble in 1.8 parts of cold water, and in 45 parts of alcohol. It is used as a preservative of catsups, fruit products, soft drinks, wines, codfish, nut butter, and similar products. In England it is used in milk.

Long, Herter, and Chittenden of the Referee Board of Consulting Scientific Experts, after independent experiments, conclude that sodium benzoate in small doses (less than 0.5 gram per day) is not injurious to health and in large doses (up to 4 grams per day) has not been found to exert any deleterious effects on the general health nor to act as a poison in the general acceptance of the term. Accordingly this preservative is allowed under the federal law provided the presence and amount are declared on the label.\*

Many manufacturers do not use benzoate in any of their products, thus avoiding the obnoxious declaration of its presence or justifying a declaration of its absence.

**Detection of Benzoic Acid.**—Extract with ether or chloroform as directed for salicylic acid. If it is desired to test for both preservatives divide the extract into two parts and evaporate in separate dishes. A considerable amount of benzoic acid is apparent in the residue as shining crystalline scales or needles.

In the author's experience a better procedure than evaporating the ether solution is to extract the benzoic acid from the ether by shaking with dilute ammonia, evaporate the solution of ammonium benzoate nearly to dryness, and apply tests to the concentrated solution.

(1) *Ferric Chloride Test.*—A portion of the residue from the ether extract is dissolved in ammonia, and evaporated over the water-bath until neutral to test paper. The residue is stirred in a few drops of warm water, and filtered through a small filter into a narrow test-tube. A drop of neutral ferric chloride (prepared by precipitating a portion of the iron from a solution of the salt by ammonia and filtering) is added, and in the presence of benzoic acid a flesh-colored precipitate of ferric benzoate

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\* Food Inspection Decision, 104.



is produced, very characteristic and unmistakable, because of its peculiar color, when the solution in which the test is made is colorless. It occasionally happens, however, in the case of jellies, jams, and ketchups, that these preparations are artificially colored with a dyestuff that persists by its depth of color in obscuring that of the ferric benzoate, especially when only a small amount of benzoic acid is present. Again, in such products as sweet pickles, a precipitate of basic ferric acetate might also come down with the ferric benzoate, and thus confuse. In such cases one of the following methods should be carried out.

(2) *Sublimation Method*.<sup>\*</sup>—Evaporate an ammoniacal solution of the ether extract till neutral in a large watch-glass, by the aid of a gentle heat. Fasten with clips or otherwise a second watch-glass to the first, edge to edge, so as to form a double convex chamber, with a cut filter-paper between. Place upon a small sand-bath and heat. Benzoic acid, if present, will sublime upon the surface of the upper glass in minute needles, recognizable under the microscope. It may further be tested by determining the melting-point, or by treating with ammonia, evaporating, and applying the ferric chloride test as above.

(3) *Mohler Method Modified by Heide and Jakob*.<sup>†</sup>—Evaporate the ether extract to dryness, take up the residue in 1 to 3 cc. of third-normal sodium hydroxide, and evaporate to dryness. To the residue add 5 to 10 drops of concentrated sulphuric acid and a small crystal of potassium nitrate. Heat for ten minutes in a glycerol bath at 120° to 130° C. (never higher), or for twenty minutes in a boiling water-bath, thus forming meta-di-nitro benzoic acid. After cooling add 1 cc. of water and make decidedly ammoniacal; boil the solution, to break up any ammonium nitrite which may have been formed. Cool and add a drop of fresh colorless ammonium sulphide, without allowing the layers to mix. A red-brown ring (ammonium meta-di-amido benzoic acid) indicates benzoic acid. On mixing, the color diffuses through the whole liquid; on heating it finally changes to greenish yellow, owing to the decomposition of the amido acid, thus distinguishing benzoic from salicylic or cinnamic acids. Both the latter form amido compounds, which are not destroyed by heating. The presence of phenolphthalein interferes with this test.

(4) *Peter Oxidation Method*.<sup>‡</sup>—This method, depending on the for-

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<sup>\*</sup> Annual Report, Mass. State Board of Health, 1902, p. 486.

<sup>†</sup> Zeits. Unters. Nahr. Genussm., 19, 1910, p. 137.

<sup>‡</sup> U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 160.



mation of salicylic acid, is not applicable in the presence of this acid or saccharin, which also oxidizes to salicylic acid.

Transfer a portion of the residue, say 0.1 gram, from the ether or chloroform extraction to a large test-tube, and dissolve in from 5 to 8 cc. of concentrated sulphuric acid. Add from 0.5 to 0.8 gram of barium peroxide in successive small portions, shaking the tube in cold water. This should produce a permanent froth on the sulphuric acid solution. After standing for half an hour, fill the test-tube three-quarters full of water, shake, cool quickly, and filter. Extract the filtrate with ether or chloroform, and test the extract for salicylic acid.

The Jonescu test\* is a modification of the Peter method employing hydrogen peroxide. Peter in his original process used this reagent.

**Determination of Benzoic Acid.**—*La Wall and Bradshaw Method.*—*Modified.*—This process is based on principles brought to notice by Moerck.† Although originally devised for catsup,‡ it has been modified by Bigelow § and Dunbar || so as to be applicable to various classes of foods. The details which follow are those elaborated by Dunbar and adopted by the A. O. A. C.

1. *Preparation of Solution.*—(a) *General.*—Grind in a sausage-machine, if solid or semi-solid, and thoroughly mix. Transfer about 150 grams to a 500-cc. flask, add enough pulverized sodium chloride to saturate the water in the sample, make alkaline with sodium hydroxide or milk of lime, and dilute to the mark with saturated salt solution. Allow to stand at least two hours with frequent shaking and filter. If the sample contains large amounts of matter precipitable by salt solution follow a method similar to that given under (e); if large amounts of fats are present it is well to make an alkaline extraction of the filtrate before proceeding as directed under "Extraction and Titration."

(b) *Catsup.*—To 150 grams of the sample add 15 grams of pulverized sodium chloride. Transfer the mixture to a 500-cc. graduated flask, using about 150 cc. of saturated salt solution for rinsing. Make slightly alkaline to litmus paper with strong sodium hydroxide and complete the dilution to 500 cc. with saturated salt solution. Allow to stand at least two hours with frequent shaking and then filter through a large folded

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\* Jour. pharm. chim. [6], 29, 1909, p. 523.

† Penn. Pharm. Assn. Proc., 1905, p. 181.

‡ Amer. Jour. Pharm., 80, 1908, p. 171.

§ U. S. Dept. of Agric., Bur. of Chem., Bul. 122, p. 68.

|| Ibid., 1909, Bul. 132, p. 138; Circ. 66, p. 14



filter. If difficulty is experienced, centrifuge or squeeze the mixture through a muslin bag before filtering.

(c) *Jellies, Jams, Preserves, and Marmalades*.—Dissolve 150 grams of the sample in about 300 cc. of saturated salt solution. Add 15 grams of pulverized sodium chloride. Make alkaline to litmus-paper with milk of lime. Transfer to a 500-cc. graduated flask, and dilute to the mark with saturated salt solution. Allow to stand at least two hours with frequent shaking, centrifuge, if necessary, and filter through a large folded filter.

(d) *Cider and Similar Products Containing Alcohol*.—Make 250 cc. of the sample alkaline to litmus-paper with sodium hydroxide and evaporate on the steam-bath to about 100 cc. Transfer to a 250-cc. flask, add 30 grams of pulverized sodium chloride and shake until dissolved. Dilute to the mark with saturated salt solution, allow to stand at least two hours with frequent shaking, and filter through a folded filter.

(e) *Salt or Dried Fish*.—Transfer 50 grams of the ground sample to a 500-cc. flask with water. Make slightly alkaline to litmus-paper with strong sodium hydroxide and dilute to the mark with water. Allow to stand at least two hours with frequent shaking and filter through a folded filter. Pipette at least 300 cc. of the filtrate into a second 500-cc. flask, add 30 grams of pulverized sodium chloride for each 100 cc., shake until dissolved, and dilute to the mark with saturated salt solution. Mix thoroughly and filter off the precipitated protein matter on a folded filter.

2. *Extraction and Titration*.—Pipette a convenient portion of the filtrate (100 to 200 cc.), obtained as above, into a separatory funnel. Neutralize to litmus-paper with hydrochloric acid (1 : 3) and add an excess of 5 cc. In the case of salt fish, protein matter usually precipitates on acidifying, but this does not interfere with the extraction. Extract carefully with chloroform, using, for 200-cc. aliquots, successive portions of 70, 50, 40, and 30 cc., and proportional quantities for smaller aliquots. To avoid emulsion, shake each time cautiously. The chloroform layer usually separates readily after standing a few minutes. If an emulsion forms, stir the chloroform layer with a glass rod. If this does not break up the emulsion, draw it off into a second funnel and shake sharply once or twice. If this also fails, centrifuge the emulsion for a few moments. Draw off with great care as much of the clear chloroform solution as possible after each extraction. If not contaminated with the emulsion, it is unnecessary to wash the chloroform extract.

Transfer the combined chloroform extract to a dish, rinsing with chloroform, evaporate to dryness at room temperature, either sponta-



neously or in a current of dry air, and dry overnight (or, in case of catsup, until no odor of acetic acid can be detected) in a sulphuric acid desiccator. Dissolve the residue of benzoic acid in 30 to 50 cc. of neutral alcohol, add about one-fourth this volume of water, a drop or two of phenolphthalein solution and titrate with twentieth-normal sodium hydroxide. One cc. of the standard solution is equivalent to 0.0072 gram anhydrous sodium benzoate.

In the absence of a blast an electric fan may be used for evaporating the extract. If it is impracticable to evaporate the chloroform spontaneously or by means of a blast it may be transferred from the separatory funnel to a 300-cc. Erlenmeyer flask, rinsing the separatory funnel three times with 5 or 10 cc. of chloroform. Distil very carefully to about one-fifth the original volume, keeping the temperature down so that the chloroform comes over in drops, not in a steady stream. Then transfer the extract to a porcelain evaporating dish, rinsing the flask three times with 5 or 10 cc. portions of chloroform, and evaporate to dryness spontaneously.

The evaporation of the chloroform is best effected by delivering to the dish a blast of air dried by means of a calcium chloride bottle.

*Hilyer Method.\**—This method is valuable as a check on the La Wall and Bradshaw method. After titrating the benzoic acid obtained as described in the preceding section, proceed as follows:

Evaporate to dryness the accurately neutralized solution (which should not have even a slight alkaline reaction), and redissolve in a few cubic centimeters of alcohol saturated with silver benzoate. Filter if not clear, wash with a few drops of alcohol, and treat with 10 to 15 cc. of a saturated solution of silver nitrate in alcohol. Collect the precipitate in a Gooch crucible, care being taken that the asbestos filter is so prepared as to afford as rapid a filtration as possible, wash with alcohol, and finally with a little ether, heat in a water-oven until the ether is removed, cool, and weigh. Care must be taken to perform all the operations as quickly as possible to avoid separation of silver oxide.

*West's Distillation Method.†*—1. *Apparatus.*—The special form of double flask for distillation in a current of steam is the same as that employed by Hortvet ‡ in determining the volatile acids of wine (Fig. 115).

\* A. O. A. C. Proc. 1908, U. S. Dept. of Agric., Bur. of Chem., Bul. 122, p. 74; Circ. 66, p. 15.

† Jour. Ind. Eng. Chem., 1, 1909, p. 190.

‡ Ibid., 1, 1909, p. 31.



The steam tube leading from the outer to the inner flask, being introduced half-way up the side of the inner flask, makes it possible to connect the apparatus in such a way that at the beginning of the operation the water in the outer flask will reach to the height of the contents of the inner flask. The side tube leading from the neck of the outer flask is provided with a rubber tube and pinch-cock for use in relieving the steam pressure and avoiding the danger of drawing the contents of the inner flask over into the outer flask.

2. *Process.*—Weigh into the inner flask of the apparatus 10 grams, add 1.5 to 2.0 grams of paraffin free from volatile matter, and connect with the condenser. Add 10 cc. of concentrated sulphuric acid, drop by drop, through the funnel tube at such a rate as to complete the addition in two or three minutes, mix thoroughly by gentle agitation, and allow to stand five or ten minutes after all apparent action of the sulphuric acid has stopped. Measure 150 cc. of distilled water into the outer flask, heat the water slowly to boiling, and continue the boiling until 100 cc. of distillate have been collected, the rate of distillation being such as to yield this amount in twenty-five to thirty minutes.

Filter the distillate into a separatory funnel, and rinse receiver and filter with two 10-cc. portions of water. Shake with three portions of ether, using 50 cc., 30 cc., and 20 cc., and wash the combined ether extracts by shaking with four 50-cc. portions of water and a last portion of 25 cc., which portion should not require more than a drop of tenth-normal alkali for neutralization, indicating the complete removal of volatile acids. Transfer the ether extract to a tared, wide-mouthed flask, and distil off the ether on the water-bath as quickly as possible. At just the point where ebullition of the ether ceases, remove the flask from the bath, blow air into it to remove the last traces of ether, and dry in a desiccator over night, or until constant weight is secured.

The benzoic acid may also be determined by titration, in which case the filtration of the distillate, also the drying and weighing of the acid, may be omitted. The crystals of benzoic acid are dissolved in alcohol carefully neutralized immediately before each analysis, and the solution titrated with tenth-normal alkali.

#### SULPHUROUS ACID AND THE SULPHITES.

Free sulphurous acid in the form of sulphur fumes is extensively employed to bleach molasses, to disinfect wine casks, and to bleach and preserve dried fruits. This process is known as "sulphuring." It is



stated that the sulphur dioxide combines with the acetaldehyde of wines forming aldehyde-sulphurous acid, which is comparatively harmless. In the case of dried fruits it is believed to form compounds with the sugars.

The sulphurous acid salts most commonly employed as food preservatives are the bisulphites of sodium and calcium,  $\text{NaHSO}_3$  and  $\text{Ca}(\text{HSO}_3)_2$ . Others used to some extent are the normal sodium sulphite, and also potassium and ammonium sulphite. The sulphites are usually commercially prepared by passing sulphurous acid gas through strong solutions of the carbonates. Acid sulphites are formed by an excess of the sulphurous acid in the solution of the sulphite. The acid sulphites are distinguishable from the sulphites by their reaction with litmus paper, the former being acid, while the latter are neutral or feebly alkaline. All of these salts have a bitter, salty, and highly sulphurous taste, and possess a very pungent, irritating odor. With the exception of normal calcium sulphite, all of the above are readily soluble in water.

The sulphites are most commonly used as preservatives of fruit juices, ketchups, fruit and vegetable pulps, wines, malt liquors, and meat products. They are frequently mixed with other antiseptics, as with the salts of salicylic and benzoic acids.

**Detection and Determination of Sulphurous Acid.**—The same methods are used for the detection of sulphurous acid as for its quantitative determination, except that in the former case weighed quantities need not be employed, and the precipitate obtained by the barium sulphite method need not be weighed. A qualitative method employing iodate-starch paper is described on page 238.

**Distillation Method.**—This method is adapted to all food products whether solid or liquid.

Place 50 to 200 grams of the material in a 500-cc. flask, add water, if necessary, and 5 cc. of a 20% solution of phosphoric acid, and distil in a current of carbonic acid into water containing a few drops of bromine, until 150 cc. have passed over. If sulphides are present, as is true of decomposed meat products and possibly other foods, the steam from the distilling-flask before entering the condenser should be passed through a flask containing 40 cc. of a 2% neutral solution of cadmium chloride\* of a 1% solution of copper sulphate.† These solutions effectually remove the hydrogen sulphide, without retaining any appreciable amount of

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\* Horne, U. S. Dept. of Agric., Bur. of Chem., Bul. 105, 1907, p. 125.

† Winton and Bailey, Jour. Amer. Chem. Soc., 29, 1907, p. 1499.



sulphurous acid. To avoid escape of sulphurous acid the condenser tube should dip below the surface of the bromine solution.

The method and apparatus may be simplified without material loss in accuracy by omitting the current of carbon dioxide, adding 10 cc. of phosphoric acid instead of 5 cc. and dropping into the distilling-flask a piece of sodium bicarbonate weighing not more than a gram, immediately before attaching the condenser.

When the distillation is finished, boil off the excess of bromine, dilute to about 250 cc., add 1 cc. of concentrated hydrochloric acid, heat to boiling, and add, drop by drop while boiling, an excess of barium chloride solution. Allow to stand overnight in a warm place, filter (preferably on a Gooch crucible with a compact mat of woolly asbestos), wash with hot water, ignite at a dull red heat, and weigh as barium sulphate.

Molstad \* distils in a current of carbon dioxide gas into 3% hydrogen peroxide and titrates the resultant sulphuric acid with N/10 sodium hydroxide.

*Direct Titration Method.*†—This method is applicable to sauternes and other white wines and to beer, but should not be used for other materials, unless found by experiment to yield accurate results.

To 25 grams of the sample, finely divided in water if solid or semi-solid, add 25 cc. of a normal solution of potassium hydroxide in a 200-cc. flask. Shake thoroughly, and set aside for at least fifteen minutes with occasional shaking; 10 cc. of sulphuric acid (1 : 3) are then added with a little starch solution, and the mixture is titrated with N/50 iodine solution, introducing the iodine solution quite rapidly, and adding it till a distinct fixed blue color is produced. One cc. of the iodine solution is the equivalent of 0.00064 gram  $\text{SO}_2$ .

#### FORMIC ACID.

Formic acid ( $\text{HCOOH}$ ) is a colorless liquid at temperatures above  $8.3^\circ \text{C}$ . It boils at  $101^\circ \text{C}$ ., has a pungent odor and strong caustic action when applied to the skin, causing great pain and ulceration. It occurs naturally in the bodies of certain ants (hence the name) and in small quantities in various vegetable and animal substances.

On a commercial scale formic acid is usually prepared by heating glycerol with oxalic acid, the glycerol ester first formed being saponified

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\* Tids. Kem. Farm., 11, 1914, p. 281.

† U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 90.



by a fresh portion of the oxalic acid and the formic acid separated by distillation.

Formerly this acid was considered to be less active as a preservative than acetic acid, but more recently it has been shown to be very powerful, a water solution containing less than 0.1% entirely preventing the growth of yeasts and certain bacteria. Recently a 60% solution has come into use as a preservative for fruit products.

**Detection of Formic Acid.**—*Bacon Method*.<sup>\*</sup>—Strongly acidify the solution (which must not contain formaldehyde) with phosphoric acid and distil about one-third of it. To the distillate add dilute sulphuric acid and magnesium filings in sufficient quantities to cause a vigorous but not a violent evolution of hydrogen. In case quite a large quantity of acid is present in the distillate it is not necessary to add any sulphuric acid. If the amount of formic acid is small (about 0.1%) continue the action for one hour; if larger quantities are present the reaction will be complete in a few minutes. Test the solution for formaldehyde by the methods given on page 881.

*Woodman and Burwell Method*.<sup>†</sup>—Distil 50 grams of the material with 20 cc. of 20% phosphoric acid, heating the liquid during the process until 200 cc. have condensed. Mix the distillate with 2 cc. of 30% acetic acid, add 20 cc. of milk of lime (100 grams CaO per liter), or sufficient to neutralize the acid, evaporate to small bulk over a free flame, then to dryness on the water-bath, and subject to dry distillation in a test-tube heating finally to redness and passing the distillate into 3 cc. of water contained in another test-tube cooled in ice water. Test the distillate for formaldehyde.

*Shannon Method*.<sup>‡</sup>—Distil in a current of steam about 1000 cc. of the solution, collecting 2500 cc. of distillate in a receiver containing 5 cc. of lead cream. (The latter is prepared by adding sodium hydroxide to a solution of lead nitrate until a faint pink color appears with phenolphthalein and washing the precipitate 8 to 10 times by decantation.) Shake and as the lead dissolves add a few cc. more of the cream until all the formic acid is combined. Evaporate to about 50 cc., filter and allow to crystallize in a desiccator. Wash the needle-like crystals of lead formate with absolute alcohol and dry on filter-paper.

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<sup>\*</sup> U. S. Dept. of Agric., Bur. of Chem., Circ. 74.

<sup>†</sup> Tech. Quart., 21, 1908, p. 1.

<sup>‡</sup> Jour. Ind. Eng. Chem., 4, 1912, p. 526.



An aqueous solution of the crystals should reduce silver nitrate, mercuric or platinum chloride solution on warming and should yield with sulphuric acid on warming in a test-tube, carbon monoxide, which burns in the tube. Distilled with concentrated phosphoric acid, the crystals yield formic acid, identified by the acid reaction, the reducing action on the metallic salts as given above, and the formation of formaldehyde when treated according to the Bacon test.

**Determination of Formic Acid.**—*Fincke Method*.<sup>\*</sup>—Dilute 25 to 50 grams of the material to 100 cc., add 1 gram of tartaric acid and distil in a current of steam until the distillate amounts to 1000 to 1500 cc. In the case of vinegar nearly neutralize with sodium or calcium carbonate before distillation.

Add sodium hydroxide to the distillate to slight alkaline reaction, evaporate to 300 cc., add 3 to 5 grams of sodium acetate and sufficient mercuric chloride reagent (100 grams of mercuric chloride and 30 grams of sodium chloride per liter) so that the amount of mercuric chloride added is at least fifteen times the amount of formic acid present.

If the quantity of formic acid present is minute, the neutralized distillate should be evaporated to 25 cc. and only 0.2 gram of sodium acetate and 2 cc. of the mercury reagent added.

Heat on a steam bath under a reflux condenser for two hours, collect the mercurous chloride on a Gooch crucible, wash with water, and finally with alcohol and ether. Dry at 100° C. for one hour and weigh. Calculate the formic acid, using the factor 0.0975.

Both Fincke and Kreis † call attention to the formation of formic acid from sugars in the presence of acids, hence the necessity for quantitative determination, ignoring mere traces. Kreis states that by avoiding the addition of acid, heating the distilling flask in a water-bath, and collecting 1 liter of distillate, less than 5 mg. of formic acid are formed from sugars. Merl ‡ distils under diminished pressure (10 to 15 mm.), heats in a bath at 60° C. ether in a current of air or steam, the temperatures of the boiling liquid being about 35 to 43° C., treats the distillate with calcium carbonate, and proceeds in other respects as in the regular Fincke method. In this manner the formic acid from decomposed sugars does not exceed 1.49 mg.

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<sup>\*</sup> Zeits. Unters. Nahr. Genussm., 21, 1911, p. 1; 22, 1911, p. 88; 23, 1912, p. 255; 25, 1913, p. 386.

† Mitt. Lebensm. Hyg., 3, 1913, p. 205.

‡ Zeits. Unters. Nahr. Genussm., 27, 1914, p. 733.



Adam \* in all the samples of bouillon cubes examined by him found formic acid formed by the treatment of starch with nitric acid during manufacture.

If sulphurous acid is contained in the material, oxidize in an alkaline solution with hydrogen peroxide and remove the excess of peroxide with freshly precipitated mercuric oxide. In case salicylic acid is present add 1 gram of sodium chloride for each 50 cc. of the distillate.

To separate from formaldehyde or other aldehydes pass the vapor from the distilling flask through a boiling suspension of 1 gram of calcium carbonate in 100 cc. of water before condensing. Separate the suspended calcium carbonate by filtering and treat the filtrate as described. Seeker,† to avoid the interference of sulphur dioxide, uses barium carbonate instead of calcium carbonate.

*Bacon Method.*‡—Distil the solution containing the formic acid with a small quantity of phosphoric acid until the distillate is no longer acid. If the volume of the distillate is too large to be conveniently handled, neutralize it with sodium hydroxide and evaporate to a convenient volume. Add an excess of platinic chloride and sufficient acetic acid to make the solution strongly acid (usually about 1 or 2 cc. of glacial acetic acid for less than 1 gram of formic acid), and boil the solution for one hour, using a reflux condenser. Collect the reduced platinum in the usual manner and weigh. The weight of the platinum multiplied by 0.472 equals the formic acid present.

#### FLUORIDES, FLUOSILICATES, AND FLUOBORATES.

These substances all possess strong antiseptic qualities, and while no instances are recorded of the use of the last two classes of compounds in this country, the use of fluorides as a preservative of beer is practiced to some extent. The salt most commonly used is ammonium fluoride ( $\text{NH}_4\text{F}$ ), preparations of this salt being sold commercially under various trade names as beer preservatives. Ammonium fluoride exists as small, deliquescent, hexagonal, flat crystals. Its taste is strongly saline. It is soluble in water, and slightly soluble in alcohol. Sodium fluoride ( $\text{NaF}$ ) occurs as clear, lustrous crystals, soluble in water.

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\* Arch. Chem. Mikros., 9, 1916, p. 77.

† Jour. Assn. Off. Agr. Chem., 1, 1915, p. 264.

‡ U. S. Dept. of Agric., Bur. of Chem., Circ. 74.



**Detecton of Fluorides.**—*Modification of Blarez' Method.\**—Thoroughly mix the sample and heat 150 cc. to boiling. Add to the boiling liquid 5 cc. of a 10% solution of barium acetate. Collect the precipitate in a compact mass, using to advantage a centrifuge, wash upon a small filter, and dry in the oven. Transfer to a platinum crucible, first breaking up the dry precipitate and then adding the filter ash to the crucible. Prepare a glass plate (preferably of the thin variety commonly used for lantern-slide covers) as follows: First thoroughly clean and polish, and coat on one side by carefully dipping while hot in a mixture of equal parts of Canauba wax and paraffin. Near the middle of the plate make a small cross or other distinctive mark through the wax with a sharp instrument, such as a pointed piece of wood or ivory, which will remove the wax and expose the glass without scratching the latter. Add a few drops of concentrated sulphuric acid to the residue in the crucible, and cover with the waxed plate, having the mark nearly over the center, and making sure that the crucible is firmly imbedded in the wax. Place in close contact with the top or unwaxed surface of the plate a cooling device, consisting of a glass cylinder the bottom of which is closed with a thin sheet of pure rubber. Keep the cylinder filled with ice water, so that the wax does not melt. Heat the bottom of the crucible gently over a low flame or on an electric stove for an hour. Remove the glass plate and indicate the location of the distinguishing mark on the unwaxed surface of the plate by means of gummed strips of paper, melt off the wax by heat or a jet of steam, and thoroughly clean the glass with a soft cloth. A distinct etching will be apparent on the glass where it was exposed, if fluoride be present.

**Detection of Fluoborates and Fluosilicates.**—*Nivière and Hubert Method.†*—To 200 cc. of the sample add lime water to alkaline reaction, evaporate to dryness, and ignite. Extract the partially burned residue with water acidified with acetic acid and filter. Ignite the insoluble residue and extract again with dilute acetic acid, filter, add the second filtrate to the first, and test this for boric acid (page 885).

Incinerate the filter with the insoluble portion containing calcium silicate or fluoride, if present, transfer to a test-tube, mix with some silica, and add a little concentrated sulphuric acid. Attach to the test-tube a small U-tube containing a very little water. Heat the test-tube for half

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\* Mass. State Board of Health An. Rep., 1905, p. 498. Chem. News, 91, 1905, p. 39.

† Monit. sci., 1895 [4], 9, 324.



an hour in a water-bath kept below boiling. In the presence of fluoride, silicon fluoride will be generated and will be decomposed by the water in the U-tube, forming a gelatinous deposit on the walls.

If both boric and hydrofluoric acids are found, the compound present is undoubtedly a borofluoride. If no boric acid is found, but silicon fluoride is detected, repeat the operation, but without the added silica. If the silicon skeleton is then formed, fluosilicate is indicated.

#### BETA-NAPHTHOL

Beta-naphthol ( $C_{10}H_7OH$ ) is a phenol, occurring naturally in coal-tar, but the commercial product is more commonly prepared artificially from naphthalene by digesting the latter with sulphuric acid, and fusing the product with alkali. It is a colorless, or pale buff-colored powder, with a faint phenolic odor and a sharp taste. It is slightly soluble in water, and readily soluble in alcohol, ether, and chloroform. Its melting-point is  $122^{\circ}C$ . In alcoholic solution it is neutral to litmus.

It is used to some extent in alcoholic solution as a preservative of cider.

**Detection of Beta-Naphthol.**—*Bube* \* states that if an ethereal extract of beta-naphthol is evaporated to dryness, and the residue dissolved in hot water made first faintly alkaline with ammonia, and then faintly acid with very dilute nitric acid, a beautiful rose color will be developed on the addition of a drop of fuming nitric acid or of a nitrite. He declares the test to be a delicate one, but it is apparently sometimes obscured by interfering substances, which the ether may dissolve. It should also be carried out in a faint light, as strong sunlight affects the color.

Ferric chloride, when applied to an aqueous solution of beta-naphthol, produces a greenish coloration.

Shake about 50 grams of the sample to be tested with chloroform in a separatory funnel, evaporate the chloroform extract to a small volume (say 1 or 2 cc.), transfer to a test-tube, add 5 cc. of an aqueous solution of potassium hydroxide (1 : 4), and warm gently. If beta-naphthol is present, a deep-blue color will appear in the aqueous layer, turning through green to light brown.

#### ASAPROL, OR ABRASTOL

These are trade names for calcium  $\alpha$ -mono-sulphonate of beta-naphthol,  $Ca(C_{10}H_6SO_3OH)_2$ , a white, odorless, scaly powder, sometimes

\* Analyst, 13, 1888, p. 52.



slightly reddish, obtained by the action of heated sulphuric acid on beta-naphthol, the resulting compound being afterwards treated with a calcium salt. It is readily soluble in water and alcohol, and is neutral in reaction. Its taste is at first slightly bitter, but rapidly changes to sweet. It decomposes at about 50° C.

\* The writer is unaware of any instance of the presence of this substance in foods, but its character is such as to adapt it for use as a preservative of wines and possibly other food products. It has long been regarded as a possible preservative, and the analyst should be prepared to encounter it at any time.

**Detection of Asaprol.**—*Sinibaldi's Method*. \*—The portion of the solution to be tested (say 50 cc.) is made slightly alkaline with ammonia, and shaken with 10 cc. of amyl alcohol in a separatory funnel. Alcohol is often useful in breaking up an emulsion if there is one. Separate the amyl alcohol extract, which if turbid is filtered, and evaporate to dryness. Wet the residue with about 2 cc. of nitric acid (1 : 1), heat on the water-bath till the volume is about 1 cc., and wash with a few drops of water into a narrow test-tube. Next add about 0.2 gram of ferrous sulphate and ammonia in excess, a drop at a time, constantly shaking the solution. If a reddish-colored precipitate is formed, it is dissolved by the addition of a little sulphuric acid, and further additions of ferrous sulphate and ammonia are made as before. When a dark-colored or green precipitate appears, add 5 cc. of alcohol, dissolve in sulphuric acid, shake, and filter. If abraistol be present to the extent of 0.01 gram or more, a red coloration is observed, while in its absence, the filtrate is colorless or faintly yellow.

If the solution to be tested is a fat, it should be melted and extracted with hot 20% alcohol, which is evaporated to dryness, and the above test carried out on the dry residue.

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\* Mon. Sci., 1703 (4), 7, p. 842; U. S. Dept. of Agric., Bur. of Chem., Bul. 59, p. 91.



## CHAPTER XIX.

### ARTIFICIAL SWEETENERS.

UNDER this head are included the intensely sweet coal-tar derivatives, such as saccharin, dulcin, and glucin, that possess no food value whatever in themselves. From their high sweetening power, in some cases several hundred times that of cane sugar, they are capable, when used in minute quantity, of imparting an appropriate degree of sweetness to food products, which, on account of the use of inferior materials, or by reason of the presence of inert or less sweet adulterants, would otherwise be lacking in this property.

Such canned vegetables as sweet corn and peas are subject to treatment with saccharin, especially if by their age and condition before canning they are wanting in the sweet, succulent taste inherent in the fresh product.

The sweetening power of commercial glucose is considerably less than that of cane sugar, so that when large admixtures of the glucose are used in such products as jellies, jams, honey, molasses, maple syrup, etc., to the exclusion of cane sugar, the presence of the glucose might in some cases be suggested by the bland taste of the food, unless reinforced by one of the artificial sweeteners.

The analyst should therefore be on the outlook for one or another of these concentrated sweetening agents in all of the above classes of foods, especially in saccharine products wherein glucose is found to predominate largely over the cane sugar, while the taste is not lacking in sweetness.

### SACCHARIN.

*Saccharin* or *Gluside*, Benzoyl sulphimide ( $C_6H_4.CO.SO_2NH$ ), is a white powder, composed of irregular crystals, whose melting-point, when pure, is about  $224^{\circ} C$ . It is prepared from toluene, which by treatment with concentrated sulphuric acid is first converted into a mixture of



ortho- and para-toluene sulphonic acids. These are further converted into corresponding chlorides, and from the orthochloride, by treatment with ammonia, the imide is formed. It is soluble in 230 parts of cold water, 30 parts of alcohol, and 3 parts of ether. It is sparingly soluble in chloroform, but readily soluble in dilute ammonia. It is from 300 to 500 times as sweet as cane sugar, and, unlike cane sugar, it is not, when pure, charred by the action of concentrated sulphuric acid even on heating. Its aqueous solution is distinctly acid in reaction. Pure saccharin, when heated under diminished pressure, can be sublimed without decomposition.

The addition of 1 part of saccharin to 1000 parts of commercial glucose renders the latter as sweet as cane sugar.

The sodium salt of saccharin is readily soluble in water, and has nearly the same sweetening power as saccharin.

Saccharin, according to Fahlberg and List,\* has antiseptic properties. Squibb states that it is about equal to boric acid in this respect.

The use of saccharin in foods, other than those designed for invalids, is not allowed under the federal law.† This decision was reached after the Referee Board found that quantities over 0.3 gram and especially over 1 gram per day used for a considerable time were liable to produce digestive disturbances.‡

**Detection of Saccharin in Foods.**—If the sample to be tested is a solution or syrup, render it acid, if not already such, with phosphoric acid, and extract with ether. In case of canned vegetables and similar goods, finely divide the material by pulping or maceration in a mortar, dilute with water, and strain through muslin. Acidify the filtrate, and extract with ether.§ If an emulsion forms, use a centrifugal machine (p. 21). Separate the extract, evaporate off the ether, and test the residue for saccharin as follows:

(1) Add to the residue, if it tastes sweet, a few cubic centimeters of hot water, or preferably a very dilute solution of sodium carbonate, in which saccharin is more soluble. An intensely sweet taste is indicative of its presence. This test, if applied directly, will sometimes fail, especially in the case of beer, by reason of the extraction by the ether of various

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\* Jour. Soc. Chem. Ind., 4, p. 608.

† Food Inspection Decision 146.

‡ U. S. Dept. of Agric., Rep. 94, Washington, 1911.

§ Allen states that a purer residue is obtained if the sample of beer be treated with lead acetate, and filtered before extraction with ether.



bitter principles, such as hop resins, which by their strong, bitter taste mask the sweet taste of saccharin in the residue. Spaeth \* recommends that such bitter substances be removed before extraction, which is done by treatment of 500 cc. of the beer with a few crystals of copper nitrate, or with a solution of copper sulphate. The flocculent precipitate formed need not be filtered off, but the liquid is preferably concentrated by evaporation to syrupy consistency, acidified with phosphoric acid, and extracted with three successive portions of a mixture of ether and petroleum ether. After extraction, separation, and evaporation of the solvent, dissolve the residue in weak sodium carbonate. As small a quantity as 0.001% of saccharin can be detected in the final alkaline solution by its sweet taste.

(2) *Bornstein's Test.*†—Heat the residue from the ether extraction of the acidified sample with resorcin and a few drops of sulphuric acid in a test-tube till it begins to swell up. Remove from the flame, and, after cooling till the action quiets down, again heat, repeating the heating and cooling several times. Finally cool, dilute with water, and neutralize with sodium hydroxide. A red-green fluorescence indicates saccharin. Gantner ‡ states that it is useless to apply this test to beer, in view of the fact that ordinary hop resin gives the same fluorescence.

(3) *Schmidt's Test.*§—The residue is heated in a porcelain dish with about a gram of sodium hydroxide || for half an hour at a temperature of 250° C., either in an air-oven or in a linseed oil bath. This converts the saccharin if present into sodium salicylate. Dissolve the fused mass in water, acidify, and extract the solution with ether. Test the ether residue in the regular manner for salicylic acid with ferric chloride (p. 888). This test can obviously be applied only in the absence of salicylic acid, which should first be directly tested for.

It is recommended that a mixture of equal parts of ether and petroleum-ether is preferable to the use of ether alone as a solvent of saccharin, as such a mixture, while readily dissolving saccharin, does not, like ether, dissolve other substances, which might form salicylic acid when fused with sodium hydroxide.

**Determination of Saccharin.**—When saccharin is fused with an alkali and potassium nitrate, the sulphur is oxidized to sulphuric acid. On

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\* Zeits. angewandte Chem., 1893, p. 579.

† Zeits. anal. Chem., 27, p. 165.

‡ Ibid., 32, 309.

§ Rep. Anal. Chem., 30; Abs. Analyst, 12, p. 200.

|| Potassium hydroxide cannot be used instead of sodium hydroxide for the fusion.



this principle depends the following method of Reischauer: \* A known quantity of the beer or other liquid to be tested is concentrated by evaporation to about one-third its original volume, acidified with phosphoric acid, and extracted by repeated portions of ether. The combined ether extract is evaporated to small volume, and transferred to a platinum crucible, in which it is further brought to dryness. It is then cautiously ignited with a mixture of about 6 parts sodium carbonate and 1 part potassium nitrate. Dissolve the fusion in water, acidulate with hydrochloric acid, and determine the sulphuric acid in the usual manner with barium chloride. The weight of the precipitated barium sulphate, multiplied by 0.785, gives the weight of saccharin. In view of the fact that only small quantities of saccharin are used in beer and other foods, it is best to employ a large portion of the sample for analysis.

#### DULCIN.

Dulcin or sucrol, para-phenetol carbamide ( $C_2H_5O.C_6H_4.NH.CO.NH_2$ ) is a white powder, composed of needle-like crystals, sparingly soluble in cold water, ether, petroleum ether, and chloroform. It dissolves in 800 parts of cold water, 50 parts of boiling water, and 25 parts of 95% alcohol. It is readily soluble in acetic ether. Its melting-point is about  $173^{\circ}C$ . It is not readily sublimed without decomposition. Dulcin is about four hundred times sweeter than cane sugar.

When a mixture of dulcin and dilute sodium hydroxide is subjected to distillation, phenetidin goes over with the steam into the distillate. When this is heated with glacial acetic acid, phenacetin is formed, which may be tested for as follows: Boil with hydrochloric acid, dilute with water, cool, filter if turbid, and add a few drops of a solution of chromic acid. A deep-red color indicates phenacetin.

**Detection of Dulcin in Foods.**—In view of the comparatively slight solubility of dulcin in ether and chloroform, acetic ether is the best solvent for purposes of removing it from foods, first making it alkaline.

(1) *Bellier's Method*.†—A portion of the sample to be tested is made alkaline and extracted with acetic ether. In the case of certain products it is best to subject them to varied preliminary treatment, depending on the case in hand. With such products as thin fruit syrups, simply make alkaline and shake out with acetic ether. In the case of thick fruit syrups, confectionery, and preserves, dilute with water, add an excess of basic

\* Abst. Analyst, 11, p. 234.

† Ann. de Chim. Anal., 1900, V, pp. 333-337; Abs. Analyst, 26, p. 43.



lead acetate, remove the lead by precipitation with sodium sulphate, filter, and make the filtrate alkaline.

With wines, add 2 grams of mercuric acetate and a slight excess of ammonia, shake, and filter.

With beer, add to 200 cc. 2 or 3 grams of powdered sodium phosphotungstate, and a few drops of sulphuric acid, shake, allow to stand for a few minutes, and filter. Make the filtrate alkaline with ammonia.

Having thus obtained a clarified solution, use from 50 to 200 cc. of neutral acetic ether to say 500 cc. of the alkaline solution, and shake in a separatory funnel. Separate the extract, filter, and evaporate to dryness. If the dulcin exceeds 0.04 gram per liter, crystals will be apparent in the residue. If fats and resins are present in the residue, make repeated extractions with hot water, and evaporate to dryness. The purified residue is finally brought to dryness in a porcelain dish, and treated with 1 or 2 cc. of sulphuric acid and a few drops of a solution of formaldehyde. Let it stand for fifteen minutes, and afterwards dilute with 5 cc. of water. A turbidity or precipitate indicates dulcin.

(2) *Jorissen's Test*.<sup>\*</sup>—The residue from the acetic ether extract of an alkaline solution of the sample is treated with 2 or 3 cc. of boiling water in a test-tube, and a few drops of mercuric nitrate † are added. Heat the tube and its contents for five minutes in a boiling water-bath, withdraw, and disregarding any precipitate, add a small quantity of lead peroxide. On the subsidence of the precipitate, which quickly occurs, a fine violet color appears for a short time in the clear upper layer in presence of 0.001 gram of dulcin.

(3) *Morpurgo's Method*.<sup>‡</sup>—To the acetic ether residue, evaporated to dryness in a porcelain dish, add a few drops of phenol and concentrated sulphuric acid, and heat a few minutes on the water-bath. After cooling, transfer to a test-tube, and with the least possible mixing pour ammonia or sodium hydroxide over the surface. A blue zone at the plane of contact between the two layers indicates dulcin.

**Determination of Dulcin.**—For a quantitative determination, Bellier's method is carried out on a weighed or measured portion of the sample, as follows: In the case of alcoholic beverages first expel the alcohol by

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<sup>\*</sup> Chem. Zeit. Rep., 1896, p. 114.

<sup>†</sup> The mercuric nitrate is prepared by dissolving 2 grams of mercuric oxide in dilute nitric acid, adding sodium hydroxide solution till a slight permanent precipitate is formed, diluting to 15 cc., and decanting the clear liquid.

<sup>‡</sup> Zeits. anal. Chem., 1896, 35, p. 104; U. S. Dept. of Agric., Bur. of Chem., Bul. 65 p. 89.



evaporation, and make up to the original volume with water. Treat the various food preparations with the appropriate clarifying reagents, as in Bellier's qualitative test (p. 908), and, after filtering and making alkaline, extract twice with 50 cc. each of acetic ether. The residue is purified if necessary by extraction with hot water as above described, and the final residue is dissolved in 1 to 5 cc. of concentrated sulphuric acid. A few drops of formaldehyde are added. The solution is allowed to stand for fifteen minutes, and then diluted to ten times its volume with distilled water. After twenty-four hours, collect the precipitate on a tared filter, wash with water, dry, and weigh.

#### GLUCIN.

This comparatively new sweetening agent is the sodium salt of a mixture of the mono- and di-sulphonic acids of a substance having the composition  $C_{19}H_{16}N_4$ . In the market it appears as a light-brown powder, readily soluble in water. It is insoluble in ether and chloroform. It decomposes without melting at about  $250^{\circ}C$ . It is three hundred times sweeter than cane sugar.

A color reaction with glucin is obtained by dissolving it in dilute hydrochloric acid, cooling by immersing the test-tube in water, and to the cold solution adding a little sodium nitrite solution. Finally, to the liquid is added a few drops of an alkaline solution of beta-naphthol, and a red coloration is produced. With resorcin or salicylic acid in alkaline solution, the color will be yellow.



## CHAPTER XX.

### FLAVORING EXTRACTS AND THEIR SUBSTITUTES.

OF the three great groups of organic compounds essential for nutrition, the fats and proteins in a state of purity are almost tasteless, as is also true of starch, dextrin, and cellulose of the carbohydrate group. Only the sugars have a pronounced taste. The flavor of food products, aside from their sweetness, is largely due to minor constituents, such as organic acids, ethers, essential oils, etc., which serve chiefly to render the products acceptable to the palate, thereby contributing to their digestibility. Many culinary preparations lacking in flavor, but not in nutritive value, are commonly mixed with substances which supply this deficiency. Spices and flavoring extracts belong to the class of materials added mainly if not entirely for their zest-giving properties.

By far the most extensively used flavoring extracts are those of vanilla and lemon, and in comparison with these the sale of all other varieties is comparatively insignificant. These two favorite extracts are employed in nearly every household, and form a necessary adjunct to almost all forms of desserts, cakes, and confections, as well as to a wide variety of commercial preparations. Others of some importance are extracts of orange, almond, wintergreen, peppermint, rose, and certain spices. Imitation fruit flavors are used in cheap confectionery, ice cream, etc., and are of questionable wholesomeness.

#### VANILLA EXTRACT.

The **Vanilla Bean** is the source of pure vanilla extract, besides being used in chopped form directly as a flavoring agent. It is the fruit of the plant of the *Vanilla planifolia*, or flat-leaved vanilla. This climbing, perennial plant belongs to the orchid family, and is indigenous to Central and South America and the West Indies, but by far the highest prized beans are cultivated in Mexico. While different varieties differ in some details, the best cured beans of commerce, as a rule, are from 20 to 25 cm. in length and from 4 to 8 mm. thick, drawn out at their ends and curved



at the base. They are rich dark brown in color, of a soapy or waxy nature to the touch, deeply rifted lengthwise, and covered with fine frost-like crystals of vanillin. When cut cross-wise, the bean exudes a thick, odorless juice, containing calcium oxalate crystals.

The cross-section of the bean is ellipsoidal in shape. The thick brown walls inclose a triangular cavity, in which are the lobed placentas. Between these are papillæ, secreting a finely granular, yellow, balsam-like substance that contributes much to the flavor of the extract, and helps to give the cut bean its delicious odor.

When first gathered, the beans are yellowish green, fleshy, and without odor, developing their peculiar consistency, color, and smell by the process of fermentation or "sweating," which differs in various countries. According to the best methods the beans are sun-dried for nearly a month, being alternately pressed lightly between the folds of blankets, and exposed to the air. After the curing, they are packed in bundles.

Quicker methods of curing consist of the use of artificial heat and calcium chloride for drying, but the products so prepared are considered inferior in quality.

The Mexican vanilla beans are of the choicest grade, and command a high price, sometimes reaching fifteen dollars per pound. The Bourbon beans, grown in the Isle of Réunion, are next in grade. These beans are shorter than the Mexican and much less expensive. They resemble the Tonka bean in odor. Beans from Seychelles and Mauritius are even shorter than the Bourbon beans, and are largely exported to England. Cheaper varieties are those from South America, which do not bring half the price of the Mexican beans, and the cheapest are the Tahiti beans and so-called "vanillons," or beans of the wild vanilla (*Vanilla pompona*). These latter are used more in sachet powders and perfumes, possessing an odor not unlike heliotrope.

**Composition of the Vanilla Bean.**—The following are results of the analyses of two varieties of vanilla beans, according to König:

	A.	B.
Water.....	25.85	30.94
Nitrogen bodies.....	4.87	2.56
Fat and wax. ....	6.74	4.68
Reducing sugar. ....	7.07	9.12
Non-nitrogen substances. ....	30.50	32.90
Cellulose.....	19.60	15.27
Ash .....	4.73	4.53



**Vanillin.**—This body ( $C_8H_8O_3$ ) is the methyl ether of protocatechuic aldehyde, and often occurs on the surface of the bean in fine crystalline needles. It has a sharp but pleasant flavor, is soluble with difficulty in cold water, but readily soluble in hot water, ether, alcohol, and chloroform. Its melting-point is  $80^\circ$  to  $81^\circ$  C. and it sublimes at  $280^\circ$ . It is present in vanilla beans according to Winton and Berry\* in amounts varying from 1.20% to 3.50%. While the lowest percentage was found in the cheapest bean (Tahiti) the highest was found in a bean of medium quality (Comores). Mexican beans, the choicest on the market, contained 1.80% to 2.20%.

While vanillin may be readily extracted by alcohol and other solvents from the beans, such a product would be far too expensive to compete with the commercial synthetic vanillin, an artificial product, chemically identical with the vanillin from the bean. Synthetic vanillin was formerly made from the glucoside coniferin by oxidation with chromic acid. It is now largely obtained by oxidizing the eugenol of clove oil with alkaline potassium permanganate.

If ferric chloride be added to an aqueous solution containing vanillin, a dark-blue coloration will be produced.

Besides vanillin, the bean contains wax, fat, sugar, tannin, gum, resin, and delicate odoriferous principles not yet studied.

**Exhausted Vanilla Beans** are sometimes found on sale, which have been deprived of their vanillin by being soaked in alcohol, after which they are coated with some artificial substitute, presenting the same frosty appearance as the natural vanillin crystals. This may be accomplished by rolling the beans in benzoic acid. Benzoic acid crystals are readily distinguished from those of vanillin under the microscope.

**Preparation of Vanilla Extract.**—Vanilla extract is a dilute alcoholic tincture of the vanilla bean, sweetened by cane sugar. To be perfectly pure it should contain no other added substances, with the possible exception of glycerol, and many of the best brands are free from this. In practice it is variously prepared, but the following method of the U. S. Pharmacopœia (1890) is a typical one:

“Vanilla, cut into small pieces and bruised, 100 grams.

“Sugar, in coarse powder, 200 grams.

“Alcohol and water, each, a sufficient quantity to make 1000 cc.

“Mix alcohol and water in the proportion of 650 cc. of alcohol to

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 152, 1912, p. 146.



350 cc. of water. Macerate the vanilla in 500 cc. of this mixture for twelve hours, then drain off the liquid and set it aside. Transfer the vanilla to a mortar, beat it with the sugar into a uniform powder, then pack it in a percolator, and pour upon it the reserved liquid. When this has disappeared from the surface, gradually pour on the menstruum, and continue the percolation, until 1000 cc. of tincture are obtained."

**Composition of Authentic Extracts.**—The tables on pp. 915 and 916 gives summaries of analyses by Winton and Berry \* and Winton, Albright, and Berry † of extracts made by the U. S. P. process (1890) from different varieties, grades, and lengths of vanilla beans. As the process employed did not exhaust the beans as thoroughly as certain commercial processes involving soaking the beans for weeks or even months, the residues after preparing the U. S. P. extracts were further exhausted by soaking for five months in 60% alcohol and the extracts thus obtained analyzed with the results summarized at the bottom of the first table.

A study of the average figures for the different grades and different lengths, irrespective of variety, showed an increase of vanillin but a decrease in normal lead number and color value from the lowest to the highest grade and also from the shortest to the longest bean.

**Influence of Different Menstrua on Composition.**—Winton and his coworkers have found that the composition of the extract was not affected by omission of the sugar entirely, and also that when glycerol was substituted for sugar the only constant affected was the color value, which was somewhat increased. When 35% alcohol was substituted for the 62% alcohol of the above process the percentage of vanillin was not altered, but the normal lead number, the percentages of color in the lead filtrate and insoluble in amyl alcohol and the ash were increased while the color value of the extract itself and the acidity were decreased. In the preparation of a pure extract the use of alcohol weaker than 45% is not commercially practicable owing to difficulties in percolation.

Dean and Schlotterbeck, ‡ in preparing vanilla extract with 50% alcohol alone and with 50% alcohol containing 0.4% of potassium carbonate, obtained the following results: normal lead number 0.57 and 1.00, red 23 and 39, yellow 76 and 96, and ratio of red to yellow 1 : 3.3 and 1 : 2.5 respectively. Results using smaller amounts of alkali were intermediate. A better flavor was obtained without the addition.

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 152, 1912, p. 146.

† Jour. Ind. Eng. Chem., 7, 1915, p. 516.

‡ Ibid., 8, 1916, pp. 607, 703.



The same authors have made extensive investigations on the influence of method of preparation on the quality of the extract.

## COMPOSITION OF AUTHENTIC VANILLA AND TONKA EXTRACTS

Variety of Bean.	Number of Samples.	Length of Bean.	Vanillin.	Normal Lead Number.	Color Value.				Per Cent of Total Color in Lead Filtrate.		Ratio of Red to Yellow.		Per Cent of Total Color Insoluble in Amyl Alcohol.
					Extract (Total Color).		Lead Filtrate.*		Red.	Yellow.	Extract.	Lead Filtrate.	
					Red.	Yellow.	Red.	Yellow.					
		cm.	%	%					%	%	1 :	1 :	
Mexican.....	13												
Maximum.....		23	0.20	0.68	56	154	2.0	8.0	6	9	3.8	5.6	24.4
Minimum.....		15	0.15	0.47	19	55	1.0	4.8	4	5	2.6	4.0	19.0
Average.....		19	0.17	0.58	32	97	1.5	6.5	5	7	3.1	4.5	21.2
Bourbon.....	16												
Maximum.....		22	0.22	0.63	55	127	2.4	8.2	8	10	3.9	5.0	30.3
Minimum.....		10	0.13	0.44	22	65	1.4	5.8	4	5	2.3	2.8	21.3
Average.....		16	0.18	0.52	30	94	1.9	7.0	6	8	3.2	3.8	26.6
Seychelles.....	9												
Maximum.....		22	0.21	0.60	50	162	3.4	14.6	7	9	3.6	5.0	29.4
Minimum.....		10	0.16	0.45	22	77	1.0	5.0	4	6	2.5	4.0	22.7
Average.....		16	0.19	0.51	33	107	1.8	7.9	5	8	3.2	4.5	25.6
Madagascar.....	9												
Maximum.....		23	0.30	0.63	47	148	2.6	11.5	7	9	3.5	5.1	30.3
Minimum.....		11	0.16	0.40	25	85	1.4	6.2	4	6	2.7	3.5	23.2
Average.....		16	0.22	0.50	34	111	2.0	8.7	6	8	3.2	4.5	26.8
Comores.....	16												
Maximum.....		21	0.31	0.74	40	140	2.6	12.6	8	9	3.8	5.3	30.3
Minimum.....		10	0.12	0.40	22	70	1.4	6.0	5	6	2.8	3.4	20.4
Average.....		15	0.18	0.59	31	99	1.9	7.7	6	8	3.2	4.1	26.7
South American...	3												
Maximum.....			0.23	0.58	50	155	2.6	10.4	6	6	3.1	4.0	29.4
Minimum.....			0.19	0.49	42	117	1.8	6.8	4	6	2.5	3.5	20.0
Average.....			0.21	0.52	46	134	2.3	8.5	5	6	2.9	3.8	23.3
Ceylon.....	3												
Maximum.....		20	0.08	0.67	61	195	7.6	32.6	12	17	3.6	4.6	50.0
Minimum.....		12	0.07	0.57	40	145	1.4	6.4	4	4	3.2	4.1	22.7
Average.....		16	0.08	0.62	48	162	4.3	18.2	8	11	3.4	4.3	36.1
Java.....	3												
Maximum.....		20	0.24	0.61	45	177	3.2	13.4	7	10	3.9	4.3	35.7
Minimum.....		10	0.22	0.44	44	130	2.4	10.4	5	6	3.0	4.1	32.2
Average.....		15	0.23	0.50	44	150	2.9	11.1	6	8	3.4	4.2	34.5
Tahiti.....	2												
Maximum.....			0.11	0.50	17	50	0.6	3.5	4	8	3.0	5.8	18.8
Minimum.....			0.11	0.44	15	40	0.6	3.1	4	7	2.7	5.2	16.0
Average.....			0.11	0.47	16	45	0.6	3.3	4	8	2.9	5.5	17.4
Vanillons.....	1		0.06	0.52	42	107	1.4	6.6	3	6	2.5	4.7	22.2
Tonka Beans †.....	2												
Maximum.....				0.11	5	19	0.5	2.4	10	13	3.8	4.8	31.2
Minimum.....				0.11	5	18	0.5	2.4	10	13	3.6	4.8	30.3
Average.....				0.11	5	19	0.5	2.4	10	13	3.7	4.8	30.8
All Analyses ‡.....	71												
Maximum.....		23	0.31	0.74	56	177	3.4	14.6	8	10	3.9	5.8	35.7
Minimum.....		10	0.11	0.40	15	40	0.6	3.1	4	5	2.3	2.8	16.0
Average.....		16	0.19	0.54	32	102	1.8	7.6	6	8	3.2	4.2	25.5
All Analyses ‡.....	71												
(2d Extraction)													
Maximum.....		23	0.07	0.11	17	62	0.5	2.2	7	6	5.7	6.5	
Minimum.....		10	0.01	0.03	4	21	0.1	0.8	2	2	2.5	3.0	
Average.....		16	0.03	0.05	9	32	0.3	1.2	3	4	3.4	4.6	

\* Calculated to volume of extract.

† Coumarin: Maximum, 0.27%; minimum, 0.22%; average, 0.25%.

‡ Excluding Ceylon, Vanillons, and Tonka Beans.



## COMPOSITION OF AUTHENTIC VANILLA AND TONKA EXTRACTS

Variety of Bean.	Acidity of Extract, cc. N/10 Alkali per 100 cc.			Ash, Gram per 100 cc.			Alkalinity of Ash, cc. N/10 Acid per 100 cc.		
	Total.	Equivalent to Vanillin.	Other than Vanillin.	Total.	Water- soluble.	Water- insoluble.	Total.	Water- soluble.	Water- insoluble.
Mexican:									
Maximum.....	52	13	42	0.422	0.349	0.073	53	40	13
Minimum.....	42	10	27	0.297	0.246	0.037	36	29	7
Average.....	46	11	35	0.359	0.301	0.058	45	35	10
Bourbon:									
Maximum.....	51	14	38	0.373	0.319	0.080	47	34	18
Minimum.....	35	8	22	0.263	0.220	0.043	35	25	9
Average.....	40	11	29	0.317	0.259	0.058	40	27	13
Seychelles:									
Maximum.....	42	13	30	0.316	0.262	0.058	47	30	17
Minimum.....	35	10	22	0.251	0.213	0.038	34	24	9
Average.....	39	12	27	0.293	0.243	0.050	39	27	12
Madagascar:									
Maximum.....	47	20	35	0.326	0.271	0.060	46	53	14
Minimum.....	42	10	26	0.220	0.193	0.027	34	26	8
Average.....	45	14	31	0.284	0.239	0.045	39	28	11
Comores:									
Maximum.....	47	20	30	0.432	0.357	0.081	54	38	17
Minimum.....	34	8	14	0.229	0.182	0.037	33	22	9
Average.....	40	12	28	0.333	0.272	0.061	45	31	14
South American:									
Maximum.....	52	15	37	0.344	0.295	0.054	42	30	12
Minimum.....	44	12	32	0.305	0.261	0.044	38	26	12
Average.....	49	14	35	0.325	0.276	0.049	40	28	12
Ceylon:									
Maximum.....	49	5	45	0.443	0.386	0.060	56	44	12
Minimum.....	33	4	28	0.361	0.313	0.048	43	33	10
Average.....	39	5	34	0.409	0.354	0.055	49	38	11
Java:									
Maximum.....	52	15	37	0.349	0.299	0.050	49	38	11
Minimum.....	45	14	30	0.290	0.246	0.044	38	31	7
Average.....	48	15	33	0.311	0.265	0.046	42	34	8
Tahiti:									
Maximum.....	33	7	26	0.288	0.249	0.042	37	29	8
Minimum.....	30	7	23	0.221	0.179	0.039	30	23	7
Average.....	31	7	24	0.254	0.214	0.040	33	26	7
Vanillons.....	38	4	34	0.263	0.214	0.048	38	30	7
Tonka beans:									
Maximum.....	5	.....	5	0.132	0.122	0.010	15	12	3
Minimum.....	5	.....	5	0.163	0.156	0.007	16	14	2
Average.....	5	.....	5	0.147	0.139	0.008	15	13	2
All Analyses: *									
Maximum.....	52	20	42	0.432	0.357	0.081	54	40	18
Minimum.....	30	7	14	0.220	0.179	0.027	30	22	7
Average.....	42	12	30	0.319	0.265	0.054	42	30	12

\* Excluding Ceylon, Vanillons, and Tonka beans.



**The Tonka Bean** forms the basis of many of the cheaper so-called vanilla extracts on the market. It is the seed of the large tree, native to Guiana, known as *Dipterix* (or *Coumarouna*) *odorata*. The pods are almond-shaped, and contain a single seed, from 3 to 4 cm. long, shaped like a kidney bean, of a dark-brown color, having a thin, shiny, rough, brittle skin, and containing a two-lobed oily kernel.

*Coumarin* ( $C_9H_6O_2$ ), the active principle of the Tonka bean, is the anhydride of coumaric acid. It occurs in the crystalline state between the lobes of the seed kernel. Coumarin occurs also in many other plants. It may be extracted from the beans by treatment with alcohol. It crystallizes in slender, colorless, needles, melting at  $67^{\circ}$  C. It has a fragrant odor and burning taste. It is very slightly soluble in cold water, but readily soluble in hot water, ether, chloroform, and alcohol. One pound of cut beans yields by alcoholic extraction about 108 grains of coumarin. The latter may be synthetically prepared by heating salicylic aldehyde with sodium acetate and acetic anhydride, forming aceto-coumaric acid, which decomposes into acetic acid and coumarin.

The author has found that an aqueous solution of coumarin, unlike vanillin, forms a precipitate when iodine in potassium iodide is added in excess, the precipitate being at first brown and flocculent, afterwards, on shaking, clotting together to form a dark-green, curdy mass, leaving the liquid perfectly clear.

**U. S. Standards.**—*Vanilla extract* is the flavoring extract prepared from the vanilla bean, with or without sugar or glycerin, and contains in 100 cc. the soluble matters from not less than 10 grams of the vanilla bean.

*Vanilla bean* is the dried, cured fruit of *Vanilla planifolia* Andrews.

*Tonka extract* is the flavoring extract prepared from tonka bean, with or without sugar or glycerin, and contains not less than 0.1% by weight of coumarin extracted from the tonka bean, together with a corresponding proportion of the other soluble matters thereof.

*Tonka bean* is the seed of *Coumarouna odorata* Aublet (*Dipteryx odorata* (Aubl.) Willd.).

**The Adulteration of Vanilla Extract** consists chiefly in the use of coumarin or extract of the Tonka bean, and in the substitution of artificial vanillin, either alone or with coumarin, for the true extractives of the vanilla bean. Imitation vanilla flavors more often consist of a mixture of either tincture of Tonka or coumarin with vanillin in weak alcohol, colored with caramel, or occasionally with coal-tar colors. Or the exhausted marc from high-grade vanilla extract is macerated with hot water and extracted, the extract being reinforced with



artificial vanillin or coumarin, or both. A pure vanilla extract possesses certain peculiarities with regard to its resins and gums that distinguish it from the artificial, or indicate whether or not it has been tampered with. While it is possible to introduce artificial resinous matter in the adulterated brands with a view to deceiving the analyst, it is almost impossible to do this without detection, since different reactions are readily apparent in this case from those of the pure extracts.

Prune juice is said to be used to give body and flavor to vanilla extract. The writer has found spirit of myrcia or bay rum in a sample of alleged vanilla extract, containing also vanillin and coumarin. The adulterant in this sample was present to such an extent as to be unmistakable by reason of the odor.

*Factitious Vanilla Extracts* are ordinarily indicated (1) by the presence of coumarin, (2) by the peculiar reactions of the resinous matter, or by the entire absence of these resins, (3) by the scanty precipitate with lead acetate, and (4) by the abnormally low or high content of vanillin.

The following figures show the content of vanillin and coumarin in a few typical cheap "vanilla" extracts, selected from a large number examined by the author. All of these were entirely artificial, and ranged from 5 to 20 per cent by weight of alcohol.

	Vanillin, Per Cent.	Coumarin, Per Cent.
A.....	0.040	0.074
B.....	None	0.172
C.....	None	0.330
D.....	0.250	None
E.....	0.025	0.144

As a rule these cheap artificial preparations possess considerable body and flavor, but the latter is of a much grosser nature than the genuine vanilla extract, with the delicate and refined flavor of which they are not to be mistaken by any one at all familiar with both varieties.

Winton and Bailey\* have found as high as 2.55% of vanillin in imitation extracts. They also have detected the presence of acetanilide in amounts varying up to 0.15%. This substance at one time was extensively employed as an adulterant of vanillin, hence its presence in imitation extracts prepared from such vanillin. It is not only worthless as a flavor, but is a menace to health.

\* Conn. Agric. Exp. Sta., Rep. 1905, p. 131.



In the limits of composition for standard vanilla extract given on page 915, the range in vanilla content is from 0.11 to 0.31%.

#### METHODS OF ANALYSIS OF VANILLA EXTRACT

**Detection of Artificial Extracts.**—The presence of coumarin or Tonka tincture to any appreciable extent in vanilla extract is usually recognizable by the odor, to one skilled in examining these flavors. The odor of coumarin is more pungent and penetrating than that of vanillin, and in mixtures is apt to predominate over the milder and more delicate odor of vanillin.

Add normal acetate of lead solution to a suspected extract. The *absence* of a precipitate is conclusive evidence that it is artificial. If a precipitate is formed, much information may be gained by its character. A pure vanilla extract should yield with lead acetate a heavy precipitate, due to the various extractives. The precipitate should settle in a few minutes, leaving a clear, supernatant, partially decolorized liquid. If only a mere cloudiness is formed, this may be due to the caramel present, and in any event is suspicious.

**Examination of the Resins.**—Resin is present in vanilla beans to the extent of from 4 to 11 per cent, and the manufacturer of high-grade essences endeavors to extract as much as possible of this in his product. This he can do by the use of 50% alcohol, in which all the resin is readily soluble, or by employing less alcohol and relying on the use of alkali to dissolve it. A pure extract free from alkali should produce a precipitate, when a portion of the original sample is diluted with twice its volume of water and shaken in a test-tube.

When, moreover, the alcohol is removed from such an extract, the excess of resin is naturally precipitated.

The character of the resins extracted from the vanilla bean is so different from that of other resins as to furnish conclusive tests, worked out by Hess\* as follows: 25 to 50 cc. of the extract are de-alcoholized by heating in an evaporating-dish on the water-bath to about one-third its volume. Make up to the original volume with water, and, if no alkali has been used in the manufacture of the preparation, the resin will be in the form of a brown, flocculent precipitate. To entirely set free the resin, acidify, after cooling, with dilute hydrochloric acid, and allow to stand till all the resin has settled out, leaving a clear supernatant liquid. The resin may be quantitatively determined, if desired, by filtering, wash-

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\* Jour. Am. Chem. Soc., 21 (1899), p. 721.



ing, drying, and weighing, but in this case should stand for a long time before filtering.

The resin is collected on a filter, washed, and subjected to various tests. A piece of the filter with the attached resin is placed in a beaker, containing dilute potassium hydroxide. Pure vanilla resin dissolves to a deep-red color, and is reprecipitated on acidifying with hydrochloric acid. Dissolve another portion of the precipitate in alcohol, and divide the alcoholic solution into two portions, to one of which add a few drops of ferric chloride, and to the other hydrochloric acid. Pure vanilla resin shows no marked coloration in either case, but foreign resins nearly all give color reactions under these conditions.

**Tannin.**—Test a portion of the filtrate from the resin for tannin by the addition of a few drops of a solution of gelatin. A small quantity of tannin only should be indicated, if the extract is pure, a large excess tending to show added tannin.

**Determination of Vanillin and Coumarin.**—*Modified Hess and Prescott Method.*—This process, in its original form devised by Hess and Prescott,\* has been modified by Winton, collaborating with Silverman,† Bailey,‡ Lott,§, and Berry,|| in order to prevent loss of coumarin, detect the presence of acetanilide, and permit the determination of normal lead number in the same weighed portion. It depends on the principle that ammonia water, acting on the ether solution of vanillin and coumarin, forms with the aldehyde vanillin a compound soluble in water, but does not affect the coumarin, which remains in solution in the ether.

Weigh 50 grams of the extract directly into a tared 250-cc. beaker with marks showing volumes of 80 and 50 cc., dilute to 80 cc., and evaporate to 50 cc. in a water-bath kept at 70° C. Dilute again to 80 cc. with water and evaporate to 50 cc. Transfer to a 100-cc. flask, rinsing the beaker with hot water, add 25 cc. of standard lead acetate solution (80 grams of C. P. crystallized lead acetate, made up to one liter), make up to the mark with water, shake, and allow to stand eighteen hours at a temperature of from 37° to 40° C., in a bacteriological incubator, in a water-bath provided with a thermostat, or in any other suitable apparatus.

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\* Jour. Am. Chem. Soc., 21, 1899, p. 256.

† Ibid., 24, 1902, p. 1128.

‡ Ibid., 27, 1905, p. 719.

§ A. O. A. C. Proc. 1909, U. S. Dept. of Agric., Bur. of Chem., Bul. 132, p. 109.

|| U. S. Dept. of Agric., Bur. of Chem., Circ. 66.



Filter through a small dry filter and pipette off 50 cc. of the filtrate into a separatory funnel.

If a determination of normal lead number is desired, pipette off 10 cc. of the filtrate into a beaker, and proceed as described on page 925. In the latter case, the water used throughout the process should be boiled until free from carbon dioxide. If coloring with caramel is suspected determine the color value of the original extract and the filtrate (p. 926).

To the 50 cc. of the filtrate in the separatory funnel, add 20 cc. of ether and shake. Draw off carefully the aqueous liquid, together with any ether emulsion and then remove the clear ether solution to another separatory funnel. Repeat the shaking of the aqueous liquid with ether three times, using 15 cc. each time.

Shake the combined ether solutions four or five times with 2% ammonium hydroxide, using 10 cc. for the first shaking and 3 cc. for each subsequent shaking. In drawing off the ammoniacal solution, care should be taken not to allow any of the ether solution to pass through with it. Reserve the ammoniacal solution for the determination of vanillin.

Transfer the ether solution to a weighed dish and allow the ether to evaporate at room temperature. Dry in a sulphuric acid desiccator and weigh. If the residue is pure coumarin, it should have a melting-point of 67° C., respond to the Leach test, and be completely soluble in three or four portions of petroleum ether (boiling-point 30° to 40° C.), stirring with each portion fifteen minutes.

If a residue remains in the dish after decanting off the last portion of the petroleum ether solution, acetanilide should be looked for (p. 925).

Add to the ammoniacal solution 10% hydrochloric acid to slightly acid reaction. This should be done without delay, as the ammoniacal solution on standing grows slowly darker with a loss of vanillin. Cool, and shake out in a separatory funnel with four portions of ether, as described for the first ether extraction. Evaporate the ether solution at room temperature in a weighed dish, dry over sulphuric acid, and weigh. The residue should be pure vanillin free from any appreciable amount of color and with a melting-point of 80° C.

If the percentage of vanillin is not desired, and coumarin only is to be separated for gravimetric determination, the author has found that good results are usually obtained by simply treating the dealcoholized original sample with ammonia, extracting it with 3 or 4 portions of chloroform in a separatory funnel, and evaporating the combined chloroform extract in a tared dish at a temperature not exceeding 60° in the oven.



Many of the precautions employed in carrying out the above processes for vanillin and coumarin determination may be dispensed with if these substances are simply to be tested for qualitatively.

**Determination of Vanillin.**—*Folin and Denis Method.*\*—This method is based on the fact that vanillin (as well as other mono-, di-, and trihydric phenol compounds), when treated in an acid solution with phosphotungstic-phosphomolybdic acid, gives on addition of an excess of sodium carbonate, a beautiful deep blue color. It yields accurate results, requires but 5 cc. of the material, and is exceedingly rapid. An analyst familiar with the process can make ten or twelve determinations in an hour, whereas, working under favorable conditions, he would not be able to make the same number of determinations by the Hess and Prescott method in less than three days. For inspection purposes the latter method has the advantage that the vanillin and coumarin are obtained in crystalline form for subsequent tests; furthermore coumarin, normal lead number, and color value of the lead filtrate are determined in one weighed portion.

1. *Reagents.* (a) Standard Vanillin Solution. Dissolve 0.1 gram of pure vanillin in water and make up to 1 liter.

(b) Phosphotungstic-phosphomolybdic Acid Reagent. To 100 grams of pure sodium tungstate and 20 grams of phosphomolybdic acid (free from nitrates and ammonium salts) add 100 grams of syrupy phosphoric acid (containing 85 per cent  $H_3PO_4$ ) and 700 cc. of water. Boil over a free flame for one and one-half to two hours, cool, filter, if necessary, and make up with water to 1 liter. An equivalent amount of pure molybdic acid may be substituted for the phosphomolybdic acid.

(c) Sodium Carbonate Solution. Prepare a solution of the c.p. salt, saturated at room temperature.

(d) Lead Solution. Dissolve 50 grams each of basic and neutral lead acetate in water and make up to 1 liter.

2. *Process.* Pipette 5 cc. of the extract or substitute into a graduated 100-cc. flask, add about 75 cc. of cold tap water and 4 cc. of lead solution, make up to the mark with water and shake. Filter rapidly through a folded filter paper and pipette 5 cc. of the filtrate, corresponding to 0.25 cc. of the extract, into a 50-cc. graduated flask. Into another 50-cc. graduated flask pipette 5 cc. of the standard vanillin solution, which volume contains 0.0005 gram of vanillin. To each flask add from a pipette 5 cc. of the phosphotungstic-phosphomolybdic reagent, directing the stream against the neck

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\* Jour. Ind. Eng. Chem., 4, 1912, p. 670.



in such a manner as to wash down any adhering vanillin. Shake the flasks by a rotary motion, allow to stand for five minutes, then fill to the mark with saturated sodium carbonate solution. Thoroughly mix the contents of the flask by inverting several times and allow to stand for ten minutes in order that the precipitation of sodium phosphate may be complete. Filter rapidly through folded filters and compare the color of the deep-blue solutions, which must be clear, in the colorimeter.

In this, as in all colorimetric methods, a slight cloudiness of the solution of the unknown, by cutting off more light than the standard, gives a low reading and correspondingly high result.

Calculate the grams of vanillin per 100 cc. as follows:

$$P = \frac{0.0005R \times 100}{0.25r} = \frac{R}{5r},$$

in which  $P$  is the grams of vanillin per 100 cc.,  $R$  is the reading of the standard solution and  $r$  is the reading of the unknown solution in the colorimeter.

*Estes Method.\*—1. Alcoholic Extracts.*—To 5 cc. of the vanilla extract in a 50-cc. graduated flask, add 6 cc. of water and 1.5 cc. of acid mercuric nitrate reagent, prepared by dissolving metallic mercury in twice its weight of concentrated nitric acid (sp.gr. 1.42) and diluting with 25 times its weight of water. Make up at the same time a standard solution, using 5 cc. of 1% aqueous vanillin solution, 6 cc. of water, and 0.5 cc. of the reagent. Heat the two flasks in boiling water for twenty minutes, cool rapidly, make up to the mark, filter, and compare the intensity of the violet to violet red colors formed.

*2. Non-alcoholic Extracts.*—Proceed as above except that 1.0 cc. instead of 1.5 of acid mercuric nitrate reagent is used.

**Detection of Coumarin.**—*Leach Test.*—The residue, believed to be coumarin, obtained by the Hess and Prescott method, is identified by the following test: Add a few drops of water, warm gently, and add to the solution a little iodine in potassium iodide. In presence of coumarin a brown precipitate will form, which, on stirring with the rod, will soon gather in dark-green flecks. The reaction is especially marked if done on a white plate or tile.

*Wichmann Test.†*—Dilute 25 cc. of the extract with 25 cc. of water, slightly acidify, if alkaline, with sulphuric acid, and distil to dryness. To

\* Jour. Ind. Eng. Chem., 9, 1917, p. 142.

† U. S. Dept. of Agric., Bur. of Chem., Bul. 95, 1912.



the distillate, containing the vanillin and coumarin, add 15 to 20 drops of 1 : 1 potassium hydroxide, hastily evaporate to 5 cc., transfer to a test-tube and heat over a free flame until the water completely evaporates and the residue fuses to a colorless, or nearly colorless mass. Cool the melt and dissolve in a few cubic centimeters of water, transfer to a 50-cc. Erlenmeyer flask and acidify slightly with 25% sulphuric acid. Finally distil the solution (which should not exceed 10 cc.) into a test-tube containing 4 or 5 drops of neutral 0.5% ferric chloride. If coumarin is present in the original extract, a purple color will develop, the intensity being proportional to the amount of coumarin.

*The Dean Modification* \* eliminates saccharin and salicylic acid as interfering substances in the foregoing test. Dealcoholize 25 cc. of the sample or use the residue from the alcohol determination, add 5 cc. of ammonia water, and shake with 15 cc. of ether in which vanillin, salicylic acid, and saccharin are insoluble in the presence of ammonia, while coumarin is readily soluble. Separate the ether layer, evaporate to dryness on a water-bath, add 5 drops of 50% potassium hydroxide solution, dry carefully, fuse at the lowest possible temperature taking care to avoid blackening. Dissolve the mass in a few cc. of water, acidify with dilute sulphuric acid, and shake vigorously in a test-tube with 5 cc. of chloroform. Remove the chloroform with a small pipette, filter through a small plug of cotton, add 1 to 2 cc. of water containing 1 to 2 drops ferric chloride solution, and shake, noting whether or not a purple coloration is formed.

**Vanillin and Coumarin Crystals under the Microscope.**—These substances are best examined when crystallized from ether solution, and several crystallizations may be found necessary, before the best results are obtained. For examination, pour a few drops of the ether solution of the purified vanillin or coumarin directly on a slide, and allow to evaporate spontaneously. Under best conditions vanillin crystallizes from ether in long, slender needles, often radiating from central points, or forming star-shaped bundles.

Coumarin crystals are shorter and thicker than vanillin.

With polarized light pure vanillin crystals give a brilliant play of colors between crossed nicols, even without the selenite plate, while pure coumarin crystals without the selenite are almost lacking in varying colors, and show very little play, even when the selenite is employed. This sharp distinction is not true when crystallized from chloroform.

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\* Jour. Ind. Eng. Chem., 7, 1915, p. 519.



**Determination of Normal Lead Number.**—*Winton and Lott Method.\**—

Mix the 10-cc. aliquot of the filtrate from the lead acetate precipitate, obtained in the determination of vanillin and coumarin (p. 921), with 25 cc. of water, boiled until free from carbon dioxide, and a moderate excess of sulphuric acid. Add 100 cc. of 95% alcohol, and mix again. Let stand overnight, filter on a Gooch crucible, wash with 95% alcohol, dry at a moderate heat, ignite at low redness for three minutes, taking care to avoid the reducing flame, and weigh. The normal lead number is calculated by the following formula:

$$P = \frac{100 \times 0.6831 (S - W)}{5} = 13.662 (S - W),$$

in which  $P$  = normal lead number,  $S$  = grams of lead sulphate corresponding to 2.5 cc. of the standard lead acetate solution as determined in blank analyses, and  $W$  = grams of lead sulphate obtained in 10 cc. of the filtrate from the lead acetate precipitate, as above described.

The standard of the lead acetate solution as determined by blank analyses does not change appreciably on standing; it should, however, be checked from time to time, especially if the bottle is opened frequently, thus permitting absorption of carbon dioxide. In all steps of the process only water free from carbon dioxide should be used.

Pure vanilla extract of standard strength should have a normal lead number not less than 0.40. Dilution diminishes the number proportionately. For example, a mixture containing 50% of vanilla extract should have a normal lead number not less than 0.20 and so on.

**Determination of Acetanilide.**—*Winton and Bailey Method.*—If in the determination of vanillin and coumarin (p. 921) a residue is found after thoroughly stirring the coumarin with three or four 15-cc. portions of petroleum ether and decanting off the liquid; allow this residue to stand at room temperature until apparently dry and finish drying in a sulphuric acid desiccator. Weigh and deduct the weight from that previously obtained, thus obtaining the true amount of coumarin.

The residue, if acetanilide, should melt at 112° C. and respond to Ritsert's tests as given below.

If acetanilide is found in the coumarin it will also be present in the vanillin, although in smaller amount. Dissolve the weighed residue of impure vanillin in 15 cc. of 10% ammonium hydroxide solution, shake twice with ether, evaporate the ether solution at room temperature, dry

\* U. S. Dept. of Agric., Bur. of Chem., Bul. 132, 1910, p. 109; Circ. 66.



in a sulphuric acid desiccator, and weigh. Deduct this weight from the weight of impure vanillin, thus correcting for the amount of acetanilide present.

The total weight of acetanilide is found by adding the weight of the portion separated from the coumarin to that separated from the vanillin.

**Ritsert's Tests for Acetanilide.\***—Boil the acetanilide, obtained as described above, in a small beaker for two or three minutes with 2 to 3 cc. of concentrated hydrochloric acid, cool, divide into three portions, and test in small tubes (4 to 5 mm. inside diameter), or by spotting on a porcelain plate, as follows:

(1) To one portion add carefully 1 to 3 drops of a solution of chlorinated lime (1 : 200) in such a manner that the two solutions do not mix. A beautiful blue color formed at the juncture of the two liquids indicates acetanilide.

(2) To another portion add a small drop of potassium permanganate solution. A clear green color is formed if any appreciable amount of acetanilide is present.

(3) Mix the third portion with a small drop of 3% chromic acid solution. Acetanilide gives a yellow-green solution, changing to dark green on standing five minutes, and forming a dark blue precipitate on addition of a drop of caustic potash solution.

These tests are conclusive only when taken in conjunction with the melting-point.

**Determination of Glycerol.**—The presence of any considerable quantity of glycerol is apparent by the character of the residue obtained on evaporating 5 grams to dryness, in the determination of total solids. The residue, if glycerol is present in notable amount, appears of a moist consistency, even when a practically constant weight has been attained at 100° C.

To determine glycerol, proceed as with wines (page 734).

**Determination of Alcohol.**—Measure out 25 cc. of the sample, dilute to 50 cc. with water, and distil off about 20 cc. into a 25-cc. graduated receiver. Make up to the mark with water, determine the specific gravity at 15.6°, and find from the alcohol table the per cent corresponding.

**Cane Sugar and Glucose** are determined as in the case of preserves and jellies.

**Detection of Caramel.**—*Lead Acetate Method.*—Dealcoholize, precipitate with lead acetate, and filter, as described for the determination of vanillin and coumarin (page 920). If the extract is pure, the filtrate will

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\* Pharm. Ztg., 33, 1888, p. 383.



be light yellow; if colored with caramel, the filtrate will be yellow brown or deep brown, according to the amount present.

More definite conclusions may be reached by determining the color values of the original extract and the lead acetate filtrate in terms of yellow and red of the Lovibond scale and calculating the ratio of the two colors, also the percentage of each color remaining in the filtrate. The reading of the extract is made in the 1-inch cell after diluting 2 cc. to 50 cc. with 50% alcohol, while that of the filtrate is made directly in a 1-inch cell or, if very dark in  $\frac{1}{2}$ - or  $\frac{1}{4}$ -inch cell.

*Color Insoluble in Amyl Alcohol.*—Evaporate 25 cc. of the extract on a water-bath until no odor of alcohol is apparent and the liquid is reduced to a thick sirup, then proceed as described on page 785.

**Determination of Acidity.**—*Total.*—Dilute 10 cc. of the extract to 200 cc. and titrate with N/10 alkali, using phenolphthalein as indicator. Calculate to 100 cc. of extract.

*Vanillin Acidity.*—Multiply the percentage of vanillin by 65.8.

**Determination of Ash.**—*Total.*—Evaporate 10 cc. of the extract in a platinum dish and burn below redness.

*Solubility and Alkalinity of Ash.*—See page 657.

**Coal-tar Colors** are detected by the usual tests (pages 840 to 875).

### LEMON EXTRACT.

Spirit or essence of lemon of the National Formulary and former editions of the Pharmacopœia, is a 5% solution (by volume) of lemon oil in deodorized alcohol, colored with lemon peel.

This preparation was dropped from the eighth revision of the Pharmacopœia, and *Tinctura limonis corticis* or tincture of lemon peel added. The following are the directions for the preparation of the latter as given in the ninth revision:

Lemon peel, grated from the fresh fruit .....	500 grams
To make .....	1000 mls

Prepare a tincture by type process *M*, macerating the drug in 1000 mls of alcohol and completing the preparation with alcohol. Use purified cotton as a filtering medium.

**U. S. Standards.**—*Lemon Extract* is the flavoring extract prepared from oil of lemon, or from lemon peel, or both, and contains not less than 5% by volume of oil of lemon.



*Oil of Lemon* is the volatile oil obtained, by expression or alcoholic solution, from the fresh peel of the lemon (*Citrus limonum* L.), has an optical rotation (25° C.) of not less than +60° in a 100-mm. tube, and contains not less than 4% by weight of citral.

*Terpeneless Extract of Lemon* is the flavoring extract prepared by shaking oil of lemon with dilute alcohol, or by dissolving terpeneless oil of lemon in dilute alcohol, and contains not less than 0.2% by weight of citral derived from oil of lemon.

*Terpeneless Oil of Lemon* is oil of lemon from which all or nearly all of the terpenes have been removed.

The U. S. standard for lemon extract (5% of lemon oil by volume) is a fair one. In fact there are commercial extracts on the market containing as high as 12%. An extract of lemon to contain 5% of lemon oil must contain at least 80% by volume of alcohol, lemon oil being insoluble in dilute alcohol. Deodorized, or purified alcohol, commonly known as cologne spirits or perfumers' alcohol, is used in the highest-grade preparations, since the odor of ordinary commercial alcohol produces a slightly deleterious effect.

**Adulteration of Lemon Extracts.**—For making a cheap extract the cost of the lemon oil is not so important an item as that of the alcohol, and as little as possible of the latter is employed, though pure oil is doubtless used. These terpeneless extracts are made by rubbing the oil in carbonate of magnesia in a mortar, stirring in slowly a little strong alcohol, and allowing the mixture to soak for some time. A varying amount of water is added a little at a time, and the whole is shaken and again allowed to stand, sometimes for a week, before filtering. Finally the extract is filtered, and the coloring matter added, consisting sometimes of turmeric tincture and sometimes of coal-tar dyes. In these cheap extracts the per cent of alcohol often runs below 40, and as little as 4.5% by volume of alcohol has been found by the author in a commercial extract. With less than 45% of alcohol by volume, no appreciable amount of oil is dissolved, only a portion of citral, though such preparations are sometimes bottled as "pure extract of lemon." Time and again manufacturers have protested to the author that the purest oil was used by them, when notified that their brand contained no oil, or when prosecuted in court, and were with difficulty convinced that the trouble with their goods was that, on account of weak alcohol employed, the lemon oil used failed to get into the final product. It is true that a certain taste or odor of the lemon is present, even in cheap varieties wherein no oil is found, due to the fact that



even dilute alcohol, when slowly percolating through the magnesia in which the oil is finely distributed, does abstract therefrom a certain amount of citral, which is, however, but a mere shadow of the substance and body possessed by a strong alcoholic solution of oil of lemon.

In many instances, where formulas appear stating the name and per cent of ingredients, these formulas are entirely deceptive and misleading, in that the statements are not borne out on analysis.

The flavor of the cheap extracts is sometimes reinforced by the addition of such substances as citral, oil of citronella, and oil of lemongrass, but minute quantities only of these pungent materials can be used, not exceeding 0.33% in the case of citral, and 0.1% in the case of the two last mentioned oils. Cane sugar and glycerin are sometimes found.

U. S. P. tincture of lemon peel owes its color to natural substances extracted by the alcohol. This color, however, readily fades on exposure to light. Other coloring matters employed are largely coal-tar dyes, and occasionally tincture of turmeric or saffron.

During 1901 practically all the brands of lemon extract sold in Massachusetts were collected and analyzed. 167 samples were examined, representing about 100 brands, and 139 samples were classed as adulterated, based on 5% lemon oil as a standard, and depending on whether or not the contents conformed to the labels on the bottles.

The typical analyses, given in tables on page 930, are selected from the tabulated results of the above examination.\*

Forty-two samples contained no lemon oil, ranging in content of alcohol from 4% to 45%.

#### METHODS OF ANALYSIS OF LEMON EXTRACT.

A. S. Mitchell was the earliest among food chemists to systematically examine lemon extract, and to him are due the methods for determining oil of lemon, as well as various other tests now adopted provisionally by the A. O. A. C.†

**Detection of Lemon Oil in Alcoholic Lemon Extract.**—If on adding a large excess of water to the extract no cloudiness occurs, the oil may

\* An. Rep. Mass. State Board of Health, 1901, p. 459; Food and Drug Reprint, p. 41.

† Jour. Am. Chem. Soc., 21, 1899, p. 1132; U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 73; Bul. 107 (rev.), p. 159.



## LEMON EXTRACTS OF STANDARD QUALITY.

Polarization in 200-mm. Tube.	Lemon Oil, Per Cent by Volume.	Specific Gravity at 15.6° C.	Alcohol, Per Cent by Volume.	Foreign Ingredients.
30.8	9.1	0.8280	84.39	Turmeric
26.0	7.6	0.8402	80.49	
23.5	6.9	0.8352	81.74	Dinitrocresol
21.8	6.4	0.8396	82.88	
20.0	5.9	0.8335	84.24	
18.0	5.3	0.8268	86.82	
17.0	5.0	0.8496	80.06	

## INFERIOR OR ADULTERATED LEMON EXTRACTS.

Polarization in 200-mm. Tube.	Lemon Oil, Per Cent by Volume.	Specific Gravity at 15.6° C.	Alcohol, Per Cent by Volume.	Foreign Ingredients.
14.0	4.1	0.8592	77.62	Dinitrocresol
12.2	3.6	0.8644	76.08	"
11.0	3.1	0.8620	77.50	A coal-tar dye
9.9	2.9	0.8615	77.90	
8.0	2.3	0.8531	81.61	Dinitrocresol
6.8	2.0	0.8416	87.55	Tropæolin
5.0	1.5	0.8832	71.10	"
3.5	1.0	0.8939	67.68	
2.8	0.8	0.8995	65.23	Dinitrocresol
2.2	0.6	0.8941	67.69	"
1.4	0.4	0.9136	59.40	A nitro dye
0.3	0.1	0.9408	46.40	Dinitrocresol
0.0	0.0	0.9937	4.49	Tropæolin
-8.0	0.0	.....	.....	Invert sugar
27.0	0.0	.....	27.49	Cane sugar
0.0	0.0	.....	47.35	Oil other than lemon

fairly be inferred to be absent. The degree of cloudiness produced is proportional to the amount of lemon oil present.

**Determination of Lemon Oil in Alcoholic Lemon Extract.**—*Mitchell Polarization Method.*—Polarize the undiluted extract in a 200-mm. tube at 20° C. Divide the reading on the Ventzke scale by 3.4, and if cane sugar or other optically active substances are absent, the quotient expresses the per cent of lemon oil by volume. With instruments reading in circular degrees, divide the rotation in minutes at 20° C. by 62.5. If the Laurent instrument with sugar-scale is used, divide the sugar-scale reading by 4.8.

Cane sugar, though rarely found in lemon extract, is occasionally used in small amount. It is said to aid in the solution of the oil. If it is present, wash the solid residue from 10 cc. of the sample (dried on a water-bath) with three portions of 5 cc. each of ether, to remove waxy



and fatty matters, dry and weigh the residue of cane sugar, deducting 0.38 from the reading for each 0.1% of sugar so found.

*Mitchell Precipitation Method.*—Pipette 20 cc. of the extract into a Babcock milk-flask; add 1 cc. of dilute hydrochloric acid (1 : 1); add 25 to 28 cc. of water previously warmed to 60° C.; mix, and stand in water at 60° for five minutes; whirl in a centrifuge for five minutes; fill with warm water to bring the oil into the graduated neck of the flask, and repeat the whirling for two minutes; stand in water at 60° for a few minutes, and read the per cent of oil by volume. Where the oil of lemon is present in amounts over 2%, add to the percentage of oil found 0.4% to correct for the oil retained in solution. Where less than 2% and more than 1% is present, add 0.3% for correction.

Save the precipitated oil for the determination of refraction.

When the extract is made in accordance with the U. S. Pharmacopœia, the results by the two methods just given should agree within 0.2%.

To obtain per cent by weight from per cent by volume, as found by either of the above methods, multiply the volume percentage by 0.86, and divide the result by the specific gravity of the original extract.

*Howard's Modification of Mitchell's Precipitation Method.\**—Pipette 10 cc. of the extract in a Babcock milk bottle, and add in the following order, 25 cc. of cold water, 1 cc. hydrochloric acid (specific gravity 1.2), and 0.5 cc. chloroform. Close the mouth of the bottle with the thumb, and shake vigorously for not less than one minute. Whirl the bottle in a centrifuge for one and one-half to two minutes, thus forcing the chloroform and oil to the bottom of the bottle, and remove all but 3 or 4 cc. of the clear supernatant liquid by means of a glass tube of small bore connected with an aspirator.

To the residue add 1 cc. of ether, agitate thoroughly, plunge the bottle to the neck in a boiling-water bath, holding at slight angle, and rotate in the bath for exactly one minute. This step is best carried out by removing one of the small rings from a water- or steam-bath and holding the bottle in the live steam. The ether serves the purpose of steadily and rapidly sweeping out every trace of chloroform without appreciable loss of oil. Finally, cool the bottle, fill nearly to

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\* Jour. Am. Chem. Soc., 30, 1908, p. 608.



the top of the neck with water at room temperature, centrifuge for one-half minute, read the column of separated oil to the top meniscus, and multiply the reading by two, thus obtaining the per cent of oil.

This method may also be used for determining the oil in extracts of orange, peppermint, clove, cinnamon, and cassia, employing in the case of the heavier oils dilute sulphuric acid (1 : 2), instead of water, in filling the bottles before the last centrifuging.

**Determination of Lemon Oil in Non-alcoholic Lemon Extract.**—The following methods are applicable to extracts consisting of emulsions of lemon and other essential oils in mucilage of acacia, tragacanth, karaya, or other gums with or without glycerol.

*Boyles Precipitation Method.*\*—Measure 10 cc. of the emulsion into a graduated cylinder, transfer as much as possible to a 50-cc. flask, rinse the cylinder with 10-cc. portions of 95% alcohol, and with the aid of a glass rod transfer all of the emulsion and precipitated gum to the flask. Fill to the mark, shake thoroughly, and let stand about thirty minutes. Filter through a folded filter and determine the oil in a 20-cc. portion of the filtrate as in alcoholic extracts. Multiply the per cent of oil found in the filtrate by five to obtain the per cent of oil in the original emulsion. The method is applicable also to orange, almond, anise, and nutmeg extracts of the non-alcoholic type.

*Boyles Distillation Method.*\*—Measure 10 cc. of the extract into a graduated cylinder and transfer it by means of about 35 cc. of water to a side-neck distilling flask and distil with steam into a 100-cc. cassia flask. Since only 95% of the oil is recovered the amount found must be multiplied by 100 and divided by 95.

The method is also applicable to non-alcoholic orange and peppermint extracts, in the latter case the amount recovered is divided by 90 instead of 95.

**Determination of Alcohol.**—Mitchell has shown that the difference in specific gravity between oil of lemon and stronger alcohol is not so great, but that a very close approximation to the true percentage of alcohol in lemon extracts may be obtained from the specific gravity itself, assuming, of course, that foreign substances, such as sugar, glycerol, etc., are absent. In the absence of such foreign substances determine the specific gravity of the sample, ascertain from the alcohol tables on pages 690 to 703 the per cent of alcohol by volume corresponding. This gross figure

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\* Jour. Ind. Eng. Chem., 10, 1918, p. 537.



includes the lemon oil, the per cent of which should be deducted for the correct per cent of alcohol.

In the absence of oil of lemon, a measured portion of the original sample may be distilled, and the percentage of alcohol determined from the distillate in the usual manner, but when lemon oil is present, this should first be removed by diluting 50 cc. of the extract with water to 200 cc. exclusive of the oil in the sample, and shaking the mixture with 5 grams of magnesium carbonate in a flask, filtering through a dry filter, and determining the alcohol by distillation in a portion of the filtrate. The result is multiplied by four to correct for the dilution.

**Determination of Total Aldehydes.**—*Chace's Method.\**—1. *Reagents.*

—(a) *Aldehyde-free Alcohol.*—Allow alcohol (95% by vol.) containing 5 grams of metaphenylene diamine hydrochloride per liter to stand for twenty-four hours with frequent shaking. Previous treatment with potassium hydroxide is unnecessary. Boil under a reflux cooler for at least eight hours, allow to stand overnight and distil, rejecting the first 10 and the last 5 per cent which come over. Store in a dark, cool place in well-filled bottles. Twenty-five cc. of this alcohol, on standing for twenty minutes in the cooling bath with the fuchsin solution (20 cc.), should develop only a faint pink coloration. If a stronger color is developed, treat again with metaphenylene diamine hydrochloride.

(b) *Fuchsin Solution.*—Dissolve 0.5 gram of fuchsin in 250 cc. of water, add an aqueous solution of sulphur dioxide containing 16 grams of the gas, and allow to stand until colorless, then make up to 1 liter with distilled water. This solution should stand twelve hours before using, and should be discarded after three days.

(c) *Standard Citral Solution.*—Use 1 mg. of c. p. citral per cc. in 50% by volume aldehyde-free alcohol. This solution deteriorates on standing, and should not be kept over three or four days.

2. *Apparatus.*—(a) *A Cooling Bath.*—Keep at from 14 to 16° C. The aldehyde-free alcohol, fuchsin solution, and comparison tubes are to be kept in this bath.

(b) *Colorimeter.*—Any form of colorimeter, using a large volume of solution and adapted to rapid manipulation, may be used.

The comparison may also be made in Nessler or Hehner tubes.

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\* Jour. Am. Chem. Soc., 28, 1906, p. 1472. U. S. Dept. of Agric., Bur. of Chem., Bul. 122, p. 32.



3. *Manipulation*.—Weigh in a stoppered weighing flask approximately 25 grams of extract, transfer to a 50-cc. flask, and make up to the mark at room temperature with aldehyde-free alcohol. Measure at room temperature and transfer to a comparison tube 2 cc. of this solution. Add 25 cc. of the aldehyde-free alcohol (previously cooled in a bath), then 20 cc. of the fuchsin solution (also cooled), and finally make up to the 50-cc. mark with more aldehyde-free alcohol. Mix thoroughly, stopper, and place in the cooling bath for fifteen minutes. Prepare a standard for comparison at the same time and in the same manner, using 2 cc. of the standard citral solution. Remove and compare the colors developed. Calculate the amount of citral present and repeat the determination, using a quantity sufficient to give the sample approximately the strength of the standard. From this result calculate the amount of citral in the sample. If the comparisons are made in Nessler tubes, standards containing 1, 1.5, 2, 2.5, 3, 3.5, and 4 mg. should be prepared, and the trial comparison made against these, the final comparison being made with standards between 1.5 and 2.5 mg., varying but 0.25 mg.

It is absolutely essential to keep the reagents and comparison tubes at the required temperature. Comparisons should be made within one minute after removing the tubes from the bath. Where the comparisons are made in the bath (this being possible only where the bath is glass), the standards should be discarded within twenty-five minutes after adding the fuchsin solution. Give samples and standards identical treatment.

**Determination of Citral.**—*Hiltner's Method*.<sup>\*</sup>—1. *Reagents*.—(a) *Metaphenylene Diamine Hydrochloride Solution*.—Prepare a 1% solution in 50% ethyl alcohol. Decolorize by shaking with fuller's earth or animal charcoal, and filter through a double filter. The solution should be bright and clear, free from suspended matter and practically colorless. It is well to prepare only enough solution for the day's work, as it darkens on standing. The color may be removed from old solutions by shaking again with fuller's earth.

(b) *Standard Citral Solution*.—Dissolve 0.250 gram of c. p. citral in 50% ethyl alcohol and make up the solution to 250 cc.

(c) *Alcohol*.—For the analysis of lemon extracts, 90 to 95 per cent alcohol should be used, but for terpeneless extracts alcohol of 40 to 50 per cent strength is sufficient. Filter to remove any suspended mat-

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<sup>\*</sup> Jour. Ind. Eng. Chem., 1, 1909, p. 798.



ter. The alcohol need not be purified from aldehyde. If not practically colorless, render slightly alkaline with sodium hydroxide and distil.

2. *Apparatus*.—The Schreiner colorimeter (page 66) or Eggertz tubes may be used. With this latter apparatus, alcohol is added, small quantities at a time, to the stronger colored solution until after shaking and viewing transversely, the colors in the two tubes are exactly matched. Calculations are then made by establishing a proportion between the volumes of samples taken and the final dilutions.

3. *Manipulation*.—All of the operations may be carried on at room temperature. Weigh into a 50-cc. graduated flask 25 grams of the extract, and make up to the mark with alcohol (90–95 per cent). Stopper the flask and mix the contents thoroughly. Pipette into the colorimeter tube 2 cc. of this solution, add 10 cc. of metaphenylene diamine hydrochloride reagent, and complete the volume to 50 cc. (or other standard volume) with alcohol. Compare at once the color with that of the standard, which should be prepared at the same time, using 2 cc. of standard citral solution and 10 cc. of the metaphenylene diamine reagent, and making up to standard volume with alcohol. From the result of this first determination, calculate the amount of standard citral solution that should be used in order to give approximately the same citral strength of the sample under examination, then repeat the determination.

**Methyl Alcohol** has been used by unscrupulous manufacturers in lemon extracts. It is detected and determined by the refractometer method of Leach and Lythgoe (page 781).

As a confirmatory test for methyl alcohol the distillate, after testing by the Leach and Lythgoe method, may to advantage be subjected to the method of Mulliken and Scudder,\* which depends on the conversion of the methyl alcohol to formaldehyde. The latter method is also useful as a rough preliminary test on the original extract without distillation, the extract, being, however, first diluted until the liquid contains approximately 12% by weight of alcohol, shaking with magnesium carbonate, and filtering when lemon oil is present.

Oxidize 10 cc. of the liquid in a test-tube as follows: Wind copper wire 1 mm. thick upon a rod or pencil 7 to 8 mm. thick, in such a manner as to inclose the fixed end of the wire, and to form a close coil 3 to 3.5 cm. long. Twist the two ends of the wire into a stem 20 cm. long, and bend

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\* Amer. Chem. Jour., 23, 1899, p. 266.



the stem at right angles about 6 cm. from the free end, or so that the coil may be plunged to the bottom of a test-tube, preferably about 16 mm. wide and 16 cm. long. Heat the coil in the upper or oxidizing flame of a Bunsen burner to a red heat throughout. Plunge the heated coil to the bottom of the test-tube containing the diluted alcohol. Withdraw the coil after a second's time and dip it in water. Repeat the operation from three to five times, or until the film of copper oxide ceases to be reduced. Cool the liquid in the test-tube meanwhile by immersion in cold water.

*Test for Formaldehyde.*—Divide the oxidized liquid in the test-tube into two parts, testing one for formaldehyde with pure milk by the hydrochloric acid and ferric chloride test. Test the other portion by the resorcinol test for formaldehyde, page 882, avoiding an excess of the reagent.\*

**Tests for Colors.**—Evaporate a portion of the sample to dryness, dissolve the residue in water, and extract coal-tar colors if present by Arata's method, page 841, or with hydrochloric acid.

Much information may often be gained by treatment of the original extract with strong hydrochloric acid. If the color employed be turmeric, no change in color will be evident on addition of the acid. If tropæolin or methyl orange is present, the solution will turn pink, while partial decoloration of the solution indicates naphthol yellow S, and complete decoloration shows presence of dinitrocresols or naphthol yellow.

Test for turmeric by boric acid, page 821.

**Detection of Lemon and Orange Peel Coloring Matter.**—*Albrecht Method.*†—Place a few cubic centimeters of the extract in a test-tube and add slowly 3 or 4 volumes of concentrated hydrochloric acid. Place a few cubic centimeters of the extract in a second tube and add several drops of concentrated ammonia. In the presence of lemon or orange peel color the yellow tint of the original extract will be materially deepened in both cases.

**Determination of Total Solids and Ash.**—*Total Solids* are estimated by evaporating on the water-bath 10 grams of the sample in a tared dish, and drying at 100° to constant weight. If glycerol be present, it is difficult if not impossible to get a constant weight. Cane sugar and glycerol, if present, will be apparent in the residue. If capsin has been used, it will be noticed by the taste.

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\* Amer. Chem. Jour., 24, 1900, p. 451.

† U. S. Dept. of Agric., Bur. of Chem., Bul. 137, p. 71.



Burn to an ash the residue from the solids in a muffle at a low red heat, cool in a desiccator, and weigh.

**Glycerol** is determined as in wine, page 734.

**Detection of Tartaric or Citric Acid.**—To a portion of the extract in a test-tube add an equal volume of water to precipitate the oil. Filter and add one or two drops of the filtrate to a test-tube half full of cold, clear lime water. If tartaric acid is present, a precipitate will come down, which is soluble in an excess of ammonium chloride or acetic acid.

Filter off the precipitate, or, if no precipitate is visible, heat the contents of the tube. Citric acid will precipitate in a large excess of hot lime water.

**Examination of Lemon Oil.**—The oil separated from the extract in the process of determining the lemon oil by precipitation (p. 931), is most readily examined for its purity, after drying with calcium chloride, by determination of its specific gravity, its index of refraction, or its refractometric reading with the Zeiss butyro-refractometer, and its polariscopic reading.

The specific gravity and refractometric readings are determined as with fixed oils, using with the butyro-refractometer a sodium flame or yellow bichromate color-screen, which gives perfectly sharp readings without dispersion.

The table given below shows readings on the Zeiss butyro-refractometer of pure lemon oil at various temperatures, using the sodium light.

For examination of high polarizing essential oils like oil of lemon, the author employs a 50-mm. tube, in order to get the readings on the undiluted oil well within the limits of the cane sugar scale on the polariscope. If such a tube is not available, dilute the oil with an equal

READINGS ON ZEISS BUTYRO-REFRACTOMETER OF LEMON OIL.

Temperature, Centigrade.	Scale Reading.	Temperature, Centigrade.	Scale Reading.	Temperature, Centigrade.	Scale Reading.	Temperature, Centigrade.	Scale Reading.
40.0	59.4	35.0	62.8	30.0	66.3	25.0	69.7
39.5	59.7	34.5	63.1	29.5	66.6	24.5	70.0
39.0	60.1	34.0	63.5	29.0	67.0	24.0	70.4
38.5	60.4	33.5	63.8	28.5	67.3	23.5	70.7
38.0	60.8	33.0	64.2	28.0	67.7	23.0	71.1
37.5	61.0	32.5	64.5	27.5	68.0	22.5	71.4
37.0	61.5	32.0	64.9	27.0	68.4	22.0	71.8
36.5	61.8	31.5	65.1	26.5	68.7	21.5	72.1
36.0	62.1	31.0	65.6	26.0	69.0	21.0	72.5
35.5	62.4	30.5	65.9	25.5	69.3	20.5	72.8
35.0	62.8	30.0	66.3	25.0	69.7	20.0	73.2



volume of alcohol, and use the 100-mm. tube. The table given below expresses constants of pure lemon oils and of various commonly employed adulterants, as determined in the laboratory of the Massachusetts State Board of Health.

CONSTANTS OF SOME ESSENTIAL OILS.

Oil	Butyro-refractometer (Sodium Light) at—		Rotation in 100- Millimeter Tube, Ventzke Scale.	Specific Gravity at 15.6° C.
	Temp.	Reading.		
Oil of lemon (lowest) .....	25.	69.5	173.0	0.8580
“ “ “ (highest) .....	25.	71.2	184.5	0.8610
“ “ “ grass (A. Giese) .....	22.5	96.9	-10.8	0.9309
“ “ citronella (A. Giese) .....	22.5	87.1	-10.2	0.9437
Terpeneless oil of lemon (Hansel's) .....	23.	87.9	-22.0	0.9463
“ “ “ “ grass (Hansel's) ..	23.	91.0	-5.6	0.9232
Citral (A. Giese). ....	22.5	95.0	-3.6	0.9296

**Oil of Lemon** is a light-yellow liquid, having the pleasant odor of fresh lemons, and an aromatic, mild, somewhat bitter after taste. It is obtained from the grated rind of the lemon either by treatment with hot water, skimming off the oil which rises to the surface, or by pressure, or by distillation with water. It is rapidly changed by action of air and light, becoming “terpeney,” and under these conditions its solubility in alcohol seems to increase. Its composition is somewhat uncertain, but according to Wallach \* nearly 90% consists of hydrocarbons, mostly terpenes, the most important of which is the terpene limonene † of the dextro-rotate variety, also known as citrene.

Another important constituent of lemon oil is the aldehyde citral, present to the extent of from 4 to 5 per cent. To this the odor of the oil is largely due. A second aldehyde, citronellal, is also present.

A frequent adulterant of lemon oil is turpentine oil, which lowers the rotation considerably, and is thus most easily rendered apparent.

Chace ‡ detects small quantities of turpentine by the difference in crystalline form of pinene nitroso-chloride from that of limonene nitroso-chloride.

**Citral** ( $C_{10}H_{16}O$ ) is an aldehyde present in lemon oil and in oil of lemon-grass, and, while it may be separated from these oils, is prepared

\* Liebig's Annalen, 227, p. 290.

† There are two limonenes, one of which is dextro- and the other lævo-rotate. The two are completely alike in their behavior, differing only in their optical rotation.

‡ Jour. Am. Chem. Soc., 30, 1908, p. 1475.



artificially by oxidizing geraniol with chromic acid.\* It is a mobile oil, and when perfectly pure is optically inactive. The commercial citral is, however, slightly lævo-rotary, due no doubt to impurities.

**Oil of Lemon-grass** is distilled from lemon-grass, *Andropogon citratus* (D. C.), cultivated in India. It is reddish yellow in color, and has an intense lemon-like odor and taste. Very little is known of its composition, but it seems to contain several aldehydes, one of which is citronellal, and another citral. The latter, however, is its chief constituent, being present to the extent of 70 to 75 per cent.

**Citronellal** ( $C_{10}H_{18}O$ ) is an aldehyde found in various oils, especially in citronella oil, from which it is readily separated. It is made artificially by the oxidation of the primary alcohol citronellol ( $C_{10}H_{20}O$ ). It is quite strongly dextro-rotary.

**Oil of Citronella** is distilled from the grass *Andropogon nardus* (L.), growing chiefly in Ceylon, India, and tropical East Africa. It is a yellowish-brown liquid with a pleasant and lasting odor. Citronellal is present in this oil to the extent of from 10 to 20 per cent, and the oil contains also from 10 to 15 per cent of terpenes, among which are camphene.

**Tests for Citral, Citronellal, and Limonene.**†—Shake 2 cc. of the sample to be examined in a corked test-tube with 5 cc. of a solution of 10 grams of mercuric sulphate in sufficient 25% sulphuric acid to make 100 cc. Citral yields a bright-red color, which rapidly disappears, leaving a whitish compound, which floats on top. Citronellal forms a bright-yellow color, remaining for some time. Limonene forms an evanescent, faint flesh color, and leaves a white compound.

#### METHODS OF ANALYSIS OF LEMON OIL.

The following are the methods of the A.O.A.C.‡ They apply to orange as well as lemon oil.

**Determination of Specific Gravity.**—Determine the specific gravity by means of a pycnometer or a Sprengel tube at 15.6° C.

**Determination of Index of Refraction.**—Determine the index of refraction with any standard instrument, making the reading at 20° C.

**Determination of Rotation.**—Determine the rotation at 20° C. with any standard instrument using a 50-mm. tube and sodium light. The

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\* Tiemann, Berichte, 31, p. 3311.

† Burgess, Chem. and Drugg., 57, p. 732.

‡ U. S. Dept. of Agric., Bul. 137, 1911, p. 72.



results should be stated in angular degrees on a 100-mm. basis. If instruments having the sugar scale are used, the reading on orange oils is above the range of the scale, but readings may be obtained by the use of standard laevo reading quartz plates.

**Determination of Citral.—Kleber Method.**—1. *Reagents.*—(a) *Phenyl Hydrazin.*—A 10% solution of the purified chemical in absolute alcohol. A sufficiently pure product can be obtained by rectification of the commercial article, rejecting the first portions coming over which contain ammonia.

(b) *Hydrochloric Acid.*—A half normal solution.

2. *Manipulation.*—Weigh 15 grams of the sample into a small glass-stoppered flask; add 10 cc. of the phenyl hydrazin solution. After allowing to stand for half an hour at room temperature, titrate with half normal hydrochloric acid, using either methyl or ethyl orange as indicator. Titrate 10 cc. of the phenyl hydrazin reagent in the same manner. The difference in cubic centimeters of half normal acids between this titration and that of the sample, multiplied by the factor 0.076, gives the weight of citral in the sample.

If difficulty is experienced in detecting the end point of the reaction, carry out the titration until the solution is distinctly acid, transfer to a separatory funnel, and draw off the alcoholic portion. Wash the oil with water, adding the washings to the alcoholic solution, and titrate back with half normal alkali, making the necessary corrections.

*Hiltner Method.*—Proceed as under lemon extract (p. 934) weighing 2 grams of the oil, diluting to 100 cc., and using 2 cc. of this solution for the comparison.

**Determination of Total Aldehydes.**—Proceed as under lemon extract (p. 875), using from 2 to 5 grams of the sample in 100 cc. of aldehyde-free alcohol. This method should be used on orange oils the aldehydes of which are not determined by the other methods, although valuable information as to the content of added citral in the oil can be obtained by use of the Hiltner method.

**Determination of Physical Constants of the Ten Per Cent Distillate.** *Schimmel & Co. Method.*—Place 50 cc. of the sample in a 3-bulb Ladenburg flask in which the main bulb has a diameter of 6 cm. and is of 200 cc. capacity and which has the condensing bulbs of the following dimensions: 5.5 cm., 5 cm., 2.5 cm., and in which the distance from the bottom of the flask to the opening of the side arm is 20 cm. Distil the oil at the rate of 2 cc. per minute until 5 cc. have been distilled.

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\* U. S. Dept. of Agric., Bul. 137, 1911, p. 72.



Determine the refractive index and rotation of this distillate as directed above.

**Detection of Pinene.**—*Chace Method.*—Mix the 10% distillate as obtained above with 5 cc. of glacial acetic acid, cool the mixture thoroughly in a freezing bath, and add 10 cc. of ethyl nitrite; then add slowly, with constant shaking, 2 cc. of a mixture of 2 parts concentrated hydrochloric acid and 1 part water which has been previously cooled. Keep this mixture in the freezing bath during this operation and allow it to remain therein for 15 minutes. Filter off the crystals formed, using vacuum and washing with strong alcohol. Return the filtrate and washings to the freezing bath and allow them to remain for 15 minutes. Filter off the crystals formed, using the original filter-paper. Wash the two crops of crystals thoroughly with alcohol. Dry at room temperature and dissolve in the least possible amount of chloroform. Reprecipitate the nitroso-chloride crystals with methyl alcohol and mount for examination under the microscope with olive oil. Pinene nitroso-chloride crystals have irregular pyramidal ends while limonene nitroso-chloride crystallizes in needle forms.

**Determination of Alcohol.**—The amount of alcohol present in oils which have been used for the manufacture of terpeneless extracts may be approximately determined by washing repeatedly with small portions of saturated sodium chloride solution and determining the alcohol in these washings in the usual way.

#### ORANGE EXTRACT.

**Orange Oil** is a yellowish liquid, having the characteristic odor of orange, and a mild aromatic taste. It is prepared from orange peel in an analogous manner to that of lemon oil, which it somewhat resembles in chemical composition. At least 90% of orange oil, according to Walach, consists of dextro-limonene (citrene). It has a much higher specific rotatory power than lemon oil.

**U. S. Standards.**—*Oil of Orange* is the volatile oil obtained, by expression or alcoholic solution, from the fresh peel of the orange (*Citrus aurantium* L.) and has an optical rotation at 25° C. of not less than +95° in a 100-mm. tube.

*Terpeneless Oil of Orange* is oil of orange from which all or nearly all of the terpenes have been removed.

*Orange Extract* is the flavoring extract prepared from oil of orange,



or from orange peel, or both, and contains not less than 5% by volume of oil of orange.

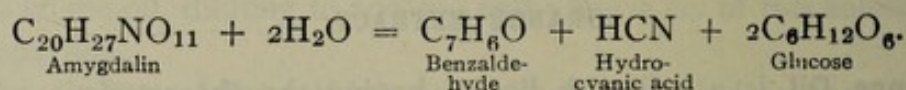
*Terpeneless Extract of Orange* is the flavoring extract prepared by shaking oil of orange with dilute alcohol, or by dissolving terpeneless oil of orange in dilute alcohol, and corresponds in flavoring strength to orange extract.

**Method of Analysis.**—Orange oil and orange extract are analyzed by the same methods as lemon oil (p. 940) and lemon extract (page 929).

In the determination of orange oil by Mitchell's polariscopic method divide the direct reading on the Ventzke scale, calculated for the 200-mm. tube, by 5.3 to obtain the per cent of orange oil by volume. To obtain the per cent by weight, multiply the per cent by volume by 0.85 and divide by the specific gravity of the extract.

#### ALMOND EXTRACT.

**Oil of Bitter Almonds** is obtained by distilling crushed bitter almonds, peach seeds, or apricot seeds with water. It should be remembered that both sweet and bitter almonds yield a bland fixed oil on pressure, which is not to be confounded with the volatile oil yielded on distillation of the bitter almonds after the fixed oil has been pressed out. Bitter almonds contain a glucoside, amygdalin, together with a ferment known as emulsin or synaptase, which, acting on the amygdalin in the distillation, produces benzaldehyde and hydrocyanic acid as follows:



The unpurified oil of bitter almonds consists largely of benzaldehyde, with a small amount of the poisonous hydrocyanic acid. Nearly all of the commercial oil is made from the cheaper apricot and peach seeds rather than those of the bitter almond, but the product is practically identical. The oil is freed from hydrocyanic acid by agitating with calcium hydrate and a solution of ferrous chloride, distilling the mixture, and drying the oil which comes over with calcium chloride.

*Benzaldehyde* constitutes 90 to 95 per cent of oil of bitter almonds, having a bitter, acrid, burning taste, and a marked almond odor. The specific gravity of the crude oil varies from 1.052 to 1.082, while that of the purified oil (benzaldehyde) at 20° is 1.0455. Its boiling-point is



180° C. On standing it becomes readily oxidizable to benzoic acid. It is readily soluble in alcohol and ether. Its solubility in water is slight, 1:300. Its index of refraction at 20° C. is 1.5446. It should be noted that the refractive indices of almond oil, whether with or without hydrocyanic acid, and of artificial benzaldehyde are nearly the same.

Benzaldehyde is produced artificially in a variety of ways, but is chiefly prepared by the action of chlorine on hot toluene. The resulting benzyl chloride is distilled with lead nitrate and water in an atmosphere of carbon dioxide, which forms benzoic aldehyde. Synthetic benzaldehyde has the same properties as the purified oil of bitter almonds, and has largely displaced it in the market, not the least of its advantages being its freedom from hydrocyanic acid.

**Almond Extract.**—Essence of bitter almonds, or *Spiritus amygdalæ amaræ*, is thus prepared according to the U. S. Pharmacopœia:

Oil of bitter almonds . . . . .	10 cc.
Alcohol . . . . .	800 cc.
Distilled water sufficient to make . . . . .	1000 cc.

Thus 1% of almond oil is present in the product.

**U. S. Standards.**—*Oil of Bitter Almonds*, commercial, is the volatile oil obtained from the seed of the bitter almond (*Amygdalus communis* L.), the apricot (*Prunus armeniaca* L.), or the peach (*Amygdalus persica* L.).

*Almond Extract* is the flavoring extract prepared from oil of bitter almonds, free from hydrocyanic acid, and contains not less than 1% by volume of oil of bitter almonds.

**Adulteration of Almond Oil.**—The official essence of the Pharmacopœia does not specify that the almond oil used be perfectly free from hydrocyanic acid, in spite of the fact that its highly poisonous nature is well known, and that it exists in the crude oil to the extent of from 4 to 6 per cent. True, but little of it is found in the extract, but in these days, when the unannounced presence in foods of such substances as antiseptics and coloring matters is regarded as questionable from a sanitary standpoint, in spite of the fact that their toxic effects on man are still matters of controversy, there should be little hesitancy in pronouncing the presence of prussic acid objectionable, especially when a pure almond oil entirely free from it is readily obtainable.

The presence of nitrobenzol or oil of mirbane as a substitute of



almond oil is to be looked for. This substance is sometimes, though incorrectly, called artificial oil of bitter almonds. It is a heavy, yellow liquid of the composition  $C_6H_5NO_2$ , readily soluble in water. Its specific gravity at  $20^\circ C.$  is 1.2039. Its boiling-point is  $205^\circ C.$  It is formed by the action of nitric acid on benzol. It possesses a highly pungent odor, somewhat like that of oil of bitter almonds, though more penetrating and less refined. Its index of refraction at  $20^\circ C.$  is 1.5517.

#### METHODS OF ANALYSIS OF ALMOND EXTRACT.

**Determination of Benzaldehyde.**—The following methods are applicable to alcoholic extracts. In the case of non-alcoholic extracts convert first into alcoholic extracts as described for lemon extract, page 932.

*Denis and Dunbar Method.\**—1. *Reagent.*—Mix 30 cc. of glacial acetic acid with 40 cc. of water, then pour in 2 cc. of phenyl hydrazine. The reagent should be made up immediately before use and discarded when more than an hour old.

2. *Method.*—Measure out two portions of 10 cc. each of the extract into 300-cc. Erlenmeyer flasks and add 10 cc. of the reagent to one flask and 15 cc. to the other. Shake, stopper tightly, and allow to stand in a dark place overnight. Add 200 cc. of distilled water and filter the precipitate of hydrazone on a tared Gooch crucible provided with a thin coat of asbestos. Wash first with cold water, finally with 10 cc. of 10% alcohol, and dry for three hours in a vacuum-oven at  $70^\circ C.$ , or to constant weight over sulphuric acid. The weight of the precipitate multiplied by the factor 5.408, will give the weight of benzaldehyde in 100 cc. of the sample. If duplicate determinations do not agree, repeat the operations, using a larger quantity of the reagent.

*Hortvet and West Method.†*—Measure 10 cc. of the extract into a 100-cc. flask, add 10 cc. of a 10% sodium hydroxide solution, and 20 cc. of a 3% hydrogen peroxide solution, cover with a watch-glass and place on a water-oven. Oxidation of the aldehyde to benzoic acid begins almost immediately and should be continued from five to ten minutes after all odor of benzaldehyde has disappeared, which usually requires from twenty to thirty minutes. If nitrobenzol be present, it will be indicated at this point by its odor. When the oxidation of the aldehyde is complete, remove the flask from the water-oven, transfer the contents

\* Jour. Ind. Eng. Chem., 1, 1909, p. 256.

† Ibid., p. 86.



to a separatory funnel, rinsing off the watch-glass, add 10 cc. of a 20% sulphuric acid solution, and cool the contents of the funnel to room temperature under the water tap. Extract the benzoic acid with three portions of 50, 30, and 20 cc. of ether, respectively, wash the combined extracts in another separatory funnel with two portions of from 25 to 30 cc. of distilled water, or until all the sulphuric acid is removed. Filter into a tared dish, wash with ether, allow to evaporate at room temperature, and finally dry over night in a desiccator, and weigh. The per cent of benzaldehyde (*B*) is obtained from the weight of the acid (*W*) by the following formula:

$$B = \frac{0.869 \times 10 \times W}{1.045}.$$

If desired the benzoic acid may be titrated, and the benzaldehyde calculated from the amount of standard alkali required for neutralization. The process is as follows: Dissolve the benzoic acid obtained as above described, except that it need not be dried in a desiccator, in 95% alcohol made neutral to phenolphthalein with tenth-normal sodium hydroxide, dilute with an equal volume of water, and titrate with tenth-normal sodium hydroxide, using phenolphthalein as indicator. The per cent of benzaldehyde (*B*) is calculated from the cc. of tenth-normal alkali (*V*) by the following formula:

$$B = \frac{V \times 0.01061 \times 10}{1.045}.$$

**Detection of Nitrobenzol.\***—Boil 15 cc. of the extract in a test-tube with a few drops of a strong solution of potassium hydroxide. Nitrobenzol produces a blood-red coloration.

**Distinction between Benzaldehyde and Nitrobenzol.**—Treat 20 cc. of the extract with 5 to 10 cc. of a cold, saturated aqueous solution of sodium bisulphite in a test-tube, and shake vigorously. Transfer to an evaporating-dish, and heat on the water-bath till the alcohol is driven off. At this stage benzaldehyde remains in the hot solution as a crystalline salt, and the solution gives off no almond odor.

Nitrobenzol, on the contrary, does not combine with the bisulphite and is insoluble, forming globules of oil on the surface of the hot liquid, and in addition giving off the pungent odor so characteristic of the substance.

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\* Holde, Jour. Soc. Chem. Ind., 13, 1893, p. 906.



**Separation of Nitrobenzol and Benzaldehyde.**—If by the qualitative test nitrobenzol is found, shake vigorously as before 50 cc. of the extract with 10 cc. of the saturated sodium bisulphite solution in a corked flask, and transfer with 100 cc. of water to a large separatory funnel. Shake out the nitrobenzol from the solution with four successive portions of petroleum ether of 15 to 20 cc. each, and after washing with water the combined petroleum ether, transfer it to a tared dish, in which it is allowed to evaporate spontaneously.

It is extremely difficult to avoid loss of some of the nitrobenzol by this process, but even if the weighed residue fails to show the full amount originally used, enough will usually be extracted to admit of testing on the refractometer, and of otherwise verifying its character.

After removal of the nitrobenzol, make the residual solution in the separatory funnel strongly alkaline with sodium hydroxide, and shake out the benzaldehyde, if present, with petroleum ether as previously described. If after making the solution alkaline no odor of benzaldehyde is apparent, the absence of benzaldehyde may be inferred.

**Distinction between Artificial Benzaldehyde and Pure Almond Oil.**—Test the final residue from the ether extract by shaking with an equal volume of concentrated sulphuric acid in a test-tube. With natural oil of almonds a clear, brilliant, but dark currant-red color is produced, while with artificial benzaldehyde, the acid produces a dirty brown color with the formation of a precipitate.

**Determination of Alcohol.**—In the absence of other flavoring substances than nitrobenzol and benzaldehyde, which are rarely present to an extent exceeding 1%, a sufficiently close approximation for most purposes can be gained by estimating the alcohol from the direct specific gravity of the extract.

**Detection of Hydrocyanic Acid.**—To a few cubic centimeters of extract in a test-tube add a few drops of a mixture of solutions of ferrous sulphate and ferric chloride, the ferrous salt being in excess. Make alkaline with sodium hydroxide, and add enough dilute hydrochloric acid to dissolve the precipitate formed by the alkali. Presence of a blue coloration or precipitate, due to the formation of Prussian blue, indicates hydrocyanic acid. The reaction is very delicate.

**Determination of Hydrocyanic Acid.\***—Hydrocyanic acid may be determined by titration with tenth-normal silver nitrate solution. 25 cc.

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\* Vielhaber, Arch. Pharm. (3), 13, p. 408.



of the extract are measured into a flask, and 5 cc. of freshly prepared magnesium hydroxide suspended in water are added, or enough to make the reaction alkaline.

A few drops of a solution of potassium chromate are then introduced, and the tenth-normal silver nitrate solution added till, with shaking, the formation of the red silver chromate indicates the end-point. 1 cc. of silver solution equals 0.0027 gram of hydrocyanic acid.

#### WINTERGREEN EXTRACT.

**Wintergreen Oil.**—True oil of wintergreen is obtained by distillation from the leaves of the wintergreen plant (*Gaultheria procumbens* L.). Gildermeister and Hoffmann\* state that the specific gravity at 15° is 1.180 to 1.187, the boiling-point 218 to 221° C. It is slightly lævoptatory ( $a_D = -0.0^\circ 25'$  to  $-1^\circ$ ).

Oil of betula or sweet birch is distilled from the bark of the black birch (*Betula lenta* L.). It has the same specific gravity and boiling-point as oil of wintergreen, but unlike the latter is optically inactive. It differs somewhat from oil of wintergreen in taste and odor, but is hardly distinguishable in these respects from synthetic methyl salicylate.

Both oil of wintergreen and oil of sweet birch consist almost entirely of methyl salicylate, the former containing, according to Power and Kleber,† as much as 99.8% of this substance.

**U. S. Standards.**—Oil of *Wintergreen* is the volatile oil distilled from the leaves of the *Gaultheria procumbens* L.

*Wintergreen Extract* is the flavoring extract prepared from oil of wintergreen, and contains not less than 3% by volume of oil of wintergreen.

*Spirit of Gaultheria* of the U. S. P. is a mixture of 50 cc. of oil of wintergreen and 950 cc. of alcohol. It accordingly contains 5% by volume of the essential oil.

**Adulteration of Wintergreen Extract.**—Synthetic methyl salicylate is very commonly substituted for both wintergreen and sweet birch oil, and sweet birch oil in turn for wintergreen oil. The production of true wintergreen oil is small, the so-called natural wintergreen oil of commerce being usually sweet birch oil. The sense of smell is the best

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\* The Volatile Oils. Translated by Kremers, Milwaukee, 1900, p. 588.

† Pharm. Rund., 13, p. 228.



means of distinguishing the two oils; polarization is of rather uncertain value, owing to low rotatory power of the wintergreen oil.

**Determination of Wintergreen Oil.**—*Hortvet and West's Method.\**—Measure 10 cc. of the extract into a 100-cc. beaker, add 10 cc. of 10% potassium hydroxide solution, and heat the mixture over a boiling water-bath until the odor of oil of wintergreen has disappeared and the liquid is reduced to about one-half its original volume. By this treatment the methyl salicylate is converted into the potassium salt. Liberate the salicylic acid by the addition of an excess of 10% hydrochloric acid, cool, and extract in a separatory funnel with three portions of 40, 30, and 20 cc. of ether respectively. Pour the combined ether extracts through a dry filter into a weighed dish, wash the filter with 10 cc. of ether, evaporate filtrate and washings slowly at 50° C., dry one hour in a desiccator, and weigh. The per cent of wintergreen oil by volume (*M*) is obtained from the weight of salicylic acid (*S*) by the following formula:

$$M = \frac{1.101 \times 10 \times S}{1.18}.$$

*Howard's Method.*—Proceed as described on page 931, except that the heavy oil is brought into the graduated portion of the Babcock bottle by addition of dilute sulphuric acid (1:2), taking care that the acid is not over 25° C. and avoiding agitation.

#### PEPPERMINT EXTRACT

**Peppermint Oil** is obtained from various plants of the genus *Mentha*, which are commonly classed as sub-species or varieties of *M. piperita*. Owing in large part to the botanical differences in the plants from which

	Specific Gravity.	Rotation, $\alpha_D$ .	Total Menthol, Per Cent.
American . . . . .	0.905 to 0.920	—18° to —33°	48 to 60
English . . . . .	0.900 to 0.910	—22° to —33°	56 to 66
Japanese . . . . .	0.895 to 0.900	—30° to —42°	70 to 91
Saxon . . . . .	0.900 to 0.915	—25° to —33°	54 to 68
German . . . . .	0.899 to 0.930	—27° to —33°	
French . . . . .	0.918 to 0.920	—5° to —9°	43 to 46
Russian . . . . .	0.905 to 0.910	—17° to —22°	50.2

\* Jour. Ind. Eng. Chem., 1, 1909, p. 90.



it is made, peppermint oil from different regions differs greatly in its chemical and physical constants as shown by the table on bottom of page 948, compiled from figures given by Gildermeister and Hoffmann.\*

**U. S. Standards.**—*Peppermint* is the leaves and flowering tops of *Mentha piperita* L.

*Oil of Peppermint* is the volatile oil obtained from peppermint, and contains not less than 50% by weight of menthol.

*Peppermint Extract* is the flavoring extract prepared from oil of peppermint, or from peppermint, or both, and contains not less than 3% by volume of oil of peppermint.

**Analysis of Peppermint Extract.**—Owing to the wide variation in the rotatory power of peppermint oil, only a roughly approximate idea of the oil content of peppermint extract can be gained by polarization. The variation in the percentage of menthol in the oil is also too great to permit of a method based on the amount of this constituent. Mitchell's precipitation method, as originally described (page 931), does not effect a complete separation of the oil, but Howard's modification (page 931) gives satisfactory results, and is well adapted for purposes of inspection.

Boyles' distillation method (page 932) may also be used.

#### SPEARMINT EXTRACT.

**U. S. Standards.**—*Spearmint* is the leaves and flowering tops of *Mentha spicata* L.

*Oil of Spearmint* is the volatile oil obtained from spearmint.

*Spearmint Extract* is the flavoring extract prepared from oil of spearmint, or from spearmint, or both, and contains not less than 3% by volume of oil of spearmint.

#### SPICE EXTRACTS.

Alcoholic solutions of the essential oils of spices are used to some extent instead of the spices themselves. The following are the definitions of these extracts and the oils from which they are prepared, as adopted by the joint committee on standards and the U. S. Secretary of Agriculture:

**U. S. Standards.**—*Anise Extract* is the flavoring extract prepared from oil of anise, and contains not less than 3% by volume of oil of anise.

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\* The Volatile Oils. Translated by Edward Kremers, Milwaukee, 1900.



*Oil of Anise* is the volatile oil obtained from the anise seed.

*Celery Seed Extract* is the flavoring extract prepared from celery seed or the oil of celery seed, or both, and contains not less than 0.3% by volume of oil of celery seed.

*Oil of Celery Seed* is the volatile oil obtained from celery seed.

*Cassia Extract* is the flavoring extract prepared from oil of cassia, and contains not less than 2% by volume of oil of cassia.

*Oil of Cassia* is the lead-free volatile oil obtained from the leaves or bark of *Cinnamomum cassia* Bl., and contains not less than 75% by weight of cinnamic aldehyde.

*Cinnamon Extract* is the flavoring extract prepared from oil of cinnamon, and contains not less than 2% by volume of oil of cinnamon.

*Oil of Cinnamon* is the lead-free volatile oil obtained from the bark of the Ceylon cinnamon (*Cinnamomum zeylanicum* Breyne), and contains not less than 65% by weight of cinnamic aldehyde and not more than 10% by weight of eugenol.

*Clove Extract* is the flavoring extract prepared from oil of cloves, and contains not less than 2% by volume of oil of cloves.

*Oil of Cloves* is the lead-free, volatile oil obtained from cloves.

*Ginger Extract* is the flavoring extract prepared from ginger, and contains in each 100 cc. the alcohol-soluble matters from not less than 20 grams of ginger.

*Nutmeg Extract* is the flavoring extract prepared from oil of nutmeg, and contains not less than 2% by volume of oil of nutmeg.

*Oil of Nutmeg* is the volatile oil obtained from nutmegs.

*Savory Extract* is the flavoring extract prepared from oil of savory, or from savory, or both, and contains not less than 0.35% by volume of oil of savory.

*Oil of Savory* is the volatile oil obtained from savory.

*Star Anise Extract* is the flavoring extract prepared from oil of star anise, and contains not less than 3% by volume of oil of star anise.

*Oil of Star Anise* is the volatile oil distilled from the fruit of the star anise (*Illicium verum* Hook).

*Sweet Basil Extract* is the flavoring extract prepared from oil of sweet basil, or from sweet basil, or both, and contains not less than 0.1% by volume of oil of sweet basil.

*Sweet Basil, Basil*, is the leaves and tops of *Ocimum basilicum* L.

*Oil of Sweet Basil* is the volatile oil obtained from basil.

*Sweet Marjoram Extract, Marjoram Extract*, is the flavoring extract



prepared from the oil of marjoram, or from marjoram, or both, and contains not less than 1% by volume of oil of marjoram.

*Oil of Marjoram* is the volatile oil obtained from marjoram.

*Thyme Extract* is the flavoring extract prepared from oil of thyme, or from thyme, or both, and contains not less than 0.2% by volume of oil of thyme.

*Oil of Thyme* is the volatile oil obtained from thyme.

**Determination of Essential Oil in Alcoholic Cinnamon, Cassia, and Clove Extracts.**—*Howard's Method.*—Proceed as with wintergreen extract, page 948.

*Hortvet and West's Method.\**—Place 10 cc. of the extract and 50 cc. of water in a separatory funnel, and extract with three portions of ether measuring respectively 50, 30, and 20 cc. Wash the combined extracts successively with 25 and 30 cc. of distilled water, and filter through a dry funnel into a wide-mouth flask, washing out the funnel and filter with a little ether. In the case of cinnamon extract, transfer the ether extract before filtering to a 150-cc. flask, shake for a few minutes with some granulated calcium chloride, then filter in the manner described. Evaporate off the ether as rapidly as possible on a boiling water-bath until only a few drops remain. At this point remove the flask from the bath, and rotate rapidly for a few minutes, spreading the residue over the sides of the flask. The rapid evaporation of the remaining ether cools the flask to near room temperature. When the odor of ether has disappeared, stopper the flask and weigh.

In the case of cassia and clove oils, where the ether extract is not first dried with calcium chloride, a slight cloudiness gathers on the flask as the last traces of ether disappear, due to the presence of a little moisture. In such case allow the flask to stand on the balance-pan until the film disappears, requiring not longer than two or three minutes, then stopper, and weigh.

The per cent of oil by volume (*V*) is calculated from the weight of oil (*W*) by the following formula:

$$V = \frac{100 \times W}{10 \times 1.050}.$$

The oil thus extracted may be used for determination of the refractive index. After dissolving in a little alcohol it may be tested with ferric chloride solution. By this test cinnamon oil gives a green, cassia oil a brown, and clove oil a deep blue, coloration.

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\* Jour. Ind. Eng. Chem., 1, 1909, p. 88.



**Determination of Essential Oil in Non-alcoholic Cinnamon, Cassia, and Clove Extracts.**—*Boyles Modification of the Howard Method.\**—Dilute 10 cc. of the sample with 95% alcohol to 50 cc., as in the case of lemon, and filter. Place 10 cc. of the filtrate in a separatory funnel containing 50 cc. of water, add 1 cc. of hydrochloric acid (1 : 1), and shake out four times with 25-cc. portions of ether. Wash the combined ether extracts twice with water and then shake for a few minutes with about 5 grams of granular calcium chloride. Place a small piece of cotton in the outlet of the separatory funnel and draw the ether into a tared beaker. Evaporate the ether on a boiling water-bath, place in a desiccator for three minutes, and weigh. Divide the weight found by the specific gravity of the oil to obtain the per cent of oil by volume.

**Determination of Essential Oil in Nutmeg Extract.**—Follow Mitchell's precipitation method (page 931). In the case of non-alcoholic nutmeg extracts convert first into an alcoholic extract as described for non-alcoholic lemon extract (page 931).

**Determination of Solids in Ginger Extract.†**—Evaporate 10 cc. on a boiling water-bath to dryness, dry for two hours in a boiling water oven and weigh.

**Determination of Alcohol in Ginger Extract.†**—Proceed as with vanilla extract (page 926).

**Detection of Ginger in Ginger Extract.†**—*Seeker Method.*—Dilute 10 cc. of the extract to 30 cc., evaporate off 20 cc., decant into a separatory funnel and extract with an equal volume of ether. Evaporate the ether spontaneously in a porcelain dish and to the residue add 5 cc. of 75% sulphuric acid and 5 mg. of vanillin. Allow to stand for fifteen minutes and add an equal volume of water. In the presence of ginger extract an azure blue color develops.

**Detection of Capsicum in Ginger Extract.**—*Nelson-La Wall-Doyle Method.‡*—To 10 cc. of the extract cautiously add dilute sodium hydroxide until the solution reacts very slightly alkaline with litmus paper. Evaporate at about 70° C. to about one-quarter of the original volume, render slightly acid with dilute sulphuric acid, testing with litmus paper. Transfer to a separatory funnel, rinsing the evaporating dish with water, and extract with an equal volume of ether, avoiding emulsification by shak-

\* Jour. Ind. Eng. Chem., 10, 1918, p. 537.

† U. S. Dept. of Agric., Bur. of Chem., Bul. 137, 1911, p. 75.

‡ Jour. Ind. Eng. Chem., 2, 1910, p. 419; U. S. Dept. of Agric., Bur. of Chem., Bul. 137, 1911, p. 75; Bul. 152, 1912, p. 137.



ing the funnel gently for a minute or two. Draw off the lower layer and wash the ether extract once with about 10 cc. of water. Transfer the washed ether extract to a small evaporating dish, render decidedly alkaline with alcoholic potassium hydroxide, and evaporate at about 70° until the residue is pasty; then add about 20 cc. more of half-normal alcoholic potash and allow to stand on a steam bath until the gingerol is completely saponified, which usually requires about one-half hour. Dissolve the residue in a little water and transfer with water to a small separatory funnel. The volume should not exceed 50 cc. Extract the alkaline solution with an equal volume of ether. Wash the ether extract repeatedly with small amounts of water until no longer alkaline to litmus. Transfer the washed extract to a small evaporating dish, allow the ether to evaporate spontaneously, and finally, test the residue for capsicum by moistening the tip of the finger, rubbing it around on the bottom and sides of the dish, and then applying the finger to the end of the tongue. A hot, stinging, or prickly sensation, which persists for several minutes, indicates capsicum or other foreign pungent substances.

#### ROSE EXTRACT.

**U. S. Standards.**—*Rose Extract* is the flavoring extract prepared from otto of roses, with or without red rose petals, and contains not less than 0.4% by volume of otto of roses.

*Otto of Roses* is the volatile oil obtained from the petals of *Rosa damascena* Mill., *R. centifolia* L., or *R. moschata* L.

**Determination of Rose Oil.**—*Hortvet and West's Method*.<sup>\*</sup>—Measure 25 cc. of the extract into a separatory funnel, add 50 cc. of water, mix thoroughly, acidify with 1 cc. of hydrochloric acid (1:1), and extract with three portions of 20 cc. each of ether. Transfer the combined ether extracts to a 150-cc. flask, shake for a few minutes with some granulated calcium chloride, allow to settle until clear, then decant through a dry filter into a flat bottom glass dish previously weighed together with a cover-glass. Wash the calcium chloride and filter twice with 10 cc. of ether, and add the washings to the glass dish. Cover the dish, place in a vacuum desiccator over sulphuric acid, allow to remain until all traces of ether and alcohol are removed, and weigh. Repeat the drying in the desiccator, for one hour periods, until the weight is practically constant. The final weight, divided by 0.86 and multiplied by 5, gives the per cent of oil of rose by volume.

<sup>\*</sup> Jour. Ind. Eng. Chem., 1, 1909, p. 89.



### IMITATION FRUIT FLAVORS.

Nearly all the fruits possess distinctive flavors, which are desirable in food preparations, and which may be made to impart their flavor to such substances as confections, ice cream, dessert mixtures, jellies, etc., by simply mixing with these foods the fresh or preserved fruit or fruit juice in sufficient quantity. In many cases, however, it is not found possible or practicable to prepare from the fruits themselves an extract sufficiently concentrated to give the distinctive fruit flavor, when used in moderate quantity, and hence the use of artificial fruit essences made up of compound ethers, mixed in varying combinations and proportions to imitate more or less closely various fruit flavors.

These ethers are usually much more pungent and penetrating than the fruits which they imitate, and, while lacking the delicacy and refinement of the original fruits, serve to impart a certain semblance of the genuine flavor in a convenient and highly concentrated form.

Some of the single compound ethers possess a remarkable resemblance to particular fruits, while to imitate other fruits a mixture of various ethers and flavoring materials, such as lemon and other volatile oils, vanilla, organic acids, chloroform, etc., is necessary. These artificial essences should in all cases be sold as such, and not as "pure fruit flavors."

**Imitation Pineapple Essence** is made up by dissolving in alcohol butyric ether,  $C_4H_7(C_2H_5)O_2$ , which possesses a distinct pineapple flavor, and is prepared by mixing 100 parts of butyric acid ( $C_4H_8O_2$ ), 100 parts of alcohol, and 50 parts of sulphuric acid, and shaking. Butyric ether is sparingly soluble in water, and boils at  $121^\circ C$ .

**Imitation Quince Essence** depends as a basis on ethyl pelargonate, sometimes called pelargonic or cœnanthic ether,  $C_2H_5C_9H_{17}O_2$ , dissolved in alcohol. Pelargonic ether is formed by digestion with the aid of heat of pelargonic acid and alcohol. Pelargonic acid,  $C_9H_{18}O_2$ , is first obtained by the action of nitric acid on oil of rue. Pelargonic ether is a colorless liquid, having a specific gravity of 0.8635 at  $17.5^\circ C$ . Its boiling-point is  $227^\circ$  to  $228^\circ C$ . It is insoluble in water.

**Imitation Jargonelle Pear Essence** consists of an alcoholic solution of amyl or pentyl acetate,  $C_5H_{11}C_2H_3O_2$ . This is prepared by distilling a mixture of one part of amyl alcohol, two parts of potassium acetate, and one part of concentrated sulphuric acid. It is a colorless liquid, insoluble in water, and having a boiling-point of  $137^\circ C$ .



**Imitation Banana Essence** is made up of a mixture of amyl acetate and butyric ether.

**Imitation Apple Essence** is composed of an alcoholic solution of amyl valerianate, sometimes called apple oil,  $C_5H_{11}, C_5H_9O_2$ , prepared by mixing four parts of amyl alcohol with four of sulphuric acid, and adding

## COMPOSITION OF IMITATION ESSENCES.

	Chloroform.	Nitrous Ether.	Aldehyde.	Acetic Ether.	Formic Ether.	Butyric Ether.	Valerianic Ether.	Benzoic Ether.	Enanthic Ether.	Oil of Peppercot.	Sebacic Ether.	Methyl-salicylic Ether.
Pineapple .....	1	.....	1	.....	.....	5	.....	.....	.....	.....	.....	.....
Melon .....	.....	.....	2	.....	1	4	5	.....	.....	.....	10	.....
Strawberry .....	.....	1	.....	5	1	5	.....	.....	.....	.....	.....	1
Raspberry .....	.....	1	1	5	1	1	.....	1	1	.....	1	1
Gooseberry .....	.....	.....	1	5	.....	.....	.....	1	1	.....	.....	.....
Grape .....	2	.....	2	.....	2	.....	.....	.....	10	.....	.....	1
Apple .....	1	1	2	1	.....	.....	.....	.....	.....	.....	.....	.....
Orange .....	2	.....	2	5	1	1	.....	1	.....	.....	.....	1
Pear .....	.....	.....	.....	5	.....	.....	.....	.....	.....	.....	.....	.....
Lemon .....	1	1	2	10	.....	.....	.....	.....	.....	.....	.....	.....
Black cherry .....	.....	.....	.....	10	.....	.....	.....	5	.....	2	.....	.....
Cherry .....	.....	.....	.....	5	.....	.....	.....	5	1	.....	.....	.....
Plum .....	.....	.....	5	5	1	2	.....	.....	.....	4	.....	.....
Apricot .....	1	.....	.....	.....	.....	10	5	.....	1	.....	.....	.....
Peach .....	.....	.....	2	5	5	5	5	.....	.....	5	1	.....
Currant .....	.....	.....	1	5	.....	.....	.....	1	1	.....	.....	.....

	Amyl Alcohol.	Amyl-acetic Ether.	Amyl-butyric Ether.	Amyl-valerianic Ether.	Oil of Lemon.	Oil of Orange.	Saturated Alcoholic Solutions of				Glycerin.
							Tartaric Acid.	Oxalic Acid.	Succinic Acid.	Benzoic Acid.	
Pineapple .....	.....	.....	10	.....	.....	.....	.....	.....	.....	.....	3
Melon .....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	3
Strawberry .....	.....	3	2	.....	.....	.....	.....	.....	.....	.....	2
Raspberry .....	.....	1	1	.....	.....	.....	5	.....	1	.....	4
Gooseberry .....	.....	.....	.....	.....	.....	.....	5	.....	1	1	.....
Grape .....	.....	.....	.....	.....	.....	.....	5	.....	3	.....	10
Apple .....	.....	.....	.....	10	.....	.....	.....	1	.....	.....	4
Orange .....	.....	1	.....	.....	.....	10	1	.....	.....	.....	10
Pear .....	.....	2	.....	.....	.....	.....	.....	.....	.....	.....	2
Lemon .....	.....	.....	.....	.....	10	.....	10	.....	1	.....	5
Black cherry .....	.....	.....	.....	.....	.....	.....	.....	1	.....	2	.....
Cherry .....	.....	.....	.....	.....	.....	.....	.....	.....	.....	1	3
Plum .....	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....	8
Apricot .....	1	.....	1	.....	.....	.....	1	.....	.....	.....	4
Peach .....	2	.....	.....	.....	.....	.....	.....	.....	.....	.....	5
Currant .....	.....	.....	.....	.....	.....	.....	5	.....	1	1	.....



the mixture when cold to five parts of valerianic acid. The specific gravity of amyl valerianate is 0.879 at 0° C. and its boiling-point is 188° C.

The table on p. 955, prepared by Kletzinsky, shows the composition of a large variety of these artificial essences. The numerals in the various columns indicate the parts by volume to be added to 100 parts of deodorized alcohol.

**Determination of Esters.**—Add to 25 grams of the extract 2 cc. of sodium hydroxide solution (100 grams in 100 cc. of water), 100 cc. of water and heat under a reflux condenser one half-hour. Acidify with 5 cc. of dilute sulphuric acid (1:4), add a few pieces of pumice stone, distil in a current of steam and titrate the distillate with tenth-normal alkali, using phenolphthalein as indicator. The number of cc. required represents the total volatile acids free and combined. Determine free volatile acids, if present by direct distillation and titration of the distillate. The difference between the two titrations is calculated as ethyl acetate.



## CHAPTER XXI.

### VEGETABLE AND FRUIT PRODUCTS.

#### CANNED VEGETABLES AND FRUITS.

STRICTLY speaking all varieties of canned foods found in the market, whether meats, fruits, or vegetables, in order to be entirely beyond criticism, should not differ from the corresponding freshly cooked varieties which they are intended to replace, excepting that they are free from bacteria. Such perfection is, however, difficult to attain and most commercial canned products, even if made from the best materials and free from chemical preservatives or artificial colors—now frowned on by American manufacturers—contain metallic impurities accidentally derived from the containers in which they are sealed or, less often, from the vessels in which they are prepared. In spite of the objections, canned foods form a convenient, and in some cases indispensable means of furnishing both necessities and luxuries for the table. The canning of foods is especially useful for preserving them during long periods of time, for enabling certain fruits and vegetables to be enjoyed out of season, and for furnishing supplies in a convenient manner to inaccessible places where fresh foods are not readily obtainable, as in the case of armies in the field, of vessels at sea, of campers in the woods, etc. Canned goods in great variety are used in nearly every household.

When it is considered that in the United States alone several hundred million cans of tomatoes, corn, and peas are packed in a single year, to say nothing of an ever-increasing variety of other foods, some idea may be gained of the enormous proportions to which the canning industry has grown. It is comforting to know that, in view of their wide-spread consumption, the greater portion of such foods found on the market are comparatively harmless, as is evidenced by the fact that few cases of injury to health have been directly traceable to their use.

**Method of Canning Food.**—Various modifications as to details exist with different products and in different localities, but in general the principle of canning in tin is the same in all cases. The fresh product is



cleaned carefully, the refuse removed by shelling, paring, or other treatment, in most cases "blanched" (immersed in hot water for a period of time), and packed into cans. A weak brine to which has been added a little sugar in the case of corn, peas, etc., is added to vegetables and a syrup of various strengths to fruits. If the soldered can is used the cap is attached at this stage of the process, if the sanitary can, it is left open and "exhausted" at a temperature slightly below the boiling-point of water. In either case the process of sterilization is then carried out by heating in a saline solution or a dry retort at a suitable temperature above  $100^{\circ}\text{C}$ . The soldered can at this point is punctured to allow the escape of the compressed air, then closed with a drop of solder, while the sanitary can is sealed by double seaming on the cover using a special cement to insure tightness.

The canning of peas is fully described by Bitting\* and of other vegetables and fruits by Zavalla.†

Cooked vegetables and fruit products put up in glass jars or bottles are tightly sealed when hot, either with screw-caps or with some form of cover held by a clamp, or with metal or hard-rubber caps fitting over a flanged mouth. Commonly a soft-rubber ring is inserted between the cover and the mouth of the jar or bottle. The material of the cover is generally either glass, porcelain, or metal. Cork stoppers are, however, sometimes pressed into the mouths of the bottles, and made extra tight therein with sealing-wax. These stoppers are occasionally soaked in paraffin. Thus the contents of the jar may be exposed to porcelain, glass, metal, rubber, or cork, according to the material of the cover and the method of sealing.

The preservation of food by canning was long thought to be due to the perfect exclusion of air, but is now known to depend on the perfect sterilization, or destruction of bacteria, and it has been proved that as far as keeping qualities are concerned, it makes no difference whether or not air is present in the can, if the contents are sterile, though for purposes of inspection the vacuum, in the case of tin cans, is of great use, in that as a natural consequence of the vacuum, when the goods are sound, the ends of the cans are usually concave. The highest aim of the canner should be to retain in his product as far as possible the appearance, palatability, and nutritive value of the freshly cooked food.

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 125, 1909.

† Canning of Fruits and Vegetables, New York, 1916.



**Proximate Analyses** of canned vegetables and fruits, as found on the market, have been made by various authors, and are useful in showing the food value of the products. The results in the table as given below are from Atwater and Bryant's compilations. There is a lack of data on samples of known origin on which suitable standards may be based.

PROXIMATE COMPOSITION OF CANNED VEGETABLES AND FRUITS.\*

	No. of Analyses.	Water.	Protein.	Fat.	Total Carbohydrates.	Crude Fiber.	Ash.	Fuel Value per Pound.
<b>CANNED VEGETABLES.</b>								
Artichokes.....	3	92.5	.8	.....	5.0	.6	1.7	110
Asparagus.....	14	94.4	1.5	.1	2.8	.5	1.2	85
Beans, baked.....	21	68.9	6.9	2.5	19.6	2.5	2.1	600
" string.....	29	93.7	1.1	.1	3.8	.5	1.3	95
" Lima.....	16	79.5	4.0	.3	14.6	1.2	1.6	360
Brussels sprouts.....	1	93.7	1.5	.1	3.4	.5	1.3	95
Corn, green.....	52	76.1	2.8	1.2	19.0	.8	.9	455
Peas, green.....	88	85.3	3.6	.2	9.8	1.2	1.1	255
Pumpkin.....	7	91.6	.8	.2	6.7	1.1	.7	150
Squash.....	5	87.6	.9	.5	10.5	.7	.5	235
Succotash.....	12	75.9	3.6	1.0	18.6	.9	.9	455
Tomatoes.....	19	94.0	1.2	.2	4.0	.5	.6	105
<b>CANNED FRUITS.</b>								
Apples, crab.....	1	42.4	.3	2.4	54.4	.....	.5	1,120
Apple sauce.....	1	61.1	.2	.8	37.2	.....	.7	730
Apricots.....	1	81.4	.9	.....	17.3	.....	.4	340
Blackberries.....	1	40.0	.8	2.1	56.4	.....	.7	1,150
Blueberries.....	3	85.6	.6	.6	12.8	.....	.4	275
Cherries.....	1	77.2	1.1	.1	21.1	.....	.5	415
Peaches.....	3	88.1	.7	.1	10.8	.....	.3	220
Pears.....	4	81.1	.3	.3	18.0	.....	.3	355
Pineapples.....	1	61.8	.4	.7	36.4	.....	.7	715
Strawberries.....	1	74.8	.7	.....	24.0	.....	.5	460

\* U. S. Dept. of Agric., Exp. Sta. Bul. 28, p. 70.

The determination of the drained solids is often made with the view of detecting, especially in tomatoes, an excess of water added as such or as drainage from a better grade, but there are various unavoidable influences which affect the results. In addition to natural variations of canned tomatoes Bigelow \* has shown that freezing, agitation during shipment, and other factors exert no little influence, while McGill † found as great variation in the different cans of the same brand as between the average of different brands. Obviously this determination is not applicable to canned vegetables such as pumpkin, which have little or no liquor.

\* Jour. Assn. Off. Agr. Chem., 3, 1917, p. 1.

† Lab. Int. Rev. Dept. Canada, Bul. 357, 1916.



**DECOMPOSITION.**—"Swells."—In the case of canned vegetables and fruit products, decomposition rarely results in the formation of ptomaines even after the can has long been open, though these toxins are sometimes formed in canned meat and fish. Spoilage is readily apparent after opening a can, from a cursory examination of its contents. The appearance, taste, and odor will not fail to indicate the unfitness of the contents for food, if decomposition is at all advanced. It is, however, often of great advantage to detect spoiled cans without opening. As a rule, when a can is spoiled, it is usually in the condition termed "blown," i.e., with its ends convex, instead of normal or concave.

Doremus\* has shown that when the cans have become putrid carbon dioxide and hydrogen are the chief gases to be found.

According to Prescott and Underwood,† although nearly all forms of bacterial decomposition are accompanied by bulging of the ends of the cans, there are some exceptions. In the souring of canned sweet corn, which they trace to at least twelve varieties of bacteria, it is exceptional that swelling occurs. These "flat sours" are detected as follows: Boil the cans for an hour, causing the ends of all to swell, then cool, and set aside for eight hours, during which the sound cans will snap back, while the unsound will continue convex, by reason of the fact that the swelling in this case is due to the generation of gas by the bacteria present.

Ordinarily, in the factory inspection of canned goods before shipping, not only are the bulged cans or "swells," as they are termed, sifted out, but the condition of the cans is tested by sounding or striking the cans. If the contents are sweet, a peculiar note is produced when the can is struck, readily distinguishable from the dull tone of the unsound can by anyone familiar with the work.

"**Springers**" are cans which may appear normal in ordinary weather but have bulged ends on hot days. Baker,‡ who has made a special study of springers, has found that the gas present consists chiefly of carbon dioxide formed during processing, nitrogen from the air remaining after oxygen has combined with the tin or iron or else contents of the can, and usually hydrogen. He concludes that this abnormality may be largely obviated by avoiding over filling, closing the can while hot, thus producing a vacuum, avoiding delay in the process of canning, and using enameled cans for foods with high acidity.

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\* Jour. Amer. Chem. Soc., 19, 1897, p. 733.

† Tech. Quart., 10, 1897, p. 183; 11, 1898, p. 6.

‡ 8th Int. Cong. App. Chem., 18, 1912, pp. 39, 45.



Bigelow \* believes the hydrogen is formed by organic acids acting on the iron of the can, hence non-acid foods rarely form springers due to hydrogen. He further states that the three staples, tomatoes, peas, and corn, neither attack the metal considerably nor form springers from this cause. When the bulging is pronounced he advocates condemning the product because of the iron taste and the difficulty of distinguishing such springers from swells due to biological decomposition.

**METALLIC IMPURITIES.**—**Salts of Lead and Tin** are commonly met with in varying amounts in nearly all classes of products put up in tin. The quantity dissolved depends largely on the character of the tin plate used in the manufacture of the can, as well as on how the solder is applied. Much depends also on the nature of the food product and its acidity. Formerly much danger was apprehended from the use of the so-called *terne* plate as a material for cans. This consists of an alloy of lead and tin, coated on iron plate and intended for use as roofing. Sometimes two parts of lead to one part of tin are found in *terne* plate. Only the better grades of bright tin plate should be used in canning. There is reason to believe that no *terne* plate is at [present used in cans. In 1892 the plating alloy of 47 samples of tin cans in which peas had been put up were examined in the Bureau of Chemistry of the U. S. Department of Agriculture,† and the amount of lead found varied from 0% to 13%. Only 4 samples were found to exceed 5%, and 24 contained less than 1%.

The construction of the can should be such that practically no soldered surface is exposed to the contents, the joints being lapped and soldered on the outside. In spite of this, however, it is not unusual to find cans soldered on the inside, or lumps of solder in the can from the sealing of the tapped hole. From 51% to 65% of lead was found in the solder taken from the interior of twenty-four of the cans mentioned in the preceding paragraph.‡

Cans lacquered on the inside to prevent contact of the metal with the food are coming into use but as yet are not an unqualified success. Some of the lacquers which have proved most efficient are objectionable because of their lead content.

**Action of Fruits and Vegetables on Tin Plate.**—A large variety of canned products have been examined in the laboratory of the Massachu-

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\* Nat. Can. Assn. Res. Lab., Bul. 2, 1914.

† Bul. 13, p. 1036.

‡ Ibid., p. 1038.



setts State Board of Health, with a view to determining the quantity of tin contained in solution. The results have shown that though notable traces of tin were found in acid fruits and rhubarb, and large traces in some green vegetables, canned blueberries were found to contain, as a rule, much more tin in solution than any other canned goods examined. It is assumed that the tin was, at least in considerable part, still held in solution by the fruit acids, inasmuch as the metal was found in the filtered juice. In every instance the inner tin lining was found to be extensively corroded, and in some cases it had been almost entirely dissolved

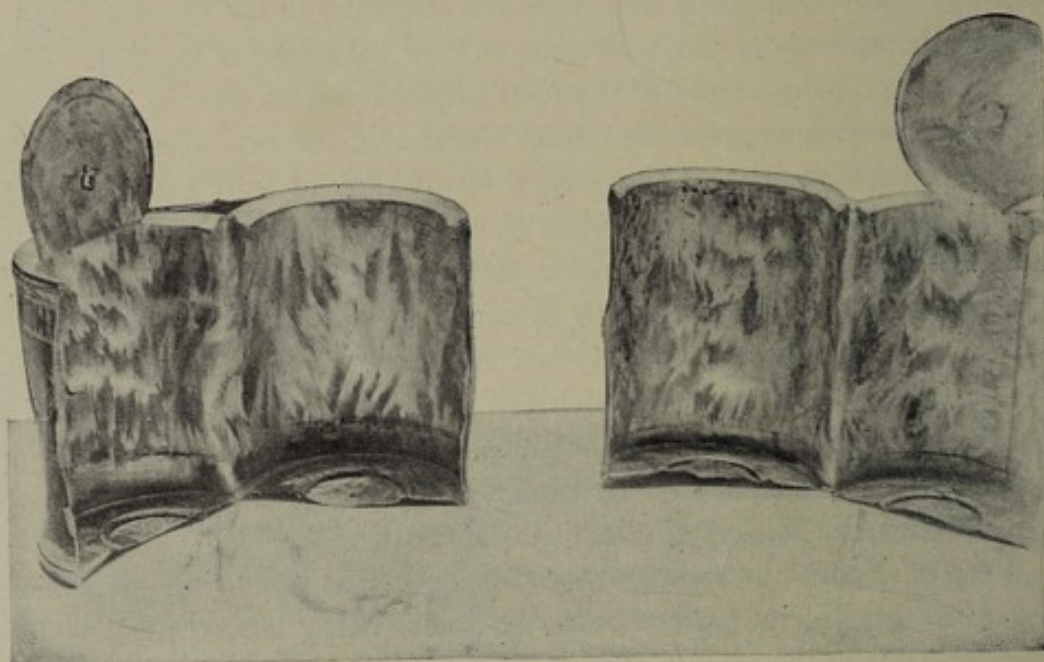


FIG. 119.—Interior of Blueberry Cans, Cut Open to Show the Corrosion by Acid of the Fruit Juice.

off, leaving the underlying iron bare. Fig. 119 shows the appearance of two of these cans, split open to show the inner surfaces. The corrosion is apparent. Eleven samples of canned blueberries, representing seven brands, were examined in 1894 by Worcester, showing an amount of tin solution (calculated as  $\text{SnO}_2$ ) varying from 0.066 to 0.27 gram per can of 615-cc. capacity.

In 1899 samples of various canned products were examined for lead and tin in the author's laboratory, the results of which are thus summarized:\*

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\* An. Rep. Mass. State Board of Health, 1899, p. 623.



	Tin, Grams.	Lead, Grams.	Capacity of Can, cc.
Strawberries. ....	.....	.....	615
Highest. ....	.0393	.0004	
Lowest. ....	.0124	.0000	
Raspberries. ....	.....	.....	615
Highest. ....	.0848	.0002	
Lowest. ....	.0725	.0001	
Blueberries. ....	.....	.....	615
Highest. ....	.2226	.0021	
Lowest. ....	.0056	.0004	
Tomatoes. ....	.....	.....	950
Highest. ....	.0515	.0004	
Lowest. ....	.0146	.0001	
String beans. ....	.....	.....	650
Highest. ....	.0499	.0003	
Lowest. ....	.0065	.0008	
Peas. ....	.....	.....	615
Highest. ....	.0046	.0000	
Lowest. ....	.0024	.0001	
Corn. ....	.....	.....	615
Highest. ....	.0101	.0011	
Lowest. ....	.0045	.0001	
Lima beans. ....	.0064	.0004	650
Succotash. ....	.0039	.0001	650
Squash. ....	.....	.....	950
Highest. ....	.1793	.0087	
Lowest. ....	.1577	.0003	
Pumpkin. ....	.1844	.0019	950
Rhubarb. ....	.3506	.0002	615
Asparagus. ....	.1249	.0001	930
Mutton broth. ....	.0114	.0001	950
Tomato soup. ....	.0023	.0002	370
Salmon. ....	.0319	.0001	470
Lobster. ....	.0411	.0001	430

A wide range of variation exists in the amount of tin dissolved. Pumpkin and squash, for example, dissolve surprisingly large quantities, considering the supposed inert nature of these vegetables.

In samples of canned sardines put up in mustard, vinegar, and oil, the Massachusetts Board has found as high as 0.376 gram of tin in a half-pound can. In these cases the corrosion of the interior of the cans was very marked.\*

**Effect of Time on Amount of Tin Dissolved.**—A series of experiments was conducted by the author in 1899 † on the action of various fruit acids on tin, with a view to ascertaining, among other facts, whether or not the element of time exerts an appreciable difference in the results.

Samples of various canned fruits and vegetables were titrated for

\* The U. S. Government, pending further investigation, permits 300 mg. of tin per kilo in canned goods. F. I. D. No. 126.

† An. Rep. Mass. State Board of Health, 1899, p. 624.



their acidity. It was found that certain samples of canned blueberries, for instance, had an acidity of about one-twentieth normal. In the case of strawberries, the acidity was about one-sixth normal. Canned raspberries were found to be about one-tenth normal in acidity, while the acidity of canned tomatoes varied from one-tenth to one-fourteenth normal. Solutions of one-fifth, one-tenth, and one-fifteenth-normal malic acid, one-tenth and one-fifteenth-normal tartaric acid, one-tenth and one-fifteenth-normal citric acid, and one-tenth-normal acetic acid were prepared and sealed in pint glass jars, having about the same capacity as the ordinary-sized tin fruit cans, each jar containing an amount of tin plate equivalent to the interior exposed surface of a can. Solutions thus sealed were kept for three months, six months, and a year, and examined at the end of these respective periods for tin. The results showing the amount of tin found at the end of three months in each case are given in the following list:

#### ACTION OF FRUIT ACIDS ON TIN IN THREE MONTHS.

Acid.	Grams of Tin in One Pint of Solution.	Acid.	Grams of Tin in One Pint of Solution.
N/5 malic.....	0.0578	N/15 tartaric.....	0.0246
N/10 ".....	0.0201	N/10 citric.....	0.0374
N/15 ".....	0.0197	N/15 ".....	0.0236
N/10 tartaric.....	0.0382	N/10 acetic.....	0.0019

It was found that, as a rule, the amount dissolved in three months was the same as in six months or even a year.

Tenth-normal acetic acid sealed in jars with tin plate, as in the case of the fruit acids, dissolved in three months 0.0019 gram, and in six months 0.0083 gram of tin, which is much less than was obtained with fruit acids of the same strength, and with the samples of sardines referred to on page 263.

Bigelow and Bacon find that shrimps contain monomethylamin, which corrodes the cans in which they are packed. Their experiments with volatile alkalis and amino acids present in vegetables of low acidity indicate that the corrosive action of certain vegetables is due to substances of this group.

**Influence of Different Weights of Tin Coating.**—A committee of the National Cannery Association, the American Sheet and Tin Plate Company, and the American Can Company has reported results of extensive experi-



ments on the action of various canned foods on cans with tin plate coatings of from 0.9 to 3.0 pound of tin per base box. Determinations of tin and iron were made at intervals. The figures in the following table show the extreme amounts of tin dissolved from the lowest and highest weights of plate and during short and long periods. The amounts of iron dissolved were more nearly uniform, the maximum being 90 mg. per kilo.

## TIN IN CANNED VEGETABLES AND FRUIT

Mg. per Kilo

	StringBeans.		Corn.		Peas.		Pumpkin.		Tomatoes.		Apples.	
	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.	Min.
Packed 1½-5 mo.												
0.9-lb. plate...	97	75	13	3	32	9	82	31	175	42	166	34
3-lb. plate....	161	126	14	3	27	15	194	37	61	36	189	29
Packed 10½-13 mo.												
0.9-lb. plate...	220	154	...	....	26	12	476	44	148	58	203	27
3.0-lb. plate...	338	174	...	...	26	14	824	174	186	36	228	35

The committee in its conclusion states: "The lustre and the resistance to rusting increase somewhat with increased weight of coating. In other respects, with the exception of some instances in those classes of foods that have a tendency to perforate, the conclusion from this work is that the value of different weights of tin coating on food containers is for all practical purposes the same with average weights of from 1 to 3 pounds of tin per base box."

**Salts of Lead.**—While it is a fact that the material of the tin plating usually found in cans is comparatively low in lead, the same is not always true of the metal caps used to cover some of the bottled goods. The French "haricots verts" are usually sold in wide-mouthed bottles, closed by a disk of very soft metal. In one instance this metal cap, which came in contact with the liquid contents of the bottle, was found to contain 93½% of lead. Of the various kinds of bottles in which are sold cheap carbonated drinks known as "pop," one style has a stopper consisting of a metallic button surrounded by a rubber ring. These metallic buttons consist of tin and lead in varying proportions. Inasmuch as the inclosed liquor was usually found to be quite acid in reaction, the danger of prolonged contact with the metallic portion of the stopper is evident.

The following table gives the percentage of lead found in the stoppers



of this character, together with the amount of lead contained in the liquor.\*

Character of Sample.	Per Cent of Lead in Stopper.	Amount of Lead in Contents of Bottle in Milligrams.†
Blood orange .....	50.7	0.31
Birch beer. ....	35.0	Large trace
Ginger.....	32.2	0.40
Strawberry A. ....	8.8	0.20
Strawberry B. ....	6.5	0.30
Sarsaparilla A. ....	8.5	0.19
Sarsaparilla B. ....	3.5	0.17
Lemon. ....	7.5	0.27
Miscellaneous (20 samples)		
Maximum.....	50.3	1.05
Minimum. ....	3.8	0.01

† Capacity of bottle about  $\frac{1}{2}$  pint.

Besides the above tabulated samples, twenty were found with stoppers containing less than 3% of lead. While the amount of lead found in the contents of the bottles was in no case very large, it was enough to condemn the use of lead in the manufacture of such stoppers. That the amounts of lead found in the contents of the bottles vary quite irrespective of the percentage of lead in their stoppers, may be ascribed to various causes, such as the difference in the acidity of the liquors, and the length of time that the liquor has been in contact with the stopper. Furthermore, the more soluble metal of an alloy is attacked by an acid with an energy which is not proportional to the percentage of that metal in the alloy.

**Salts of Zinc.**—The presence of zinc salts in canned foods is largely accidental, and is generally due either to the contact of the acid fruits and vegetables with galvanized iron in the canneries, to the occasional use of brass vessels, or to the zinc chloride used as a soldering fluid. Hilgard and Colby † have examined empty tin cans fresh from the manufacturer, and found zinc chloride in notable quantity in the seams, and especially in the empty space of the lap at the bottom of the can, where it could easily be acted on by the contents. The average amount of soluble zinc chloride found in the "lap" alone amounted to three-fourths of a grain per can. It was furthermore ascertained that it was not the practice of canners to wash the cans before packing, so that zinc present in canned goods may thus readily be accounted for.

\* An. Rep. Mass. State Board of Health, 1897, p. 571.

† Rep. Cal. Agric. Exp. Sta., 1897-8, p. 159.



Zinc chloride is commonly used in machine soldering, but should be displaced by rosin.

Holgard and Colby found in some spoiled cans of asparagus, where the acidity was unusually high, an average of 6.3 grains of zinc chloride per large can.

Zinc salts are said to have been used for greening peas, but their use for this purpose is not common. Zinc chloride is the salt used, and a natural yellowish-green tint is imparted when properly applied. The process has been kept secret.

**Salts of Copper.**—While copper in canned goods is sometimes accidental, its presence being due to the use of copper or brass vessels in the canneries, its chief interest to the food analyst lies in the use of copper sulphate for greening peas and other vegetables. The artificial greening of vegetables is much more commonly practiced in France than in the United States.

French canners of peas, beans, Brussels sprouts, etc., are frequently so lavish in the use of sulphate of copper that the goods as found on our markets can in some cases hardly be said to resemble the freshly cooked products in color. Oftentimes, indeed, they possess such a deep green as to be positively distasteful to the average American palate, though evidently this unnatural hue is craved in Europe. The use of copper in such foods is often rendered apparent by the most cursory examination.

In this country the use of copper was commonly in smaller amounts than in France even before regulations prohibiting its use were adopted.

Complaint in court for this form of adulteration under the general food law as it exists in most states would naturally be brought under one of two clauses:

1st. As being colored, whereby the product appears of greater value than it really is, or

2d. As containing an ingredient injurious to health.

An ingenious claim is sometimes advanced by the defendant in opposition to clause 1, to the effect that copper sulphate is added, not to give an artificial green color, but to preserve the original green of the chlorophyll or natural color of the fresh peas,\* so that it will not be destroyed by subsequent boiling.

This point was argued in a strongly contested court case brought in Massachusetts for copper in French peas.†

\* The term used by the French to describe this process is *reverdissage* or "regreening."

† An. Rep. Mass. State Board of Health, 1892, p. 605.



As Worcester\* has shown, the fallacy of this argument can be easily demonstrated. If it were true that the copper acts as a preservative of the chlorophyll, a pure extract of chlorophyll should, by the addition of copper sulphate, be prevented from destruction on boiling, and again, on once destroying the color of the chlorophyll by boiling, it would be impossible to restore it by further boiling it with copper sulphate.

As a matter of fact, if an extract of chlorophyll is boiled with a dilute solution of copper sulphate, its color is at once destroyed, and a brown precipitate is thrown down. On the other hand, if yellow or white peas or beans devoid of chlorophyll are boiled with copper sulphate, they are colored green, the depth of color depending on the strength of the copper solution. When peas or other vegetables are thus colored, very little copper is found, as a rule, in the liquid contents of the can, but the copper is chiefly confined to the solid portions. Green compounds are produced by boiling albumins with copper salts, due to the formation of albuminate, or in the case of peas, leguminate of copper. Harrington† states that it is possible to color eggs an intense green by boiling with copper sulphate.

Examination of a large number of brands of canned vegetables greened by copper, as bought in Massachusetts, showed that the amount used varied from a trace to 2.75 grams per can, calculated as copper sulphate. In justice to the consumer, who may be cautious about taking into his system copper salts, as well as to those who are indifferent to their use, it is no more than fair that all cans should have a label, plainly stating the quantity present. In the Massachusetts market, labels like the following are not uncommon: "This package of French Vegetables contains an equivalent of Metallic Copper not exceeding three-quarters of a grain."

Copper as a coloring matter has been most commonly found in peas, beans, and Brussels sprouts. Copper salts in minute quantity have been found in Massachusetts in canned tomatoes, clams, and squash, as well as in pickles.

**Salts of Nickel.**—Sulphate of nickel has been employed instead of sulphate of copper for greening vegetables. According to Harrington‡ 0.25 gram of nickelous sulphate per kilogram of peas is used. The peas or other vegetables are boiled in a solution of the salt, made slightly alkaline with ammonia.

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\* Loc. cit., p. 641.

† Practical Hygiene, p. 203.

‡ Ibid., p. 205.



**Toxic Effects of Metallic Salts.**—Divergence of opinion is so great as to the toxic effects of salts of the heavy metals on the human system, when present in the small amounts commonly found in food products, that it is extremely difficult to maintain a complaint in court based entirely on the harmful effects of these salts. Since the question is one for the toxicologist or physiological chemist rather than the analyst to settle, it will not be discussed here at length.

The toxic action of lead salts is too generally recognized to need discussion.

Authorities were not agreed on the toxic action of copper as present in coppered vegetable, hence the desirability of experiments such as were conducted by the Referee Board of Consulting Scientists.\* Their results indicate that while as much as 150 mg. of copper may be contained in the coppered beans or peas eaten in a day as little as 10 mg. under certain conditions may have a deleterious action and must be considered injurious to health. Accordingly foods greened with copper are considered adulterated by the federal authorities.†

The case of tin is quite different from that of lead and copper, since it is an accidental impurity which it is not practicable to eliminate with our present knowledge; furthermore its toxicity is less. Investigations by Bigelow ‡ and Goss § show that the tin in canned goods exists usually in greater amount as insoluble than as soluble compounds.

**PRESERVATIVES.**—No class of food products stands so little in need of these added substances to arrest fermentation as canned foods, if properly prepared and, as a matter of fact, the use of antiseptics has been almost entirely discontinued.

**The Bleaching of Corn** by artificial means before canning is usually accomplished by boiling the corn with sodium sulphite, thus giving to the product an unnaturally white color. The practice seems to have been more in vogue some years ago than at present, the popular taste now apparently preferring the natural rich yellow of fresh corn.

**Saccharin** is claimed to possess antiseptic powers and is used in canned goods, but its primary purpose is as a sweetener.

**Salicylic Acid, Sodium Benzoate, and Beta-naphthol**, although formerly used, are now seldom found in canned goods.

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\* U. S. Dept. of Agric., Rep. 97, 1913.

† Food Inspection Decision 148.

‡ Jour. Ind. Eng. Chem., 8, 1916, p. 813.

§ Ibid., 9, 1917, p. 144.



**"SOAKED GOODS."**—It has become quite common, especially in the case of peas, beans, and corn, to utilize for canning purposes those that have grown old and dried, after soaking them for a long time. The presence of soaked peas in the market is generally more common in years when there is a scarcity in the pea crop. By the process of soaking, dried and matured field corn may be softened to such an extent as to be substituted for green or sweet corn in the canned product. These goods, frequently sold at a very low price, under some such tempting name as "Choice Early June Peas," are entirely devoid of that succulent property so highly prized in the fresh goods, and are altogether so inferior in quality that their sale may justly be considered as fraudulent, unless their character is specified. In some states the law provides that such a product, to be legally sold, shall have plainly marked on the label of the can the words "Soaked Goods" in letters of prescribed size.

**Detection.**—Methods of detecting soaked goods are distinctly physical rather than chemical. While chemical analysis may not be decisive, the appearance and taste of the goods furnish in most cases an unmistakable clue to their nature. Soaked goods are entirely lacking in juiciness, and in the flavors so characteristic of the various vegetables, when gathered and canned before becoming dry. The process of soaking is also said to develop the growth of the rudimentary stem of the embryo in the dried pea and bean. Peas and beans of the soaked variety are almost entirely lacking in the green color of the fresh vegetables, unless the color has been artificially supplied. The liquid is commonly milky.

In all cases it will be found that the solid grains or kernels of the peas, beans, and corn that have once been dried, though softened by the process of soaking, have much less water than the grains of the corresponding vegetables that were gathered while still soft and succulent.

#### METHODS OF ANALYSIS.

**Methods of Proximate Analysis.**—These are essentially the same as for cereal products with such variation in the preparation of the sample as is necessitated by the moist condition and lack of uniformity.

**Examination of Gases from Spoiled Cans.**—Fig. 118 shows the Doremus apparatus for puncturing the can with a hollow needle and conducting the gases into a eudiometer, where they are examined by the usual methods for gas analysis. Baker in his investigations used in conjunction with this apparatus a frame with another puncturing needle through which



water was introduced under pressure, thus forcing out all the gases through the Doremus needle.

**Determination of Drained Solids.**—Scarcely two workers have followed the same method. The results obtained depend on whether a sieve or cheese cloth is used for straining, the size of the mesh, the time allowed for draining, and whether or not any pressure is applied.

Magruder \* uses a sieve with 1 mm. round holes and allows to stand for about five minutes, stirring gently with a spatula at the beginning and end.

McGill † turns out the contents of the can upon a piece of cheese cloth of known weight spread upon a sieve 6 inches in diameter and drains for

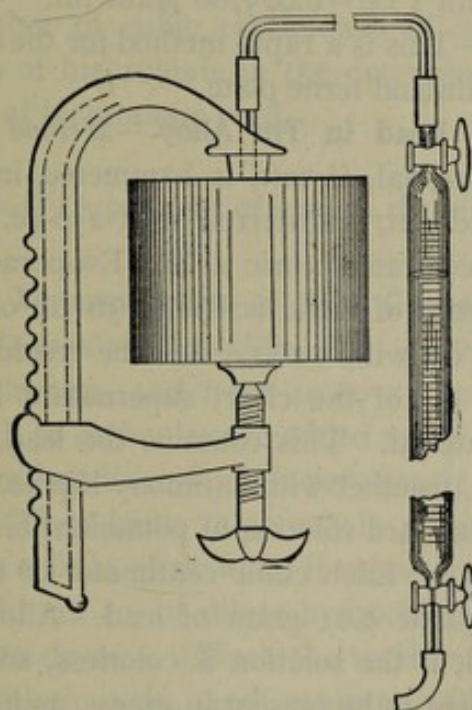


FIG. 118.—Apparatus for Collecting Gases from Spoiled Cans. (After Doremus.)

approximately two hours without pressure or until drops fall at intervals of more than five seconds.

Ladd, ‡ in addition to a  $\frac{1}{4}$ -inch mesh sieve adopted by the Canners' Associations of three states some years since, employs a cheese cloth to retain the finely divided matter, a method which, according to Bigelow's experiments, † gives 3% to 6% more drained solids than the sieve alone.

\* Jour. Assn. Off. Agr. Chem., 1, 1915, p. 199.

† Lab. Inl. Rev. Dept. Canada, Bul. 357, 1916.

‡ Jour. Assn. Off. Agr. Chem., 3, 1917, p. 1.



**Determination of Tin on Tin Plate.**—*Baker Method.\**—Cut 4 square inches of the plate, loosely fold, introduce into a 300-cc. Erlenmeyer flask with from 50 to 100 cc. of concentrated hydrochloric acid, and determine the tin by the method as described for contents (p. 875), using, however, an iodine solution of such strength that, with the size of sample employed, 10 cc. is equivalent to 1 pound of tin per base box.

For the preparation of the iodine solution, dissolve 45 grams of iodine and 65 grams of potassium iodide in a small amount of water, dilute to 4 liters, allow to stand overnight, check against solutions containing a known amount of tin and an amount of iron equivalent to that used in a sample, and dilute until 1 cc. = 0.005786 gram tin.

*Hiltner Method.†*—This is a rapid method for the determination of lead as well as tin in both tin andterne plate.

**Determination of Lead in Tin Alloy.**—*Method of Paris Municipal Laboratory.‡*—The material, if soft, is hammered into a thin plate, and 2½ grams are weighed out, transferred to a 250-cc. flask, and dissolved in 7 to 8 cc. of concentrated nitric acid. Evaporate to dryness on the sand-bath, add 10 drops of nitric acid and 50 cc. of boiling water, cool, and make up to 250 cc. with water. Let the residue settle and pour off through a filter 100 cc. of the clear, supernatant liquid, corresponding to 1 gram of the material. This contains the lead, while the tin is left behind in the residue, together with antimony if present.

Add 10 cc. of a standard solution of potassium bichromate (7.13 grams to the liter) and shake. Each cubic centimeter of this standard solution is sufficient to precipitate 0.01 gram of lead. Allow the lead chromate formed to settle, and, if the solution is colorless, add 10 cc. more of the bichromate, or sufficient to be present in excess, as indicated by the yellow color. Filter, wash, and titrate the excess of bichromate with a standard iron solution, containing 57 grams of the double sulphate of iron and ammonia and 125 grams of sulphuric acid per liter. This iron solution should be kept under a layer of petroleum, and standardized against the potassium bichromate before use.

Add, drop by drop, the iron solution to that containing the excess of bichromate. The color of the latter passes from pale green to bright green, when the chromate is completely reduced. Determine the end-

\* Relative Value of Different Weights of Tin Coating on Canned Food Containers, Nat. Can. Assn., Washington, 1917, p. 31.

† Western Chem. Metal., 4, 1908, p. 262.

‡ Girard, *Analyse des Matières Alimentaires*, Paris, 1904, p. 829.



point with a freshly prepared dilute solution of potassium ferricyanide, a drop of which is placed on a porcelain plate or tile in contact with a little of the solution titrated. A blue color is produced when the iron is present in excess. If the standard iron and bichromate solutions exactly correspond, 1 cc. of the iron solution is equivalent to 1% of lead, but the latter solution is usually a little weak.

If  $n$  = number of cubic centimeters of iron solution necessary to reduce 10 cc. of the standard bichromate,

$$1 \text{ cc. of the iron solution} = \frac{10}{n}.$$

If, now,  $r$  = number of cubic centimeters of iron solution necessary to reduce the excess of bichromate in the determination, and  $s$  = number of cubic centimeters of bichromate used,

$$s - \frac{10}{n}r = \text{per cent of lead in the alloy.}$$

**Separation and Determination of Tin, Copper, Lead, and Zinc in Canned Goods.**—*Munson's Method.*\*—The contents of the can are first evaporated to dryness, and from 10 to 15 cc. of concentrated sulphuric acid or enough to carbonize are added to the dry residue contained in a porcelain evaporating-dish, which is very gently heated over the flame till foaming ceases. Then ignite to an ash in a muffle, or carefully over the free flame, using a little nitric acid, if necessary, for oxidation of the organic matter. Add 20 cc. of dilute hydrochloric acid, and evaporate over the water-bath to dryness. Wash the residue into a beaker, slightly acidify with hydrochloric acid, and saturate with hydrogen sulphide without previous filtration. Heat the beaker on the water-bath, and pass the contents through a filter. Wash the precipitate, which contains sulphides of tin, lead, and copper, if these metals are present, while if there is zinc, it is contained in the filtrate. The precipitate is fused with sodium hydroxide in a silver crucible for half an hour, to increase the solubility of the tin, which would otherwise be dissolved with difficulty. The fusion is boiled up with hot water, acidulated with hydrochloric acid, and transferred without filtering to a beaker, in which hydrogen sulphide is added to saturation. This precipitates the sulphides of tin, lead, and copper (if these metals are present). The sulphide precipitate is collected

\* U. S. Dept. of Agric., Bur. of Chem., Bul. 107 rev., p. 62.



on a filter, and thoroughly washed with hot water, the washings being rejected. Pass through the filter several portions of boiling ammonium sulphide, using about 50 cc. in all, or till all the tin is dissolved. Precipitate the tin from the combined filtrate with hydrochloric acid, filter, wash, ignite, and weigh as stannic oxide.

The residue left on the filter, after dissolving out the tin sulphide, is then dissolved by treatment with nitric acid, which is filtered, and to the filtrate and washings ammonia is added nearly to the point of neutralization. Then add ammonium acetate. Filter off any precipitate of iron that may be formed. The filtrate is divided into two portions for determination of copper and lead. If lead is absent, determine the copper by titration with potassium cyanide\* or electrolytically (p. 634). Copper is rarely present in sufficient amount to be determined, unless used for greening the vegetables. If notable quantities of lead are present, the solution is made acid with acetic, and the lead precipitated therefrom with potassium chromate, collected on a tared filter, washed with water acidified with acetic acid, dried at 100° C., and weighed as lead chromate. Or determine the lead by color-tests, as on page 362.

For the determination of zinc, the filtrate from the first hydrogen-sulphide residue is evaporated to a volume of about 60 cc., and treated with bromine water to oxidize the iron, as well as any excess of hydrogen sulphide remaining, the excess of bromine is then boiled off, and a few drops of concentrated ferric chloride added, to make the solution distinctly yellow, if not already so. Nearly neutralize with ammonia, and precipitate the iron with ammonium acetate. Filter, wash, acidify the filtrate with acetic acid, and precipitate the zinc with hydrogen sulphide. Filter, wash, ignite, and weigh as zinc oxide.

The metals may be determined separately, as follows:

**Determination of Tin.**†—Evaporate the contents of the can to dryness, and ignite in porcelain. Fuse the ash with sodium hydroxide in a silver crucible, boil the fusion with several portions of water acidulated with hydrochloric acid, filter, and precipitate the tin from the acid solution with hydrogen sulphide. Dissolve the washed precipitate in ammonium sulphide, filter, and deposit the tin directly from this solution by electrolysis in the platinum dish which contains it, using a current of 0.5 ampere and the electrolytic apparatus described on page 634.

\* Sutton, Volumetric Analysis, 8th ed., p. 204.

† Hilger u. Leband, Zeits. Unters. Nahr. Genussm., 2, 1899, p. 795; An. Rep. Mass. State Board of Health, 1899, p. 625.



*Smith and Bartlett*\* employ the following method of solution: Weigh 50 grams of fish or 100 grams of vegetables in a porcelain dish and dry overnight. Heat from 75 to 100 cc. of concentrated sulphuric acid in a Kjeldahl flask until acid fumes are visible, then add gradually small portions of the food product, heating the acid between additions until frothing ceases. Allow to cool, then add gradually to the charred mixture 25 cc. of concentrated nitric acid, which causes the evolution of red fumes and the generation of heat. Cool, add 25 cc. of nitric acid and heat gently until all nitric fumes are expelled and the charred material is dissolved to a homogeneous solution. Boil this solution about forty-five minutes, then add from 10 to 15 grams of potassium sulphate and continue boiling from three to five hours until decolorized. Wash the digest into an 800-cc. beaker, dilute to about 600 cc. and bring to a boil. Almost all of the tin separates as stannic oxide, partially hydrated, some of which adheres to the sides of the flask, and cannot be removed by washing. Filter the contents of the beaker, thus separating the hydrated stannous oxide from all other compounds. Place the filter in the flask, to which 20 cc. of saturated sodium hydroxide and an equal volume of water have been added, boil for several minutes, then wash the sodium stannate into a beaker. Acidify with hydrochloric acid, precipitate with hydrogen sulphide, and proceed as above described.

*Hanson and Johnson*† heat a quantity of the material, containing about 25 grams of solids, with a mixture of 200 cc. of water, 100 cc. of concentrated nitric acid and 50 cc. of concentrated sulphuric acid, adding additional nitric acid from time to time and finally 25 grams of potassium sulphate.

*Baker Method*.‡—Treat 100 grams of the material with nitric and sulphuric acid as described in the preceding sections. Dilute the sulphuric acid residue, neutralize with ammonia, add hydrochloric acid until the solution contains about 2%, and thoroughly saturate with hydrogen sulphide gas. Filter the impure lead sulphide on a Gooch crucible with a false bottom, wash three or four times with water, then transfer precipitate and asbestos to a 300-cc. Erlenmeyer flask, washing with a little water, and boil with strong hydrochloric acid, adding potassium chlorate from time to time to insure complete solution of the tin sulphide as well as the elimination of the sulphur. Add a few strips of pure aluminum foil,

\* U. S. Dept. of Agric., Bur. of Chem., Bul. 137, p. 134.

† U. S. Dept. of Agric., Bur. of Chem., Bul. 152, p. 117.

‡ 8th Intern. Cong. Appl. Chem., 18, p. 35.



free from tin, until all the chlorine is eliminated, then dilute to from 30% to 40% acid strength and attach to a carbon dioxide generator provided with a scrubber and charged with pure marble and hydrochloric acid.

A bulb tube passing through one opening of a double-bore stopper serves to deliver the gas near the surface of the liquid and another bulb tube provides an exit, the latter being connected with a glass tube immersed in water to the depth of 20 cm., forming a water seal. When the flask is first attached to the carbon dioxide apparatus, lift the exit tube out of the water so as to reduce the pressure and thus force a large amount of gas through the system, expelling all air. Then raise the stopper of the flask and introduce about 1 gram of aluminum foil, which quickly reduces the tin to the metallic form with evolution of hydrogen.

Heat to boiling on a hot plate and boil for a few minutes, which causes the aluminum to disappear and changes the tin into stannous chloride, then cool in ice-water, still passing carbon dioxide through the system. Remove the stopper together with the tubes, washing the same and the sides of the flask with air-free water, prepared by boiling distilled water, adding a small amount of sodium bicarbonate and then a slight excess of hydrochloric acid.

Add starch paste and titrate directly and quickly with hundredth-normal iodine solution until a faint blue color is obtained. The iodine solution is standardized against pure tin solution or a food mixture, such as apple butter, containing an added amount of tin salt.

An alternate procedure is to add an excess of iodine solution to the flask after lifting the stopper, but while the carbon dioxide is still issuing from the neck, and titrate the excess with standard sodium thiosulphate solution.

By means of a Y-tube the current from one generator may be divided for two flasks so that duplicates may be conducted at the same time.

**Determination of Lead**, especially applicable if lead is present in small amounts only. Boil the sulphated ash of the contents of the can (obtained as on page 973) with a solution of ammonium acetate, having an excess of ammonia. The tin, zinc, and iron remain insoluble, while the copper and lead are dissolved. Filter, wash, and add a few drops of potassium cyanide to the filtrate, to prevent precipitation of copper when hydrogen sulphide is subsequently added. If the solution exceeds 40 cc., concentrate to that amount by evaporation, and transfer to a 50-cc. Nessler tube. Add hydrogen sulphide water, and make up to the mark. Compare the brown color imparted by the lead sulphide, with the colors obtained



by treating with hydrogen sulphide water in Nessler tubes various measured amounts of a standard solution of lead acetate, made alkaline with ammonia. See also page 362.

**Determination of Copper.**—(1) *Electrolytically.*—Ash the contents of the can as on page 973. Wet the ash with concentrated nitric acid, add water, and boil. Then make strongly alkaline with ammonia and filter. Unless the filtrate is colored blue, copper is absent. Transfer the filtrate to a bright tared platinum dish of 100-cc. capacity, neutralize with concentrated nitric acid, and add about 2 cc. in excess. Nearly fill the dish with water, and electrolyze with the apparatus described on page 634, using a current of about 0.3 of an ampere.

(2) *Colorimetrically.*—This method is especially applicable for small amounts of copper. The blue-colored ammoniacal solution of the ash, filtered as in (1), is transferred to a Nessler tube, and its color matched against the colors of a series of measured amounts of an ammoniacal standard solution of copper sulphate.

**Determination of Nickel.**—Boil the ash with water slightly acidified with hydrochloric acid, and without filtering, saturate with hydrogen sulphide, thus precipitating out any copper, tin, or lead. Filter and wash. Zinc and nickel, if present, are in the filtrate. Boil the filtrate to expel the hydrogen sulphide, and add sodium carbonate till slightly alkaline. Add acetic acid without filtering till the precipitate produced by the alkaline carbonate is dissolved, and then add a considerable excess of acetic acid. The zinc is precipitated by passing hydrogen sulphide through the cold dilute solution, while the nickel is held in solution by the large excess of acetic acid. Filter, and wash with hydrogen sulphide water, to which a little ammonium acetate has been added.

Make the filtrate alkaline with ammonia, precipitate the nickel with ammonium sulphide, filter, wash, ignite, and weigh as nickelous oxide.

#### KETCHUP.

**Standards.**—The following are the United States standards:

*Catchup* (*Ketchup*, *Catsup*) is the clean, sound product made from the properly prepared pulp of clean, sound, fresh, ripe tomatoes, with spices and with or without sugar and vinegar; *Mushroom Catchup*, *Walnut Catchup*, etc., are catchups made as above described, and conform in name to the substances used in their preparation.

No standard is given for *Chili Sauce*, a product made from tomatoes,



peppers, onions, vinegar, sugar, and spices, differing from ketchup in that it is not strained.

**Process of Manufacture.**—When made in the household ripe tomatoes, with or without paring and coring, are cut in pieces and boiled down to a thick pulp, strained to remove seeds and other coarse tissues and finally heated for a time with vinegar, spices, salt, and sugar. The product is bottled while hot.

Factory-made ketchup, of good quality, is prepared by practically the same process, using special apparatus for washing, pulping and concentrating. In many factories considerable time elapses before the finishing processes are carried out, the pulp being stored in barrels or better in lacquered tin receptacles until needed. Manufacturers of ketchup often purchase the barrelled or canned pulp from canning factories, confining their attention to the final processes and bottling.

In the so-called gravity process the pulped material is allowed to stand until fermentation sets in and the cellular matter rises to the surface. The clear liquid is then removed from below. In Italy it is a common practice in the manufacture of tomato paste to allow the pulp to ferment for a time, after which the fermentation is checked by the addition of salt.\*

**The Composition of Tomato Catsup** varies within wide limits due chiefly to variations in the composition of the tomatoes, the amount of fibrous material removed in screening, the degree of concentration, and the amount and composition of the substances added, particularly the vinegar. This is shown by the maximum and minimum results for total solids and acidity reported in commercial catsups by Winton and Ogden,† Street,‡ and McGill § as given in the following table:

	Total Solids.	Acidity Calc. as Acetic.
Winton and Ogden.....	42.64-6.03	2.20-0.60
Street.....	32.49-7.27	1.98-0.54
McGill.....	38.63-6.66	2.85-0.45

**Tomato Catsup from Trimmings.**—If instead of the pulp of the whole tomato the pulp of trimmings (skins, cores, etc.) from tomato canneries is

\* Daily Consular and Trade Reports, 14, 1911, p. 74.

† Conn. Agr. Exp. Sta. Rep., 1901, p. 135.

‡ Ibid., Rep., 1910, p. 521.

§ Lab. Int. Rev. Dept. Canada, Bul. 368, 1917.



used another complication is introduced. The presence of trimmings pulp obviously cannot be detected by mere determination of solids, even if no salt, sugar, or other additions were made, since the percentage of solids is dependent in large part on the degree of concentration. Bigelow and Fitzgerald\* calculated the ratio of pulp solids to filtrate solids and the percentage of insoluble solids in the total solids, in the case of whole tomato pulp and of trimmings pulp with the following results:

	Whole Tomato Pulp.	Trimmings Pulp.
Ratio of pulp solids to filtrate solids: Maximum.....	1.154*	1.241†
Minimum.....	1.091*	1.109†
Per cent of insoluble solids in total solids: Maximum.	17.4‡	14.7§
Minimum..	9.8‡	11.4§

\* 30 samples.

† 18 samples.

‡ 24 samples.

§ 7 samples.

These results do not sharply differentiate the two products and after the addition of the other constituents proper to tomato catsup the distinction would be still less marked or entirely absent.

The use of tomato trimmings for cheap catsup appears unobjectionable provided they are from sound tomatoes and have not been allowed to spoil. Too often the material is objectionable. This is especially the case when the product is improperly stored and too long a time elapses before its manufacture into catsup.

**Decayed Material.**—According to Bacon and Dunbar † fresh tomatoes contain on the average 6.5% total solids, of which 3.5% is invert sugar, 0.5% citric acid, 0.6% ash, 0.9% protein ( $N \times 6.25$ ), 0.85% crude fiber and 0.05% fat. During spoilage the sugars rapidly disappear, forming alcohol, carbon dioxide, acetic and lactic acids, the amounts of each formed depending on the organisms present. Usually the citric acid is also decomposed. A good ketchup is accordingly characterized by a high citric acid content and little lactic acid, while one made from decomposed material will usually contain little or no citric acid, but a high percent of lactic acid.

Bitting and Bitting ‡ state: "The procedure in determining wholesomeness is different from that for sterility, as in the former one must deal with

\* Jour. Ind. Eng. Chem., 7, 1915, p. 602.

† U. S. Dept. of Agric., Bur. of Chem., Circ. 78.

‡ Natl. Can. Assn. Res. Lab. Bul. 14, 1917.



the dead organisms, and is limited almost wholly to what may be seen under the microscope. It is most unfortunate that no satisfactory method has been developed to determine the presence of unfit material, as the purchaser has no means of judging from looks, taste, or smell what may have entered into these comminuted products. In the whole or large piece stock, he can judge by the gross appearance with a fair degree of accuracy. We are lacking in the fundamentals necessary for a proper examination: that is what constitutes the normal flora upon fully matured products, the abundance to which they attain under varying conditions, and what organisms cause the changes which are generally recognized as decomposition."

Howard in the examination of catsup under the federal law counts (1) rod-shaped bacteria (but ignores *micrococci*), (2) yeast cells and yeast and mold spores together (radically different bodies), and (3) number of fields containing mold filaments exceeding one-sixth the diameter of the field (i.e., determines the subdivision and distribution, rather than the amount of filaments). Details are described in government publications; \* criticisms are given by Bitting and Bitting,† Prescott, Burrage, and Philbrick,‡ and Tanner.§

**Foreign Pulp.**—Pumpkin pulp and apple sauce, the latter made often from unsound material or even pomace, have been extensively used in cheap ketchups. At the present time such compound sauces are usually labelled to show the constituents present.

**Preservatives.**—Salicylic acid, formerly used in most commercial ketchup, more recently has given place to benzoate of soda. Bitting || has shown that by using sound tomatoes and exercising proper care in the process of manufacture, ketchup can be kept without a preservative. Manufacturers themselves have corroborated this, many of the standard brands being entirely free from any antiseptic material other than spices and vinegar.

**Artificial Colors.**—Of ninety-four samples of ketchup examined in 1901 in Connecticut all but fifteen contained coal-tar colors.¶ This practice, however, is now decreasing and is indeed quite unnecessary if

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\* Howard and Stephenson, U. S. Dept. of Agric., Bul. 581, 1917; Howard, Bur. of Chem., Circ., 68, 1911.

† Natl. Can. Assoc. Res. Lab. Bul. 14, 1917.

‡ Abs. Bact., 1, 1917, p. 51.

§ Bacteriology and Mycology of Foods, New York, 1919, pp. 515, 519.

|| U. S. Dept. of Agric., Bur. of Chem., Bul. 119, 1909.

¶ Conn. Agr. Exp. Sta. Rep., 1901, p. 135.



fresh ripe tomatoes are used, dark-colored spices are avoided, and sugar is not added until the end of the process.

#### METHODS OF ANALYSIS.

The methods described under this head, except those for solids by calculation, may be used for tomato catsup, chili sauce, tomato pulp, fresh tomatoes, and canned tomatoes, provided the material is suitably sampled.

**Determination of Specific Gravity.**—*Bigelow and Fitzgerald*\* in order to remove air bubbles unavoidably introduced in pouring into the picrometer, centrifuge at 1000 revolutions per minute, add more of the material to volume, and repeat the centrifuging until there is no more contraction.

**Ash, Alkalinity of Ash, and Sodium Chloride** are determined by the methods described for jams and jellies (page 996). **Volatile Acids**, as acetic, is determined by the method described for vinegar (page 797). Tests for **Preservatives** and **Colors** are carried out as described in Chapters XV and XVI.

**Determination of Solids.**—Weigh 10 grams of the sample into a flat-bottomed metal dish 6 cm. in diameter, add water to distribute the material, evaporate to dryness, dry four hours at the temperature of boiling water, and weigh.

**Determination of Insoluble Solids.**†—Shake 20 grams of the material with hot water in a narrow cylinder, centrifuge and decant the clear liquid on a tared filter-paper and filter with the aid of suction. Repeat the operation several times, finally transferring the material to the paper. Finish the washing on the paper and dry at 100° C. to constant weight.

The filtering may be carried on to advantage on a Buchner funnel, using two or more tared filters, as the suction is liable to break a single layer.

**Determination of Sand.**†—Weigh 100 grams of the well-mixed sample into a 2- or 3-liter beaker, nearly fill the beaker with water, and mix the contents thoroughly. Allow to stand five minutes and decant the supernatant liquid into a second beaker. Refill the first with water and again mix the contents. After five minutes more decant the second beaker into a third, the first into the second, refill and again mix the first. Continue this operation, decanting from the third beaker into the sink until the

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\* Jour. Ind. Eng. Chem., 1915, 7, p. 602.

† U. S. Dept. of Agric., Bur. of Chem., Bul. 152, 1912.



lighter material is washed out from the ketchup. Then collect the sand from the three beakers into a tared Gooch crucible, dry, ignite, and weigh.

The method for the determination of ash insoluble in hydrochloric acid is not applicable to the determination of sand in tomato products, owing to the small amount and uneven distribution. Even when 100 grams are used the results are far from concordant.

**Determination of Soluble Solids.**—Subtract the percentage of insoluble solids from the percentage of total solids.

**Determination of Reducing Sugars.**—*Direct.*—Place 10 grams of the ketchup in a 100-cc. flask, add an excess of normal lead acetate, make up to the mark and filter. To the filtrate add powdered sodium sulphate or carbonate sufficient to precipitate the excess of lead and again filter. Determine the reducing powder of the filtrate by the Munson and Walker method (p. 622) and calculate as invert sugar.

*After Inversion.*—Mix 50 cc. of the solution, after clarifying and removal of the lead, as described in last paragraph, with 5 cc. of concentrated hydrochloric acid, invert in the usual manner (p. 611), nearly neutralize with sodium hydroxide and determine the reducing power as before inversion.

**Determination of Acidity.**—*Bigelow and Fitzgerald Method.\**—Dilute 20 grams of the material with at least 200 cc. of water, add 0.5 cc. of 1% phenolphthalein solution in alcohol, and titrate with N/10 sodium hydroxide solution. Add 1 cc. of N/10 hydrochloric acid, heat quickly to boiling, boil one minute to expel carbon dioxide, cool, and titrate back with the standard alkali.

One cc. of N/10 alkali is equivalent to 0.0064 gram of citric acid.

**Determination of Citric Acid.**—*Bacon and Dunbar Method.\**—Weigh 25 grams into a 250-cc. beaker, make up to approximately 200 cc. with 95 per cent alcohol, allow to stand with frequent stirring for four hours, filter through a folded filter and wash with 50 cc. of 80% alcohol. To the filtrate add sufficient water to dilute the alcohol to 50% or 60% and then add 10 cc. of 20% barium acetate solution, stir well with a glass rod, and allow to stand overnight. In the morning filter on a Gooch crucible, washing with 50% alcohol, dry for from three to four hours in an oven at 100° C. and weigh. Weight of precipitate times 0.51 equals anhydrous citric acid. This method is not applicable in the presence of malic acid, hence if apple pulp is a constituent of the ketchup, the Pratt method (page 1009), should be employed.

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\* Loc. cit.



**Determination of Lactic Acid.**—*Bacon and Dunbar Method.\**—To 100 grams of ketchup add 10 cc. of 20% normal lead acetate solution, make up to 500 cc., shake well and centrifuge. To 400 cc. of the clear portion add a moderate excess of sulphuric acid, filter, wash the precipitate with a small amount of water, and evaporate the filtrate on the steam bath to about 100 cc. Extract for from eighteen to twenty hours in a liquid extractor (Fig. 120) with washed ether. In case the quantity of lactic acid present is greater than 0.5 gram it is usually necessary to extract for a longer period. In any case it is well to re-extract for from eight to ten hours to make sure that the extraction is complete. Ether sufficiently pure for this purpose may be prepared by shaking out ordinary ether once with a sodium hydroxide solution and then ten times with small quantities of water. Evaporate on a steam bath until the ether is no longer evident, and take up the residue at once in water and filter, thus removing a small amount of coloring matter and substances other than lactic acid, which may be extracted from ketchup by ether, but which are insoluble in water. Heat the filtrate on the steam bath for some time to remove all traces of ether or alcohol. Add approximately 3 grams of sodium hydroxide and 50 cc. of a 1.5% solution of potassium permanganate from a pipette. Heat on a water-bath at 100° C. for one-half hour. At the end of that time, or before, if the color is not a decided blue-black or purple, but is green or colorless above the layer of brown precipitate, add more standard permanganate until, after heating one-half hour on a boiling water-bath, the color is a blue-black or purple. The oxidation is then complete. Make the hot solution strongly acid with 10% sulphuric acid (about 50 cc.) and run in 5% standard oxalic acid from a burette until the solution is decolorized. Titrate back any slight excess of oxalic acid with the standard permanganate solution.

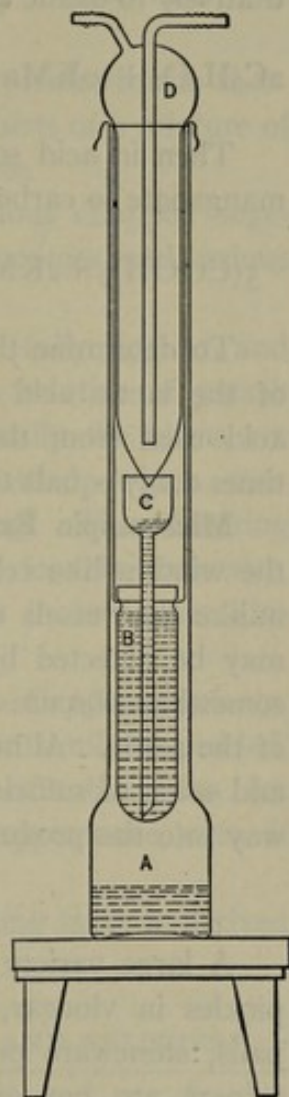


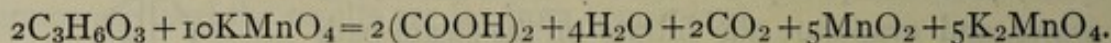
FIG. 120. — Bacon and Dunbar Extractor for Liquids. A, jacket-flash; B, extract-tube; C, funnel-tube; D, condenser.

\* Loc cit.

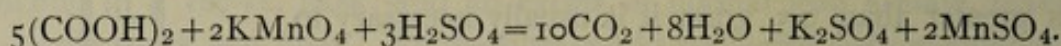


(Any standard permanganate and oxalic acid solution may be used within reasonable limits of strength.)

In alkaline solution the permanganate oxidizes the lactic acid quantitatively to oxalic acid according to the equation:



Then in acid solution, the oxalic acid is further oxidized by the permanganate to carbon dioxide and water according to the equation:



To determine the total weight of permanganate used in the oxidation of the lactic acid subtract the permanganate equivalent of the oxalic acid used from the total amount used. The weight of permanganate times 0.237 equals the weight of lactic acid.

**Microscopic Examination for Foreign Pulp.**—Apple is identified by the window-like cells of the skin, the pitted vessels of the bundles, quite unlike the vessels of the tomato, and the tissues of the core. Pumpkin may be detected by the yellow skin of the fruit with colorless stomata, somewhat obscure latex tubes and the peculiar cactus-like parenchyma of the seeds. Although only the fruit pulp is used, fragments of the skin and seeds of sufficient size to be of diagnostic importance often find their way into the product.

#### PICKLES.

A large variety of vegetables and fruits are preserved in the form of pickles in vinegar, either with or without spices, and kept in wooden pails, stoneware pots, kegs, or sealed wide-mouthed bottles. The containers are not of necessity air-tight. The commoner vegetables are usually pickled without cooking, while fruits such as peaches, pears, gooseberries, etc., are usually cooked, or at least heated. Analyses of pickles and relishes appear in the table, page 985.

*Cucumber Pickles* are the most common, and are prepared by soaking the fresh cucumbers in strong salt brine. They are then dried on frames, and afterwards treated with boiling vinegar, to which spices may or may not be added. Other vegetables pickled in similar manner, either separately or in mixture with cucumbers or "gherkins" to form "mixed pickles," are cauliflower, bean pods, white cabbage, young walnuts, and onions.



Such soft vegetables as young podded beans and beets are not treated with brine, but, after soaking in water, are directly treated with vinegar. The vinegar used for the finest pickling is of the cider, wine, or malt variety. Cheaper varieties of pickles are put up in "white wine" or spirit vinegar.

*Mustard Pickles.*—These differ from plain vinegar pickles in the character of the preserving medium, which in this case consists of a mixture of mustard and spices with the vinegar to form a thin paste.

*Piccalilli* consists of a mixture in vinegar of various chopped vegetables, such as cucumbers, cauliflower, onions, green tomatoes, and various spices.

*Olives* for pickling are picked before they have fully ripened, and the inherent bitter taste is removed by soaking in a solution of potash and lime. This is replaced by cold water, and finally the olives are transferred to the medium in which they are bottled, which consists of salt brine, either with or without flavoring. The flavoring materials employed consist of such substances as fennel, coriander, laurel leaves, and occasionally vinegar. *Ripe olives* in brine are also highly esteemed.

*Capers.*—These are the flower buds of the shrub *Capparis spinosa*, which are pickled in vinegar. Nasturtium seeds, when similarly pickled, possess a flavor much resembling capers, but their substitution for capers could readily be detected by their distinctive appearance, even if colored.

**Composition of Pickles and Relishes.**—The following table is derived from Atwater and Bryant's compilation:

CHEMICAL COMPOSITION OF KETCHUP, PICKLES, AND RELISHES.\*

	Number of Analyses	Refuse.	Water.	Protein.	Fat.	Total Carbo- hydrates	Ash.	Fuel Value per Pound.
Tomato ketchup.....	2	.....	82.8	1.5	.2	12.3	3.2	265
Horseradish.....	2	.....	86.4	1.4	.2	10.5	1.5	230
Olives, green:								
Edible portion.....	1	.....	58.0	1.1	27.6	11.6	1.7	1,400
As purchased.....	1	27.0	42.3	.8	20.2	8.5	1.2	1,025
Olives, ripe:								
Edible portion.....	1	.....	64.7	1.7	25.9	4.3	3.4	1,205
As purchased.....	1	19.0	52.4	1.4	21.0	3.5	2.7	975
Cucumber pickles.....	3	.....	92.9	.5	.3	2.7	3.6	70
Mixed pickles.....	1	.....	93.8	1.1	.4	4.0	.7	110
Spiced pickles.....	1	.....	77.1	.4	.1	20.7	1.7	395

\* U. S. Dept. of Agric., Office of Exp. Sta., Bul. 28, p. 70.



**Adulteration.**—Pickles were formerly greened in the household by the use of copper kettles and in the factory by the addition of copper sulphate.

For methods of detection and estimation of copper, see page 634. Pickles may be greened by boiling with much less harmful substances than copper salts, such, for example, as grape leaves, spinach, or parsley.

*Free Sulphuric Acid* has been found in a number of cases in the vinegar of pickles bought on the Massachusetts market. A pronounced test for chloride with nitrate of silver should not be attributed to free hydrochloric acid, since it may be due to the salt from the brine in which the pickles have been treated.

*Alum* is sometimes added to the salt solution to produce hardness and crispness in pickles. A number of samples of cucumber pickles have been found by the author to contain alum. For its detection, fuse the ash of the pickles, if free from copper, in a platinum dish with sodium carbonate, extract with boiling water, filter, and add ammonium chloride. A flocculent precipitate shows alum.

*Sodium Benzoate* and *Saccharin* have been frequently detected in sweet pickles.

**Horseradish.**—This condiment is prepared by grating the root of the perennial herb *Nasturtium armorica*, and preserving in vinegar. It is very pungent and aromatic when first prepared, but by exposure to light and air quickly loses strength. Turnip pulp is used as an adulterant.

#### PRESERVES.

Under this head are included various fruit products prepared with sugar syrup and often also with spices and vinegar. Some of these products differ little from canned fruits while others are really sweet pickles. Mince meat, although not strictly a fruit product, and fruits in cordials are classified for convenience as preserves. Jams are considered with jellies in the next section, as are also methods of analysis.

**Fruit Butter.**—According to the U. S. Standard, "fruit butter is the sound product made from fruit juice and clean, sound, properly matured and prepared fruit, evaporated to a semi-solid mass of homogeneous consistence, with or without the addition of sugar and spices or vinegar, and conforms in name to the fruit used in its preparation."

*Apple Butter* is the best-known product of this class. Unfortunately it is sometimes made from decayed fruit or even from apple pomace. Glucose is frequently substituted wholly or in part for sugar, in which case its presence should be declared on the label.



**Mince Meat.**—As prepared in the household, mince meat, the filling for mince pies, contains from 10 to 20% of lean meat and about twice as much apple. Other constituents appear in the following typical formula with statement of quantities in parts by weight: 2 parts each of meat, raisins, dried currants, and sugar, 4 parts of apples, 1 part each of suet and candied citron, 2 parts of sweet cider, wine or brandy, 1 to 2 parts of seasoning including salt, spices, and lemons or oranges.

*Standard Mince Meat* of the A.O.A.C. and the Association of State and National Dairy and Food Departments, "is a mixture of not less than 10% of cooked comminuted meat, with chopped suet, apple and other fruit, salt, and spices, and with sugar, syrup, or molasses, and with or without vinegar, fresh, concentrated, or fermented fruit juices, or spirituous liquors."

*Adulteration.*—There has been some conflict between food officials and certain manufacturers as to the proportion of meat in commercial mince meat, the manufacturers claiming that 10% is too much for the proper keeping of the product, the food officials, on the other hand, contending that the manufacturer has no right to lower the recognized standard of the housewife.

As a matter of fact the greater part of the mince meat on the market contains considerably less than 10% of meat and much of it none whatever. Glucose is a common substitute for part of the sugar, wormy or other inferior fruit is sometimes used, and benzoate of soda is added as a preservative.

*Condensed Mince Meat* is made in a commercial way from dried apples and other desiccated materials and is sold in compressed cakes with instructions for preparing from the cakes moist pie filling. As in the case of wet mince meat, glucose, wormy fruit and benzoate of soda are frequent admixtures and true meat is often omitted entirely. Wheat or rye flour is a common adulterant.

*Examination of Mince Meat.*—Meat and cereal flour may be identified by microscopic examination. Care should be taken not to confuse apple starch, which is always present in the immature fruit, with cereal starches. Meat fibers are recognized by their yellow brown color, the delicate transverse striations and their occurrence in bundles.

Determinations of nitrogen are of service in estimating the amount of meat present. Glucose and sugar are calculated from the polarization readings.

**Pie Filling.** Bakers and hotel cooks are supplied by manufacturers with filling prepared ready for use in pies. This material is shipped in pails or



tubs preserved with benzoate of soda, and may contain fruit of questionable quality as well as admixtures such as starch, glucose, and artificial colors.

**Maraschino Cherries.**—This name has been applied indiscriminately to the vivid red preserved cherries used in cocktails, punches, ice cream and confectionery. Investigation by the Board of Food and Drug Inspection has led to the decision \* that only marasca cherries, preserved in true maraschino cordial prepared by fermentation and distillation from marasca cherries, are entitled to the name maraschino cherries, although cherries of other types preserved in pure maraschino cordial may be labelled: "Cherries in Maraschino." Ordinary cherries preserved in syrup flavored with maraschino may be so labelled, but if the flavoring is oil of bitter almonds or benzaldehyde the product should be labelled as an imitation if the word maraschino is used.

Enormous quantities of white cherries of the Bigarreau or Royal Anne type, preserved in a mixture of sulphurous acid and brine, are brought into the United States from Europe and transformed into red "Maraschino cherries" or green "Crème de menthe cherries." After removal of the sulphurous acid and brine the cherries are put through a dye bath and then, being quite without taste, are flavored with oil of bitter almond or benzaldehyde, or else peppermint, and packed in syrup. Scarcely more than the cellular structure of the original cherry remains, the fruit juice with its sugars, acids, and true cherry flavor being replaced by the syrup with its sickening flavor and aroma. Even if flavored with true maraschino the metamorphoses through which the fruit passes leave it a sorry substitute for the natural cherry.

Woodman and Davis † have shown that true maraschino contains very little benzaldehyde and that cherries flavored with maraschino should not contain more than two or three times as many milligrams of benzaldehyde per 100 cc. as there are grams of alcohol in that volume, and those containing over 20 mg. of benzaldehyde but no alcohol are evidently entirely artificial.

Artificial colors, sulphurous acid and other preservatives are detected by the methods given in the chapters on colors and preservatives, benzaldehyde by the following method:

**Determination of Benzaldehyde in Maraschino Cherries.**—*Woodman and Davis Method.* †—*Reagent.*—Mix 3 cc. of glacial acetic acid with

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\* Food Inspection Decision 141.

† Jour. Ind. Eng. Chem., 4, 1912, p. 588.



40 cc. of water, add 2 cc. of C.P. phenyl hydrazine, as near colorless as possible, shake thoroughly, and filter the emulsion through several thicknesses of filter-paper. The clear filtrate should be used immediately as a turbidity appears on standing longer than five minutes.

*Process.*—Dilute 100 cc. of the liquor from maraschino cherries (or 50 cc. of maraschino liqueur) to 140 cc. and distill off 110 cc. Determine approximately the alcohol in the distillate by the pycnometer or immersion refractometer, then without delay transfer 100 cc. to a 300 cc. Erlenmeyer flask and add alcohol or water so that the solution shall contain approximately 10% of alcohol. Add 10 cc. of the reagent, stopper tightly with a rubber stopper, and shake vigorously for ten minutes. Collect the precipitate in a tared Gooch crucible, wash with cold water and finally with about 10 cc. of 10% alcohol. Dry in a vacuum desiccator for 20–24 hours at about 20 cm. pressure, or in a vacuum oven at 70–80° C. for 3 hours. Throughout the process avoid exposure of the precipitate to strong light.

Run a blank determination at the same time and deduct the weight obtained from that found in the actual analysis. Multiply the corrected weight of the precipitate by 0.5411, thus obtaining the weight of benzaldehyde.

### JAMS AND JELLIES.

Jams or marmalades are prepared from the pulp of fruits, and jellies from the fruit juices. Both jams and jellies, to be considered of the highest degree of purity, should contain nothing but the fruit pulp or juice named on the label, mixed with pure cane sugar, and, in the case of jams, the further addition of spices and flavoring materials is permissible.

For the manufacture of jam, apples, quinces, and pears are peeled, freed from cores, and sliced; berries are simply stemmed; and stone fruits, such as peaches and apricots, are peeled, and freed from stones. The material, properly prepared, is cooked with as much water as is necessary for boiling, and with the addition of an amount of sugar varying with different manufacturers. Some prefer to use equal parts of sugar and fruit, others one part sugar to two parts fruit.

In the case of jelly, the fruit is cooked in a small amount of water till soft, transferred to a bag or press, and the juice allowed to flow out spontaneously, or is squeezed out under pressure, according to the grade of jelly desired, the clearest and finest varieties being made from the juice that flows out naturally. This juice is then evaporated down with the addition of sugar to a density of from 30° to 32° Bé., which is of the



proper consistency to form a perfect jelly product after cooling, and, while still hot, is poured into the tumblers in which it is to be kept. Here, as in the case of jams, the amount of sugar varies, some using pound for pound, and others only half as much sugar as fruit. Some manufacturers clarify their jellies by mixing with the juice, while boiling, elutriated chalk, using a teaspoonful to each quart of juice. The impurities come to the surface with the chalk as a scum, and are skimmed off. This clarifying process is somewhat analogous to the defecation of sugar juices with lime, and is commonly carried out with apple jelly.

The "jellying" or gelatinizing of the final product is due to the presence in the fruit juice of pectin, or so-called vegetable jelly ( $C_{32}H_{40}O_{284}H_2O$ ), formed by the hydrolysis of pectose.

The high content of added sugar in jelly, once thought to be essential for keeping it, is now no longer considered necessary, and much less sugar is at present added than formerly. The finest grade of apple jelly, for instance, is made without any added sugar whatever.

In making the better grades of apple jelly, apple juice fresh from the press is run directly into the boiler or evaporator before any fermentation has ensued, and gelatinized by concentration. If boiled cider is wanted instead of jelly, it is drawn off at an earlier stage than in the case of apple jelly.

**Composition of Known-purity Jellies and Jams.**—In the tables on pages 992 and 993, due to Tolman, Munson, and Bigelow,\* are given results reached in the examination of the pure finished products, as well as of pure fruit juices and pulp used in their manufacture.

**Imitation Jams and Jellies.**—Only a small percentage of the products sold in the United States belong in the same class with home-made jams and jellies consisting exclusively of the fruits in mixture with cane sugar. The cheap substitutes are made up largely of apple juice and commercial glucose, sometimes containing no fruit whatever of the kind specified on the label. Sometimes an attempt is made to imitate the flavor by the addition of artificial fruit essences, but more often the same apple-glucose stock mixture of jelly, put out under a particular brand, serves to masquerade as damson, strawberry, raspberry, currant, grape, etc., differing from each other only in color, but not as a rule in flavor. A variety of artificial colors are employed, mostly coal-tar dyes. To compensate for the lack of sweetness of the glucose, a minute quantity of one of the concentrated

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\* Jour. Amer. Chem. Soc., 23, 1901, p. 349.



sweeteners, such as saccharin or dulcin, is sometimes added. Besides artificial colors, antiseptic substances are occasionally used, especially sodium benzoate.

All grades of apple stock are found in these preparations. A large source of supply is furnished by the parings and cores of canning establishments, to say nothing of the refuse of these factories, such materials being boiled with water, and the extract, variously colored to imitate the different fruits, being evaporated with commercial glucose.

*Imitation Jellies.*—While it is easy to make an excellent apple jelly by simple evaporation of the pure apple juice, even without the addition of sugar, it is impossible, or at least difficult, to obtain the proper degree of stiffness with a mixture of apple stock and commercial glucose. It is customary, in the manufacture of cheap jellies, therefore, to employ what is technically termed a "coagulator." Formerly sulphuric acid, sometimes with addition of alum was used, but at present phosphoric acid is preferred. Citric or tartaric acid is also used for this purpose, as well as to increase the acidity. Less than 1% of acid will cause the mass to gelatinize satisfactorily.

The lowest grade of apple jelly is made from the exhausted pomace, left as a residue after pressing out the juice for cider. Such stock is commonly mixed with water, and boiled down with glucose. Having been exhausted of its malic acid, pectose, and other soluble constituents, it lacks much of the flavor inherent in pure apple jelly. Various foreign gelatinizing agents are found in cheap jellies and preserves, such as starch, gelatin, and agar-agar. In the low-priced goods, starch paste has been employed. It should be remembered that starch exists in unripe apples, but hardly at all in the mature fruit, so that while mere traces of starch in jelly may be due to the use of green apples, its presence in large amounts is undoubted evidence of the admixture of starch paste.

*Imitation Jams.*—Most of the cheap jams and bottled preserves sold on the market, though reinforced with apple stock, do in reality contain masses of fruit and berries of the kind stipulated on the label, as even a casual megascopic examination will show. That such low-priced preparations really contain genuine fruit pulp is not to be wondered at, when it is considered that much of the virtue of this fruit has sometimes been previously extracted by boiling, to produce fruit juices for higher-priced goods. Or, as in the case of jams containing strawberries, raspberries, and other small fruits with seeds, the juice is apt to have been previously expressed for pure jellies, while the residues are afterwards



TABLE I.—JUICE.\*

Description of Sample.	Total Solids, Per Cent.	Ash, Per Cent.	Total Acids Calculated as H <sub>2</sub> SO <sub>4</sub> , Per Cent.	Proteids (N×6.25), Per Cent.	Sugars.				Polarization.		
					Reducing Sugar, Per Cent.	Cane Sugar Added, Per Cent.	Cane Sugar Found, Per Cent.	Added Cane Sugar Inverted, Per Cent.	Direct at 18° C.	Invert at 18° C.	Invert at 80° C.
Apple (fall pippin).....	7.95	0.47	0.627	0.543	4.00	.....	1.18	.....	-3.0	-4.6	-2.9
Blackberry.....	8.54	0.52	0.978	0.350	4.34	.....	0.00	.....	-1.5	-1.6	-1.0
Crab apple.....	5.62	0.20	0.372	0.075	2.56	.....	1.03	.....	-1.0	-2.4	-1.1
Grape (fox).....	6.67	0.49	1.686	.....	2.79	.....	0.37	.....	-0.8	-1.3	-1.1
Grape (Ives seedling).....	8.83	0.57	0.902	0.237	5.10	.....	0.89	.....	-1.2	-2.4	-0.6
Huckleberry.....	16.33	0.40	0.454	.....	11.21	.....	0.89	.....	-3.2	-4.4	-0.9
Orange (Florida navel).....	6.08	0.36	0.297	0.581	1.52	.....	2.29	.....	+1.8	-1.3	0.0
Peach.....	8.90	0.45	.....	0.218	.....	.....	4.59	.....	+4.0	-2.2	-0.7
Pear (Bartlett).....	11.65	0.45	0.345	0.087	5.87	.....	1.18	.....	-4.8	-6.4	-4.0
Pineapple.....	13.27	0.45	0.588	0.368	2.74	.....	8.96	.....	+8.4	-3.7	-1.1
Pineapple-husk juice.....	8.43	0.77	.....	0.350	.....	.....	4.73	.....	+4.1	-2.3	-0.7
Plum (Damsen).....	12.72	0.63	.....	0.431	4.86	.....	0.51	.....	+2.0	+1.3	+2.4
Plum (wild fox).....	11.23	0.64	1.576	0.137	2.87	.....	2.81	.....	+1.4	-2.4	-1.8
Mixed fruit.....	6.53	0.32	0.612	0.150	2.68	.....	0.59	.....	-1.0	-1.8	-0.9

TABLE II.—JELLY.

Apple (fall pippin).....	59.18	0.22	0.279	0.175	20.78	51.76	33.04	36.17	+24.0	-20.6	-1.2
Blackberry.....	59.63	0.33	0.475	0.243	12.51	54.89	44.90	18.20	+47.0	-20.1	0.0
Crab apple.....	63.28	0.11	0.171	0.137	34.93	57.61	23.68	58.88	+13.0	-19.0	0.0
Grape (Ives seedling).....	63.66	0.45	0.524	0.175	32.29	60.29	30.52	49.33	+22.3	-18.9	+0.2
Huckleberry.....	63.02	0.28	0.245	0.069	24.27	53.39	32.74	37.54	+24.1	-20.1	-0.4
Orange (Florida navel).....	68.56	0.30	0.171	0.418	3.95	65.59	62.52	4.91	+61.3	-23.1	-0.2
Peach.....	69.98	0.21	0.245	0.175	8.75	63.70	56.59	11.16	+53.4	-23.0	-0.6
Pear (Bartlett).....	69.12	0.34	0.181	0.156	6.58	63.09	58.46	7.33	+52.7	-26.2	-1.8
Pineapple.....	80.28	0.43	0.328	0.387	22.13	72.98	56.70	28.45	+50.4	-26.1	0.0
Pineapple-husk.....	76.34	0.73	0.352	0.350	7.40	70.22	65.22	7.12	+63.7	-24.3	-0.6
Plum (Damsen).....	45.56	0.68	1.127	0.350	19.18	38.00	22.67	40.38	+17.8	-12.8	0.0
Plum (wild fox).....	54.49	0.40	1.029	1.138	24.00	48.05	25.48	46.97	+16.7	-17.8	0.0
Plum (wild fox) boiled down.....	73.01	0.65	1.529	0.175	44.22	64.66	22.37	66.18	+7.6	-22.6	-0.6
Mixed fruit.....	66.58	0.21	0.367	0.069	39.70	59.72	24.22	40.38	+14.8	-17.9	+2.2

\* The "juice" was prepared by cooking the fruit till soft, after the addition of sufficient water to prevent scorching, and straining through a jelly bag.



TABLE III.—FRUIT.\*

Description of Sample.	Total Solids, Per Cent.	Ash, Per Cent.	Total Acids Calculated as H <sub>2</sub> SO <sub>4</sub> , Per Cent.	Proteids (N×6.25), Per Cent.	Sugars.				Polarization.		
					Reducing Sugars, Per Cent.	Cane Sugar Added, Per Cent.	Cane Sugar Found, Per Cent.	Added Cane Sugar Inverted, Per Cent.	Direct at 18° C.	Invert at 18° C.	Invert at 86° C.
Apple (fall pippin).....	8.25	0.30	0.499	.....	4.13	.....	1.03	.....	-2.2	-3.6	-1.8
Blackberry.....	9.62	0.60	0.916	0.725	5.67	.....	.....	.....	-1.1	-1.0	0.0
Crab apple.....	14.34	0.84	0.705	0.418	5.68	.....	1.70	.....	-0.7	-3.0	-0.8
Grape (Ives seedling).....	12.50	0.75	.....	.....	6.11	.....	0.29	.....	-1.6	-2.0	0.0
Orange (Florida navel).....	13.11	0.61	0.686	0.985	4.13	.....	3.33	.....	+2.0	-2.5	-0.8
Pineapple.....	13.71	0.50	0.392	0.056	8.12	.....	3.11	.....	+1.0	-3.2	-0.0

TABLE IV.—JAM.

Apple (fall pippin).....	63.22	0.20	0.282	0.175	25.52	51.31	29.11	43.22	+26.3	-13.0	+4.8
Blackberry.....	55.42	0.48	0.851	0.737	18.77	43.99	29.00	34.08	+24.6	-14.6	+1.6
Crab apple.....	41.82	0.27	0.715	0.493	14.80	36.09	23.04	35.46	+18.3	-12.8	-0.6
Grape (fox).....	61.80	0.19	0.698	0.200	50.06	54.21	3.70	92.96	-9.0	-14.0	+2.2
Grape (Ives seedling).....	56.64	0.48	0.744	0.525	33.44	42.45	11.33	73.38	+3.5	-11.8	0.0
Orange (Florida navel).....	80.52	0.44	0.433	0.944	13.61	69.13	54.23	21.55	+55.9	-17.5	+2.0
Pear (Bartlett).....	61.52	0.28	0.163	0.312	13.20	46.52	33.74	18.87	+32.3	-13.2	+1.0
Pineapple marmalade.....	73.92	0.30	0.315	0.312	14.05	60.20	46.40	22.90	+52.3	-10.3	+6.2
Plum (Damson).....	50.43	0.54	1.012	0.525	28.29	37.75	9.70	74.42	+3.1	-10.0	+1.2
Plum (wild fox).....	62.10	0.46	1.355	0.212	28.78	47.86	23.26	53.43	+13.9	-17.5	0.0

\* The composition here given is not that of the original fruit, but of the pulped mass used in the preparation of jams.



worked up with apple stock for low-priced jams. Hence the presence of pure fruit stock, or genuine berry seeds and pulp in jams, is in itself no criterion of purity, and furthermore, it is unnecessary to use hay seed and other alleged foreign seeds as adulterants of cheap jam.

**Compound Goods.**—Many states have a law legalizing the sale of "compound" goods, providing they are distinctly so labeled. In other states, as, for instance, Massachusetts, the label must plainly state the name and percentage of the ingredients. In either case the analyst must discriminate, in classifying the inferior or low-grade preparations, between those that are labeled in accordance with the law, and those that are not. Only those not properly labeled can in such cases be classed as adulterated within the meaning of the law. Where such a law prevails, probably no class of food-products is so extensively affected by it as the low-grade jams, preserves, and jellies.

The restrictions as to labeling do not in all cases eliminate the element of deception. It is hardly justifiable, for example, to boldly label an alleged "currant jelly" which contains no currant, in the following manner:

Fruit juice.....	25%
Cane sugar.....	14%
Corn syrup.....	61%
	<hr/>
	100%

The use of the term "fruit juice" surely implies to the unsuspecting purchaser that so much pure currant juice has entered into the jelly, elsewhere labeled in large letters "Currant," whereas all the juice is apple, and no currant juice has been used.

The following label is a type of those which discriminate between pure fruit and apple juice:

Fruit.....	30%
Corn syrup.....	35%
Granulated sugar.....	15%
Apple juice.....	20%
	<hr/>
	100%

**Composition of Cheaper Grades.**—Out of 66 samples of jellies, jams, and preserves analyzed by Winton, Langley, and Ogden in Connecticut, the samples being purchased in that state,\* 17 samples contained starch

\* An. Rep. Conn. Exp. Sta., 1901, p. 130.



paste, 35 were artificially colored with coal-tar dyes, and 19 contained salicylic or benzoic acid.

The following table has been compiled, showing the sugar content of some of the typical commercial jellies and jams analyzed in the laboratory of the Massachusetts State Board of Health. Nearly all of these were artificially colored, and found to contain little if any fruit, other than apple.

	Direct Polariza- tion.	Invert Polarization.		Per Cent Sucrose.	Per Cent Commer- cial Glucose
		At 20° C.	At 87° C.		
JELLY.					
Apple. ....	+64.0	+28.0	+36.0	26.8	22.1
Currant A. ....	+29.2	+20.0	+36.4	6.9	22.3
"    B. ....	+41.6	+33.9	+40.8	5.7	25.0
Grape. ....	+62.0	+34.4	+46.0	20.6	28.2
Peach. ....	+119.8	+108.8	+110.0	8.2	67.4
Pineapple ....	+114.0	+107.6	+110.0	4.9	67.4
Raspberry. ....	+112.0	+92.0	+93.6	14.9	57.4
JAM.					
Damson A. ....	+107.0	+94.4	+58.1	9.3	35.6
"    B. ....	+95.2	+90.9	+83.6	3.2	51.2
Apricot. ....	+99.0	+93.5	+85.6	4.1	52.4
Quince. ....	+49.6	+43.6	+42.0	4.5	25.7
Raspberry A. ....	+123.6	+119.2	+102.5	2.6	62.8
"    B. ....	+77.6	+65.1	+46.9	9.3	28.7
"    C. ....	+66.0	+29.5	+37.2	27.2	22.8
Pineapple ....	+119.8	+108.8	+110.0	8.2	67.4
Strawberry A. ....	+41.8	+21.3	+32.6	15.4	20.0
"    B. ....	+83.6	+72.0	+78.8	8.7	48.3

#### METHODS OF ANALYSIS.

As in the case of canned goods, but little information is to be derived as to adulteration of jams, jellies, and preserves by the ordinary determinations of moisture, ash, and nitrogen, and these are rarely made by the public analyst.

Of considerable importance in this regard, however, are the sugar determinations, made with a view to ascertaining the varieties of sugar employed, as well as their approximate proportion in the products examined. Still more important are the results of tests for preservatives, dyes, foreign gelatinous substances, and mineral acids used as coagulators.

**Preparation of the Sample.**—In the case of jams, marmalades, and preserves, separate and weigh the stones, if present, then thoroughly pulp the sample. In the case of jellies rub the sample through a sieve. Stir well before weighing out the portions for analysis.



**Determination of Total Solids.**—Weigh 4 to 5 grams of the sample into a large flat-bottomed dish (preferably of platinum) containing from 4 to 5 grams of ignited asbestos and add enough water to uniformly distribute the material. Evaporate to dryness and dry for from twenty to twenty-four hours in a boiling water-oven.

The results by this method are not strictly accurate owing to the dehydration of levulose, but for practical purposes they are sufficiently close. If extreme accuracy is required dry *in vacuo* at 70° or in a McGill oven (page 609).

The solids in a jelly may also be calculated from the specific gravity.

**Determination of Ash.**—Burn the residue from the determination of solids, or else a new portion, in a platinum dish at dull redness.

*Alkalinity of ash* is determined as described for insoluble ash in maple products (page 657).

*Chlorides and Sulphates* are detected in the ash by the usual tests. If the portion used for determination of alkalinity is also to be used for the chlorine test the titration must be made with fifth-normal nitric acid.

The presence of chlorides is an indication of glucose, as pure fruit products do not contain appreciable amounts of chlorine compounds.

**Determination of Insoluble Solids.**—*Kremla Method*.<sup>\*</sup>—Thoroughly macerate 50 grams of the sample in a mortar with warm water, then transfer to a filter and wash thoroughly with warm water, stirring well after each addition. Wash up to 500 cc., or in extreme cases up to 1000 cc., remove the insoluble solids to a dish, dry in a boiling water-oven, and weigh. Kremla employed a coarse filter paper for collecting the insoluble solids; Munson and Tolman † found muslin more satisfactory.

Reserve the filtrate for determinations of soluble constituents.

*German Official Method*.<sup>‡</sup>—Transfer a weighed portion of the sample to a graduated flask, add water, shake thoroughly and make up to volume. Allow to settle and either filter or decant off the supernatant liquid. Determine the soluble solids by evaporating and drying an aliquot. The insoluble solids are obtained by subtracting the soluble from the total solids.

**Determination of Acidity.**—Dilute an aliquot of the solution from the insoluble solids of a jam or of a solution of a jelly and titrate with standard alkali. Use phenolphthalein as indicator if the color of the

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<sup>\*</sup> Zeits. Nahr. Hyg. Waar., 6, 1892, p. 483.

<sup>†</sup> U. S. Dept. of Agric., Bur. of Chem., Bul. 65, 1902, p. 76; Bul. 66 rev., p. 13.

<sup>‡</sup> Vereinb. Unters. Beurt. Nahr. Genussm. deutsch. Reich., 2, p. 105.



solution will permit, otherwise use litmus paper. Calculate the result as sulphuric acid or as the organic acid known to predominate (see page 1008).

Some of the methods for the determination of individual acids in fruit juices (pages 1008 and 1009) are applicable to jams and jellies, but the analyst will do well to test their accuracy on mixtures of known composition, especially if substances other than fruit and sugar are present.

**Determination of Protein.**—Determine nitrogen in 5 grams of the material by the Kjeldahl or Gunning method and calculate protein, using the factor 6.25.

**Determination of Sugars.**—In products of the highest grade, wherein only cane sugar is employed, a large portion of the cane sugar is inverted in the process of boiling the jam or jelly, so that when the analyst examines it, he finds, as a rule, only a small amount of sucrose, and considerable invert sugar. The amount of cane sugar equivalent to the invert sugar may be calculated if this is thought desirable. It is further of interest to calculate, at least approximately, the percentage of commercial glucose, when present, especially in cases where the package contains a formula setting forth the amount of the various ingredients used. In such cases the analyst is naturally called upon to verify the formula, since a wide variation in percentage composition from the statement on the label would constitute an offense under same state laws.

**Polarization.**—Use half the normal weight of the preserve or jelly for the Schmidt and Haensch instrument, namely 13 grams in 100 cc. If fresh fruit or fruit juice is to be examined, use the full normal weight, 26 grams. Clarify, before making up to the mark, with subacetate of lead and alumina cream (using 2 to 3 cc. of each clarifier), filter, and obtain the direct reading; then invert in the usual manner, and obtain the invert readings at 20° C., and in the water-jacketed tube at 87° C., proceeding in detail as directed under honey, page 671.

**Calculation of Sugars.**—*Sucrose* is determined by using the Clerget-Herzfeld formula:

$$S = \frac{(a - b)100}{142.66 - \frac{t}{2}}, \quad \dots \dots \dots (1)$$

This represents the sucrose actually present as such in the preserve or jelly, and not the amount originally used. If the latter ( $S'$ ) is desired, it may be roughly calculated by the following formula:



$$S' = \frac{100b}{42.66 - \frac{t}{2}}, \quad \dots \dots \dots (2)$$

The results by this formula are too high, since part of the invert sugar was a natural constituent of the fruit.

If, after inversion, the correct reading at  $20^{\circ}$  is found to be 12 or more to the left of the zero, it can be safely inferred that no appreciable amount of commercial glucose is present, and it is unnecessary to make a third reading at  $87^{\circ}$ , unless to confirm the fact. In such a case, with cane sugar alone present, the reading at  $87^{\circ}$  will not, of course, vary much from zero.

*Invert Sugar.*—In the absence of commercial glucose, the invert sugar is calculated as follows:

$$\text{Invert sugar} = \frac{(\text{Sucrose} - \text{direct reading}) 105.3}{42.66 - \frac{t}{2}}, \quad \dots \dots (3)$$

or it may be determined directly from the copper reducing power.

Any decided reading above zero at  $87^{\circ}$  is due to the presence of commercial glucose, and when the latter is present, it is impossible to determine the invert sugar from the copper reduction or by formula No. 3. The following formula is proposed for calculating approximately the invert sugar from the polarization, in the presence of commercial glucose. While theoretically correct, the method is subject to practical limitations, which admit of only roughly approximate results in such mixtures as jelly or jam. It is perfectly accurate only in mixtures of sucrose, glucose, and invert sugar.

$$\text{Invert sugar} = \frac{\left( \begin{array}{c} \text{Reading due to glucose and} \\ \text{inverted sucrose at } t^{\circ} \end{array} \right) - \left( \begin{array}{c} \text{Invert reading} \\ \text{at } t^{\circ} \end{array} \right)}{\pm \left( 42.66 - \frac{t}{2} \right)} 105.3 \quad (4)$$

These formulas, (3) and (4), serve at best to indicate the approximate amount of invert sugar present in the sample, resulting from the inversion of a portion of the original sucrose in the natural process of manufacture of the jam or jelly, and not the total invert sugar resulting from the inversion by the analyst of all the sucrose.

The factor 105.3 is used, since, in the natural process of inversion, 100 parts of sucrose become 105.3 parts of invert sugar.

*Example.*—The invert sugar in the sample of apple jelly first on the list in the table on page 995 is calculated as follows:



Invert reading at  $t^{\circ}$  ( $20^{\circ}$ ) = 28.0.

Reading due to glucose at  $20^{\circ}$  =  $.221 \times 175 = 38.68$ .

Reading due to inverted sucrose at  $20^{\circ}$  =  $.268 \times -34 = -9.11$ .

$$\text{Invert sugar} = \frac{(38.68 - 9.11) - 28}{28.66} 105.3$$

$$= 5.76\%.$$

**Determination of Reducing Sugar.**—Proceed as described on page 982.

**Commercial Glucose.**—While it is impossible to determine the exact percentage of this substance in preserves and jellies, by reason of the varying composition of its component parts, it is quite feasible to approximate very closely to the amount present. Indeed, this approximate method of calculation, wherein glucose is treated as a chemical entity, has been found in practice to be much more close to the actual truth than results gained by methods wherein the copper-reducing power enters as a factor, or methods for determining separately dextrin, maltose, and dextrose. Calculate the commercial glucose in jellies and jams exactly as in the case of honey, page 673.

**Detection of Dextrin.\***—Add alcohol to a somewhat thick solution of the fruit product. A white turbidity is at once apparent, followed by the formation of a thick gummy precipitate if dextrin is present. In the absence of dextrin there is no turbidity, but a light flocculent precipitate.

**Determination of Dextrin.**—*Bigelow and McElroy Method.*†—Dissolve 10 grams of the sample in a 100-cc. graduated flask, add 20 mg. of potassium fluoride, and then about one-quarter of a cake of compressed yeast. Allow the fermentation to proceed below  $25^{\circ}$  C. for two or three hours to prevent excessive foaming, and then place in an incubator at a temperature of from  $27^{\circ}$  to  $30^{\circ}$  C. for five days. At the end of that time clarify with lead subacetate and alumina cream; make up to 100 cc. and polarize in a 200-mm. tube. A pure fruit jelly will show a rotation of not more than a few tenths of a degree either to the right or to the left. If a Schmidt and Haensch polariscope be used, and a 10% solution be polarized in a 200-mm. tube, the number of degrees read on the sugar scale of the instrument, multiplied by 0.8755, will give the percentage of dextrin, or the following formula may be used:

$$\text{Percentage of dextrin} = \frac{C \times 1000 \times V}{198 \times L \times W}$$

\* U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 78.

† Jour. Amer. Chem. Soc., 15, 1893, p. 668.



in which

$C$  = degrees of circular rotation;

$V$  = volume in cubic centimeters of solution polarized;

$L$  = length of tube in centimeters;

$W$  = weight of sample in solution in grams.

**Determination of Crude Pectin (Alcohol Precipitate).**—*Munson and Tolman Method.*\*—Evaporate 100 cc. of a 20% solution of jelly, or 200 cc. of the washings from the determination of insoluble solids of a jam, to 20 cc.; add slowly and with constant stirring 200 cc. of 95% alcohol and allow the mixture to stand overnight. Filter and wash with 80% alcohol by volume. Wash this precipitate off the filter paper with hot water into a platinum dish; evaporate to dryness; dry at 100° C. for several hours and weigh; then burn off the organic matter and weigh the residue as ash. The loss in weight upon ignition is called alcohol precipitate.

The ash should be largely lime and not more than 5% of the total weight of the alcohol precipitate. If it is larger than this some of the salts of the organic acids have been brought down. Titrate the water-soluble portion of this ash with tenth-normal acid, as any potassium bitartrate precipitated by the alcohol can thus be estimated.

The general appearance of the alcohol precipitate is one of the best indications as to the presence of glucose and dextrin. Upon the addition of alcohol to a pure fruit product a flocculent precipitate is formed with no turbidity while in the presence of glucose a white turbidity appears at once upon adding the alcohol, and a thick, gummy precipitate forms. Since the precipitate in the latter case consists in part of substances other than pectin bodies the results should be stated as representing "alcohol precipitate" and not "pectin."

*German Method.*†—This method, designed for juices, may also be used for jams and jellies. It differs from the Munson and Tolman method chiefly in that a smaller proportion of alcohol is used and a correction is introduced for protein.

**Detection of Coloring Matter.**—Boil white woolen cloth or worsted in a solution of the jelly or jam, acidified with hydrochloric acid, or with acid sulphate of potassium, according to Arata's method and test for

\* U. S. Dept. of Agric., Bur. of Chem., Bul. 65, 1902, p. 79; Bul. 66 rev., p. 21.

† Koenig, *Chemie d. mensch. Nahr. u. Genussm.* III, 2, 1914, p. 891.



the color on the dyed fabric by methods given in detail in Chapter XVII. Apply also the general methods described in that chapter.

**Detection of Preservatives and Concentrated Sweeteners.**—Extract an acid aqueous solution of the fruit product with ether or chloroform in a separatory funnel, and test for benzoic and salicylic acids and for saccharin in the ether extract. If dulcin is suspected, extract with acetic ether.

**Detection of Starch.\***—Heat an aqueous solution of the preserve or jelly nearly to the boiling point, and decolorize by the addition of several cubic centimeters of dilute sulphuric acid and afterwards permanganate of potassium. This treatment does not affect the starch, which is tested for with iodine in the ordinary manner in the solution after cooling. In the clear filtrate from a boiled apple pulp solution, free from added starch, little or no darkening should occur on the addition of the iodine reagent. If, however, the reagent is added to the residue of the previously boiled pulp, the presence of starch inherent in the apple is usually recognized by the blue color produced thereon.

The presence of any considerable *added* starch paste in a fruit preparation is thus readily indicated by an intense blue color obtained by adding the iodine reagent to the filtrate (free from fruit pulp).

**Detection of Gelatin.—Robin's Method.†**—Add to a thick aqueous solution of the preserve or jelly sufficient strong alcohol to precipitate the gelatin. Decant the supernatant liquid after settling, set aside part of the precipitate, and dissolve the remainder in water. Divide the latter solution in two parts, to one of which add, drop by drop, a fresh solution of tannin, which precipitates gelatin if present. To the remainder add picric acid solution, which in presence of gelatin forms a yellow precipitate. The portion of the yellow precipitate set aside is transferred to a test tube, and heated over the flame with a little quicklime. If gelatin is present, ammonia will be given off, apparent by the odor, and by fumes of ammonium chloride when a drop of hydrochloric acid on a glass rod is held at the mouth of the bottle.

**Leffmann and Beam's Method.‡**—Boil the sample with water, filter, and boil the filtrate with an excess of potassium bichromate. Cool, and add a few drops of sulphuric acid. A flocculent precipitate indicates gelatin.

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 81.

† Girard, *Analyse des Matières Alimentaires*, p. 676.

‡ *Select Methods of Food Analysis*, p. 324.



**Detection of Agar Agar.\***—The jelly is heated with 5% sulphuric acid, a little potassium permanganate is added, and, after settling, the sediment is examined by the microscope for diatoms, which will be found in large numbers if agar agar has been used.

**Detection of Apple Pulp.**—A distinct clue to the presence of apple pulp in fruit preparations is often furnished by the characteristic apple odor given off when a small amount of the sample is heated to boiling with water in a test tube. Under such conditions, the apple odor is quite apparent, as distinguished from that of other fruits, especially if the apple is the chief fruit present, or predominates in the mixture.

Apple pulp in fruit preserves, free from added starch, may usually be recognized by a microscopical examination, using iodine reagent. The cell contents of the pulp will show the characteristic blue color, undoubtedly due to portions of unconverted starch still remaining in them.

**Detection of Fruit Tissues under the Microscope.** Certain of the common fruits are readily identified in jams by their microscopic characters. This is especially true of most of the small fruits, the skins, styles and seeds being more or less characteristic in structure.

The apple differs from the quince and pear in that stone cells are lacking; the starch of the green fruit is noteworthy. Peaches, plums and apricots, while possessing skins and stone peculiar to each, when pared and freed from stones are much alike in structure. Pineapples have peculiar needle-shaped crystals. Figs are identified by the "seeds" and hairs. Citrus fruits are remarkable because of the oil cavities and spongy parenchyma. Fragments of elements of the skins and cores of fruits, although pared and cored before preparation, find their way into the finished products, furnishing evidence to the microscopist. The seeds of berries are highly characteristic.

#### DRIED FRUITS.

Desiccation is the oldest and in some respects the most satisfactory method of preserving fruits. It is an economical method, as the apparatus and the process are simple, especially if the sun's heat is utilized for the evaporation; furthermore, the cost of the containers is small and the compact form of the product reduces the cost for transportation and storage

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\* Marpmann, *Zeit. f. angew. Mikrosk.*, 1896, p. 260; U. S. Dept. of Agric., Bur. of Chem., Bul. 65, p. 81.



to the minimum. From the sanitary standpoint dried fruit has certain advantages, notably the freedom from metallic impurities from the containers; on the other hand, great care is required to protect the material during drying and handling from surface contamination.

Xanti currants as well as raisins are dried grapes of certain European varieties. These, together with figs and dates, although produced in California and the Southern States, are imported into the United States in enormous quantities from the regions adjoining the Mediterranean. Apples, prunes, apricots, peaches, and cherries, on the other hand, are produced in the United States in quantities not only sufficient for domestic needs but also for export.

California fruits, such as raisins, prunes, apricots, peaches, and pears are sun-dried, as are also raisins, figs, dates and other fruits produced about the Mediterranean. Apples are commonly dried in the United States by artificial heat, although the old process of sun drying is still practiced on a small scale in certain regions.

**Treatment with Lye.**—Preliminary to drying certain fruits, such as raisins and prunes, are often dipped in a hot but weak solution of potash, which removes the bloom and otherwise acts on the skin, thus facilitating drying. Oil is also used with the lye in preparing "oil-dipped" Smyrna raisins. These methods of treatment are quite distinct from the lye-peeling process employed in preparing peaches, apricots, and some other fruits for canning.

**Sulphuring of Fruit.**—The treatment of fruits with the fumes of burning sulphur is practiced not only to bleach and prevent discoloration, but also to ward off the attacks of insects, fungi, and bacteria. It is allowed with restrictions in most European countries and also, pending further investigation, in the United States, provided the amount of sulphur dioxide remaining in the fruit does not exceed 350 mg. per kilo, of which not more than 70 mg. is free sulphurous acid.\*

There is reason to believe that the sulphur dioxide exists in dried fruits in combination largely, if not wholly, with sugar, although possibly to some extent, as in wines, with acetaldehyde, or even with protein and cellulose.

Sulphuring when used for purposes of deception, as for example in rejuvenating old or damaged stock or when used in excessive amount, is obviously improper. Analyses by government chemists show that when

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\* U. S. Dept. of Agric., Off. of Sec., Food Inspection Decision 76.



no restrictions were placed on sulphuring as high as 3072 mg. per kilo were present in dried peaches, 2842 mg. in California apricots and 1738 mg. in evaporated apples.

**Moisture Content of Dried Fruits.**—An excessive amount of moisture in dried fruit is not only a worthless make-weight, but also facilitates the growth of molds and bacteria, causing rapid deterioration. In 1904 a law was passed in New York State requiring that dried apples contain not above 27% of moisture, determined by drying four hours at the temperature of boiling water.

**Wormy and Decomposed Dried Fruits.**—Figs, dates, and currants from Europe, also dried apples, cherries, and other fruits of domestic production often are infected with worms or are in a moldy or fermented condition due to careless drying or packing. Under the federal law such "filthy, decomposed or putrid" fruit is adulterated.

**Zinc in Dried Fruit.**—Apples dried in contact with galvanized iron trays may contain 0.01 to 0.02%, or in extreme cases, according to Looock, 0.09% of zinc as malate. This contamination may be avoided by greasing the trays, covering them with greased cloths, or using wooden trays.

### FRUIT JUICES.

Such preparations, if of the highest purity, should consist of the undiluted juices of these fruits, separated by pressure and carefully sterilized and bottled. They should contain no other fruit juice than that specified on their labels, and should be free from alcohol, added antiseptics, or coloring matter, unless the label specifies the presence of the added foreign materials. The addition of pure cane sugar to such preparations as grape juice is allowable if declared, as well as charging with carbon dioxide to form so-called carbonated drinks.

**Composition of Fruit Juices.**—Analyses of various fruit juices, pressed out in the laboratory, by Munson and Tolman are given on page 992.

The following analyses of pure fruit juices are taken from tables prepared by Winton, Ogden, and Mitchell, showing results on samples purchased in the Connecticut market, as well as on some samples made in the laboratory.\*

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\* Conn. Agric. Exp. Sta. Rep., 1899, p. 136.



	Solids.	Acids Other than CO <sub>2</sub> as Citric.	Cane Sugar.	Invert Sugar.	Polarization.			
					Direct.	After Inver- sion.	Temper- ature C.	Invert Reading at 86° C.
COMMERCIAL FRUIT JUICES.								
Blackberry .....	5.32	0.65	0.0	4.6	-1.3	-1.3	29.0	
Cherry .....	14.33	0.80	0.0	6.5	-1.9	-1.9	26.0	
Black currant .....	10.00	2.41	0.0	9.2	-2.7	-2.7	26.0	
Red currant .....	7.58	2.09	0.0	7.2	-2.1	-2.1	27.0	
Grape .....	15.29	0.91	0.0	....	-6.5	-6.5	25.0	-1.0
Lime fruit .....	7.78	6.50	0.0	0.0	0.0	0.0		
Orange .....	12.72	2.44	0.0	7.1	-2.1	-2.1	26.0	
Pineapple .....	8.07	0.81	1.5	5.1	0.0	-2.0	26.0	
Plum .....	10.81	1.00	0.0	0.3	-0.1	-0.1	26.0	
Quince .....	10.41	0.99	0.0	....	-5.0	-5.0	25.0	-2.2
Black raspberry .....	8.47	1.36	0.0	7.8	-2.3	-2.3	26.0	
Strawberry .....	5.69	0.99	0.0	5.1	-1.5	-1.5	26.0	
MADE IN LABORA- TORY.								
Peach .....	12.70	0.95	5.4	2.1	4.8	-2.2	28.0	0.0
Red raspberry .....	9.41	1.19	0.8	8.6	-1.6	-2.8	26.0	0.0
Blackberry .....	8.94	1.22	0.0	8.7	-2.4	-2.4	30.0	0.0
Huckleberry .....	11.40	0.51	0.6	....	-4.0	-4.8	30.0	-1.0
Pineapple .....	13.90	0.68	7.4	9.1	4.7	-4.8	28.0	-0.8

**Grape Juice.**—Following are the averages of analyses of grape juice made from European varieties of grapes reported by Bioletti and dal Piaz:\*

	Alcohol.	Solids Calc. from Specific Grav- ity.	Sugar.	Acidity Calc. as Tar- taric.	Vola- tile Acid.	Free Tar- taric Acid.	Cream of Tartar.	Ash.	Phos- phoric Acid.
Made in—									
Austria .....	none	21.62	19.62	0.78	0.01	0.03	0.61	0.37	0.02
California .....	none	20.60	19.15	0.53	0.03	0.07	0.59	0.19	0.04

The table given below, summarized from tables by Hartmann and Tolman,† shows the maximum, minimum, and average of analyses of 93 samples of commercial grape juice obtained under supervision at five factories in New York state and one in Ohio during the years 1912, 1913, and 1914.

\* Cal. Agr. Exp. Sta., Bul. 130, 1900.

† U. S. Dept. of Agric., Bul. 656, 1918.



	Alcohol Vol. per Cent.*	Grams per 100 cc.								Cc. N/10 Acid per 100 cc.	
		Solids.†	Sugars Calc. as Invert‡	Acidity Calc. as Tartaric.§	Free and Combined Tartaric Acid.	Free Tartaric Acid.¶	Cream of Tartar.	Ash.	Tannin and Coloring Matter.**	Alkalinity Water-soluble Ash.	Alkalinity Water-insoluble Ash.
Max.....	0.37	20.78	17.53	1.28	1.01	0.36	0.79	0.37	0.37	42.0	8.8
Min.....	0.02	14.20	11.52	0.81	0.56	0.12	0.36	0.22	0.07	19.0	3.1
Aver.....	0.12	18.33	15.31	1.01	0.74	0.23	0.54	0.29	0.24	28.7	5.1

\*By immersion refractometer.

† Brix.

‡ Direct and invert polarization practically the same.

§ Spotted into litmus solution.

|| Total tartaric acid by Hartmann and Eoff method (p. 731).

¶ Calc. from total tartaric acid and cream of tartar (p. 732).

\*\* Löwenthal method.

**Sweet Cider.**—The composition of pure, freshly expressed apple juice is shown by the following table of analyses by Browne: \*

	Specific Gravity.	Solids.	Invert Sugar.	Sucrose.	Total Sugar.	Total Sugar after Inversion.	Free Malic Acid.	Ash.	Undetermined (Pectin, etc.).	Left-handed Rotation Degrees Ventzke 400 mm. Tube.
Red astrachan. ....	1.0532	12.78	6.87	3.63	10.50	10.69	1.14	0.37	0.77	23.72
Early harvest. ....	1.0552	13.29	7.49	3.97	11.46	11.67	0.90	0.28	0.65	24.32
Yellow transparent. ....	1.0502	11.71	8.03	2.10	10.14	10.24	0.86	0.27	0.44	
Sweet bough. ....	1.0498	11.87	7.61	3.08	10.69	10.85	0.10	.....	.....	39.40
Baldwin, green. ...	1.0488	11.36	6.96	1.63	8.59	8.68	1.24	0.31	1.22	36.16
" ripe. ....	1.0736	16.82	7.97	7.05	15.02	15.39	0.67	0.26	0.87	
Ben Davis. ....	1.0539	12.77	7.11	3.85	10.96	11.16	0.46	0.28	1.07	49.00

Determination of sugars in the juice of 15 American and 5 French varieties of apples made by Eoff † showed that in every instance the amount of levulose exceeded that of dextrose and sucrose combined.

Bottled sweet cider, properly sterilized, should not differ materially from the fresh juice, and should contain no considerable amount of alcohol.

Salicylic acid, sodium benzoate and sodium or calcium bisulphite have been extensively used as preservatives. Benzoate is still used to some extent.

**Lime or Lemon Juice.**—The juice of both the lime and the lemon is known commercially as lime juice and the Canadian standard goes so far

\* Penn. Dept. Agric., Bul. 58, p. 29.

† Jour. Ind. Eng. Chem., 9, 1917, p. 587



as to recognize "various species" of *Citrus*. In former editions of the U. S. Pharmacopœia *C. limonum* was specified and the product known as lemon juice was required to conform to the following: Specific gravity at 15° C. at least 1.030, citric acid about 7%, and ash not more than 0.5%. In the 9th decimal revision neither lemon nor lime juice is given.

The table below shows the range in composition of 40 samples classed by McGill\* as genuine, together with the Canadian limits fixed by Order of Council, Jan. 28, 1915. Many of the samples were preserved with benzoic, salicylic, or sulphurous acid.

COMPOSITION OF LIME JUICE (MCGILL)

	Specific Gravity, 20° C.	Total Solids.	Acidity Calc. as Citric.	Rotation in 200-mm. Tube, ° V.
Genuine lime juice:				
Maximum.....	1.0531	12.12	10.18	+0.6
Minimum.....	1.0305	7.76	6.93	-2.2
Canadian limits:				
Maximum.....	1.040	.....	.....	+0.5
Minimum.....	1.030	8.00	7.00	-1.0

Samples examined in previous years at the laboratory of Inland Revenue, Canada, and at the Mass. State Board of Health were often found to be watered, preserved with salicylic, benzoic, or sulphurous acid, artificially colored, or otherwise sophisticated. One sample, examined by Leach purporting to be "pure West Indian lime juice, triple refined," proved to be a mixture of hydrochloric and salicylic acids, colored with a coal-tar dye, and containing no lime juice whatever.

## METHODS OF ANALYSIS.

**Total Solids, Total Nitrogen, Ash, and Sugars** are determined by the methods employed for jams and jellies (pp. 995 to 1002), **Solubility and Alkalinity of the Ash and Phosphoric Acid** as described in the chapter on vinegar (p. 795).

**Colors and Preservatives** are detected and determined as described in Chapters XVII and XVIII.

**Total Acidity.**—Titrate 10 grams of the juice, diluted to 250 cc. with freshly boiled water, with tenth-normal alkali. Use phenolphthalein as

\* Lab. Inl. Rev. Dept., Bul. 321, 1916.



indicator if the color of the juice will permit, otherwise delicate litmus paper. Calculate either as sulphuric acid or as the organic acid known to predominate.

One cc. of tenth-normal alkali is equivalent to 0.0075 gram tartaric acid, 0.0067 gram malic acid and 0.0064 gram citric acid.

**Determination of Total Tartaric Acid.**—Proceed as directed for wine, p. 731, using only 50 cc. of the sample diluted to 100 cc. and adding 20 instead of 15 cc. of alcohol.

**Determination of Malic Acid.**—*Dunbar and Bacon Method.\**—Dilute a weighed or measured amount of the fruit juice, usually 10 grams, with quite a large volume of water, add phenolphthalein, and titrate with standard alkali to a decided pink color. Weigh or measure another portion of the liquid (75 grams or cc. is a convenient amount) into a 100-cc. graduated flask, and add enough standard alkali, calculated from the above titration, to neutralize the acidity. A slight excess of alkali is not objectionable. If the solution is dark colored, add 5 or 10 cc. of alumina cream. Dilute to the mark, mix thoroughly, and filter if necessary through a folded filter.

Treat about 25 cc. of the filtrate with enough powdered uranyl acetate so that a small amount remains undissolved after two hours, 2.5 grams usually being sufficient, except in the presence of large amounts of malic acid. In case all the uranium salt dissolves more should be added. Allow to stand for two hours, shaking frequently, filter through a folded filter until clear and polarize if possible in a 200-mm. tube or, if too dark, in a 100- or 50-mm. tube. Designate this solution and reading as *A*.

Treat the remainder of the original filtrate with powdered normal lead acetate until the precipitation is just complete, avoiding a large excess and consequent solution of lead malate. Cool in an ice bath and filter through a folded filter until clear. Warm the filtrate to room temperature and add a small crystal of lead acetate. If no precipitate forms, remove the excess of lead with anhydrous sodium sulphate, filter until clear, and polarize. Designate this solution and its polarization reading as *B*. Solutions which are sufficiently clear and contain less than 10% of sugar may be polarized directly without treatment with lead acetate.

If reading *B* is negative treat a portion of solution *B* with uranyl acetate in the manner already described and polarize. Designate this as *C*. If reading *B* is positive, reading *C* need not be made.

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\* U. S. Dept. of Agric., Bur. of Chem., Circ. 76. Jour. Ind. Eng. Chem., 3, 1911, p. 826.



Polarize all solutions at a uniform room temperature with white light, using the average of at least six readings and calculating to the basis of a 200-mm. tube. If reading *C* is numerically less than reading *B*, the latter should be discarded; otherwise use reading *B* in the subsequent calculation. Multiply the algebraic difference between this reading and reading *A* by 0.036, the product being the percentage of malic acid ( $C_4H_6O_5$ ) in the solution as polarized.

*Pratt's Modification*.<sup>\*</sup>—Place a weighed amount of juice, generally 100 grams, in a 500-cc. beaker and add, with vigorous stirring, two or three times its volume of 95% alcohol. The pectin bodies are precipitated and usually in such a form that after standing a few minutes they may be gathered into a coherent mass. Decant the liquid through a filter and wash the precipitate twice with 95% alcohol. Evaporate the filtrate in a current of air on the water-bath to about 75 cc. After cooling make up to 100 cc. in a measured flask, using 10 to 15 cc. of 95% alcohol and distilled water. The temperature when the volume is finally made up to the mark should be close to that at which the polariscope readings are to be taken. Treat this solution exactly as in the original method, except that no clarification is necessary.

**Determination of Citric Acid.**—*Pratt Method*.<sup>†</sup>—This method is applicable in the presence of malic and tartaric acids, but according to Bigelow and Dunbar<sup>‡</sup> does not give accurate quantitative results although it serves to show the presence or absence of citric acid. Willaman's modification<sup>§</sup> is designed to correct the defects of the method but has not yet been rigidly tested. Dunbar and Lepper<sup>||</sup> recommend the Stahre-Kunz method. The original Pratt method is here given pending further improvement or the introduction of a better method.

1. *Apparatus*.—This consists of a 500-cc. distilling flask provided with a small dropping funnel drawn down to a small opening and protruding  $\frac{1}{2}$  inch below the stopper. In the flask is placed a glass rod with a piece of small tubing  $\frac{1}{2}$  inch long, sealed on the lower end to insure steady ebullition. This small tube should be filled with air when the heating begins. A condenser preferably of the spiral type is connected with the flask.

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<sup>\*</sup> U. S. Dept. of Agric., Bur. of Chem., Circ. 87.

<sup>†</sup> U. S. Dept. of Agric., Bur. of Chem., Circ. 88, 1912.

<sup>‡</sup> Jour. Ind. Eng. Chem., 9, 1917, p. 762.

<sup>§</sup> Jour. Amer. Chem. Soc., 38, 1916, p. 193.

<sup>||</sup> Jour. Assn. Off. Agr. Chem., 2, II, 1917, p. 175.



2. *Denigès Reagent*.—Add about 500 cc. of water to 50 grams of mercuric oxide; then add 200 cc. of concentrated sulphuric acid with constant stirring, and heat the mixture, if necessary, on a steam bath until the solution is complete. After cooling make up to a liter and filter.

3. *Determination*.—Weigh 50 grams of the fruit juice into a beaker and add 110 cc. of 95% alcohol to throw out the pectin bodies. After standing fifteen minutes filter and wash with 95% alcohol. Dilute the filtrate with water to approximately 50% alcohol content and add enough 20% barium acetate solution to precipitate the citric acid. Stir, let stand until the barium citrate partially settles, and filter. Wash twice with 50% alcohol to remove the greater part of the sugar present. Remove all alcohol from the precipitate and filter either by drying in the beaker used for precipitation or else by washing with ether before removing from the funnel. Add 50 cc. of water and 3 to 5 cc. of sirupy phosphoric acid to the beaker containing the filter-paper and precipitate and warm, thus dissolving the barium citrate completely. Filter into a 100 cc. measuring flask and wash up to the mark.

Measure an aliquot containing from 0.05 to 0.15 gram of citric acid, into the distilling flask, add 5 to 10 cc. of sirupy phosphoric acid and 400 cc. of hot water. Connect with the condenser, heat and when briskly boiling, add potassium permanganate solution (0.5 gram per liter), 1 to 2 drops per second, until a pink color persists throughout the solution. Distil off the acetone formed by the oxidation into a liter Erlenmeyer flask containing 30 to 40 cc. of Denigès reagent, continuing the distillation until 50 to 100 cc. remain in the flask. Boil the distillate gently under a reflux condenser for forty-five minutes after it turns milky. Filter hot through a Gooch crucible, wash the precipitate with water, alcohol, and finally with ether, and dry in a water-oven for half an hour. The weight of the precipitate multiplied by 0.22 gives the weight of citric acid.

#### FRUIT SYRUPS.

Two classes of these preparations are on the market, one for use in soda-fountains, and one for "family trade," intended as a basis for sweetened drinks to be diluted with water and sugar. Some are made exclusively from fruit pulp or juice and sugar, sterilized by heating and put-up in tightly sealed bottles, while others of the cheaper variety are more apt to be entirely artificial both in color and in flavor, deriving the latter principally from the wide variety of artificial fruit essences now available.



Commercial glucose is a frequent ingredient. The same classes of coal-tar dyes and antiseptics are found in these preparations as in the other fruit products. Citric or tartaric acid is frequently added to genuine fruit syrups to bring out the flavor and to imitation fruit syrups to better simulate the characters of the genuine product.

For purposes of comparison with such fruit-pulp preparations as may come to the analyst for examination, he is referred to the analysis of fruits found on pages 283 and 993.

#### NON-ALCOHOLIC CARBONATED BEVERAGES.

**Soda Water.**—Originally the beverage known as soda water was prepared by the action of an acid on sodium bicarbonate in solution and corresponded to what is now obtained by dissolving Seidlitz powders in water. Later it was found that water charged with carbon dioxide is not only more practicable commercially but also more acceptable to the palate, and this product was substituted for true soda water without change of name.

As dispensed by the pharmacist and confectioner in the United States, soda water consists of a syrup, variously flavored, mixed at the "fountain" with carbonated water. The syrup is first placed in the glass, then the carbonated water is drawn into it in a large stream and finally more added in a fine stream to mix and froth the liquid. Ice cream or liquid "cream" is used with certain flavors, eggs and milk in "egg chocolate," "egg shake" and other nutritious mixtures, a solution of calcium acid phosphate in "orange phosphate" and other phosphates—in fact there appears to be no end to the preparations and combinations introduced by ingenious vendors to quench the thirst, gratify the palate, and furnish nourishment in an easily digestible form.

**Carbonated Water**, the basis of all effervescent drinks, is prepared by charging ordinary water with carbon dioxide in a steel drum, known as the fountain. Formerly the gas was generated on the premises by the action of mineral acid on marble, but now it is obtained in liquid form in steel cylinders from mineral springs and the fermentation industries where it formerly went to waste.

The process of carbonating consists in allowing the gas to discharge into the water, rocking the fountain continually to aid absorption. A gauge indicates the pressure in the fountain, which should be about 170 pounds per square inch for soda water and somewhat less for ginger ale



and root beer. The steel drum or fountain proper is kept in the cellar or other convenient place and the carbonated water is piped to the so-called fountain where the drinks are served, or, in the case of bottled beverages, to the machine for filling the bottles.

Needless to say both the water and the gas should be free from contamination, and the introduction of metallic salts from the lead pipes and other sources should be guarded against.

**Soda Water Syrups.**—Sugar and flavors are added to carbonated beverages in the form of syrups. At the soda fountain these are drawn into the glass from small reservoirs or poured from bottles, while in the bottling works measured quantities both of syrup and carbonated water are introduced into each bottle by an automatic machine.

*Fruit Syrups* are prepared either by the manufacturer of soda water supplies or else by the pharmacist or confectioner who serves the beverages. More commonly the manufacturer supplies the fruit juice or fruit pulp in bottles or jars, spoilage being avoided either by sterilization or the use of sodium benzoate. The vendor mixes the juice or pulp with sugar syrup as needed. Orange, lemon, and lime syrups are commonly made from the oils rather than from the fresh fruit, the necessary acidity being supplied by citric acid. This acid as well as tartaric acid is also used in strawberry, raspberry and other true fruit syrups to bring out the flavor.

*Imitation Fruit Syrups* flavored with mixtures of ethers such as are described on pages 954 to 956, are frequently substituted for genuine fruit syrups at soda fountains and quite universally in the preparation of cheap bottled soda water. Aside from the deception to the consumer these mixtures are highly objectionable because of their nauseating and unwholesome properties.

*Various Syrups* not belonging under the head of fruit syrups are drawn from fountains and used in bottled beverages. Among these are vanilla, coffee, chocolate (really cocoa), ginger, sarsaparilla, and mixtures sold under distinctive names.

**Bottled Carbonated Beverages.**—To this class belong various non-alcoholic beverages known as "soda," "soft-drinks" and "temperance drinks." Some of these are high-grade articles of national or even international reputation, so prepared as to keep indefinitely, while others are cheap preparations of local manufacture sold for immediate consumption in pleasure resorts.

*Ginger Ale*, by far the best-known bottled carbonated beverage, is made



from ginger (or ginger extract) with the addition of lemon juice (or lemon oil and citric acid) and carbonated water. Capsicum extract, known in solid form as capsin, is frequently substituted in part for the ginger because of its greater pungency.

*Root Beer* was formerly brewed from a sweetened infusion of various roots and herbs, the gas being formed by a true fermentation process. A similar beverage is now made in the household, using so-called "root-beer extract," but the commercial product is commonly charged, like soda water, with carbon dioxide gas.

*Birch Beer*, formerly made by fermentation, is now merely soda water flavored with oil of birch or synthetic methyl salicylate.

*Sarsaparilla*, so called, is flavored with a mixture of oil of birch, natural or synthetic, and oil of sassafras. The dark color is due to caramel or other artificial colors.

*Lemon Soda* and *Orange Soda* are flavored respectively with terpeneless lemon and orange extract, the acidity being contributed by citric acid. Orangeade belongs in the same class. So-called blood orange soda is probably never made from blood oranges, the color being artificial.

*Vanilla Soda* is more correctly vanillin soda or vanillin and coumarin soda. The term cream soda applied to this colorless beverage is equally misleading.

*Strawberry Soda*, *Raspberry Soda* and other bottled beverages purporting to be made from fruits are commonly imitations flavored with ethers and colored with coal-tar dyes. So-called *Cherry Soda* is flavored with an extract of cherry bark or benzaldehyde.

**Sweeteners in Beverages.**—Sugar is the only proper sweetener for syrups of bottled beverages. Glucose because of its lower sweetening power is unsuited for the purpose, while saccharin and other chemical sweeteners are objectionable both because of their lack of nutritive properties and their possible injury to health. The use of saccharin, which has hitherto been extensive, is now prohibited in beverages entering into interstate commerce.

**Acids in Beverages.**—Citric and tartaric acids are used not only in imitation, but also in true fruit syrups to bring out the flavor. Lemon juice serves the same purpose, but is more expensive and does not keep so well. Calcium acid phosphate is a characteristic constituent of orange and other fruit phosphates.

**Preservatives.**—Sodium benzoate is the common preservative of bev-



erages, although its use is by no means universal. Formerly salicylic, boric and sulphurous acids and even fluorides were employed. A recent German patent names *p*-chlorobenzoic acid as a harmless preservative many times as effectual as benzoic acid.

**Artificial Colors.**—Cochineal, cudbear, caramel and the eight colors allowed by U. S. decisions are most commonly met with. The use of fuchsin, acid fuchsin, rhodamine, and other coal-tar colors has been largely discontinued.

**Foam Producers.**—Froth on soda water is cheaper to produce than the same bulk of liquid, furthermore it is sanctioned by custom.

*Soap-bark*, the bark of *Quillaja Saponaria*, a common foam producer, contains two saponins, sapontoxin and quilliac acid, both of which are poisonous. In addition these principles combine with the cholesterin of the blood and if in excess dissolve the corpuscles.

*Commercial saponin*, prepared from *Saponaria officinalis*, and consisting largely of sapotoxin, is also extensively used.

Foam producers are also used in malt liquors.

*Glycerrhizin*, the characteristic principle of licorice, also serves as a foam producer.

**Habit-forming Drugs in Beverages.**—Caffein, extract of cola leaves, and cocaine are ingredients of certain proprietary syrups and beverages, contributing their well-known stimulating properties. The use of caffein is defended on the ground that it is the active principle of tea and coffee. Opponents of this drug have pointed out that tea and coffee are recognized as improper articles of diet for children and invalids, furthermore, the presence of other constituents tends to prevent the excessive use of these beverages. Again the presence of caffein in carbonated beverages is not usually known to the consumer, and he forms the habit without proper warning.

It would be difficult to find an argument in favor of the use of a drug so potent as cocaine or products containing cocaine.

#### METHODS OF ANALYSIS.

Transfer the sample to a flask and shake at intervals for an hour or two, at room temperature, thus removing most of the carbon dioxide. Use the liquid thus obtained for the several determinations, measuring out the portions, if desired, and calculating the weight from the specific gravity.



**Total Solids, Ash, Acidity, and Individual Sugars** are determined as directed for jams and jellies (pages 995 to 999) using 25 grams of the liquid except for the polarizations, which may be made on normal quantities.

**Vanillin, Coumarin, Citral, and Methyl Salicylate** are detected and determined by the methods described under the head of Flavoring Extracts, with such modifications as are necessitated by the absence of alcohol on the one hand and the greater dilution on the other. Methods for the detection of **Ginger** and **Capsicum** are given on page 952.

**Detection of Colors, Preservatives, and Sweeteners.**—See Chapters XVII, XVIII, and XIX.

**Determination of Phosphoric Acid.**—This determination is made in so-called "orange phosphate," "raspberry phosphate" and other beverages containing calcium acid phosphate.

Treat 25 grams of the liquid according to the method described on page 362, except that the entire residue, after ignition with magnesium nitrate, is used for the determination, without aliquoting.

**Determination of Alcohol.**—Follow the method prescribed for wines (page 687). The amount of volatile oil present is seldom sufficient to appreciably affect the results.

**Detection of Saponin.**—Of the various color tests that have been proposed none has been found absolutely characteristic, especially if glycerrhizin is present, although the reactions with sulphuric acid and Fröhde reagent are of considerable value. The hæmolysis test is believed to be reliable even in the presence of glycerrhizin. Whichever test is applied the saponin should be separated from interfering substances as follows:

I. *Extraction of Saponin by the Rühle-Brummer Method.\**—In the case of soda water and other products containing organic or mineral acids (other than carbonic), to 100 cc. of the liquid add an excess of precipitated magnesium carbonate and filter. If dextrin is present, as in the case of malt liquors, evaporate 100 cc. of the liquid to 20 cc., precipitate with 150 cc. of 95% alcohol, let stand thirty minutes, then heat to boiling, filter, dilute the filtrate with water and dealcoholize, finally making up the solution to 100 cc.

To 100 cc. of the neutral, dextrin-free solution in a separatory funnel, add 20 grams of ammonium sulphate, 9 cc. of phenol and shake thoroughly. Draw off the watery layer and shake the phenol solution with a mixture

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\* Zeits. Unters. Nahr. Genussm., 5, 1902, p. 1197; 16, 1908, p. 165; 23, 1912, p. 566.



of 50 cc. of water, 100 cc. of ether, and (if necessary to avoid an emulsion) 4 cc. of alcohol. Allow to stand until the liquids separate, which usually requires twelve to twenty-four hours. Draw off the aqueous solution and evaporate nearly to dryness, finishing the drying either at 100° C. or in a desiccator, the latter being preferable if the residue is to be purified by treatment with acetone, which is usually desirable. Employ this extract, consisting of saponin and impurities, in the following tests:

II. *Tests for Saponin*.—1. *Sulphuric Acid Test*.—Rub up a portion of the extract with a few drops of sulphuric acid. Saponin is indicated by the appearance in a few minutes of a reddish color changing in half an hour to red-violet and finally to gray.

2. *Fröhde Test*.—Treat another portion in like manner with a few drops of a mixture of 100 cc. of concentrated sulphuric acid and 1 gram of ammonium molybdate. In the presence of saponin the drops in fifteen minutes become violet, changing later to green and finally to gray.

3. *Foam Test*.—Shake another portion of the extract with water and note its foam-producing properties.

In the presence of glycerrhizin none of the last three tests is reliable.

4. *Haemolysis Test*.—This process is recommended by Rusconi,\* Sormali,† and Rhüle.‡ The following details are given by Rhüle and are based on the method as described by Gadamer: §

(a) *Reagents*.—(1) *Physiological Salt Solution*.—Dissolve 8 grams of sodium chloride in water and make up to one liter.

(2) *One per cent Defibrinated Blood*.—Shake vigorously fresh ox blood in a sterilized, salt-mouth, 500-cc. bottle with 20 glass beads 5–7 mm. in diameter. Separate from the clot of fibrin and store in a sterilized container in a refrigerator. Properly cared for it should keep for several days.

Dilute with 100 volumes of physiological salt solution for use.

(3) *One per cent Blood Corpuscles*.—Centrifuge 100 cc. of the 1% defibrinated blood in physiological salt solution, pour off the clear solution containing the cholesterol and make up again to 100 cc. with the salt solution. This preparation is more sensitive than solution (2).

(b) *Process*.—Dissolve about 0.1 gram of the extract in 25 cc. of physiological salt solution, filter, and shake 1, 2, and 3 cc. of this solution

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\* Bol. Soc. Med. Chi., Pavia, 1910.

† Zeits. Unters. Nahr. Genussm., 23, 1912, p. 562.

‡ Ibid., p. 566.

§ Lehrbuch der chemischen Toxicologie. Göttingen, 1909, p. 443.



in small test-tubes with 1 cc. portions of 1% defibrinated blood. If saponin is present the liquid becomes clear in from a minute to an hour or two, depending on the amount of saponin in the beverage and the number of cubic centimeters of the solution used.

As a confirmatory test dissolve 1 mg. of cholesterol in a small amount of ether, shake with 10 cc. of the solution of the extract in salt solution, heat at 36° C., for a few hours to remove ether, avoiding concentration, and treat portions of this solution with 1% defibrinated blood as above described. Cholesterol destroys the hæmolytic action of the saponin, hence the liquids should not become clear in these tests. In order to exert this influence cholesterol should be present to the extent of 1 part to 5 parts of saponin.

If only a small amount of saponin is present the hæmolytic action can best be noted under a microscope magnifying to 300 diameters. Mount a drop of the solution of the extract in salt solution and place a drop of either solution (2) or (3) in contact with it. The saponin causes the corpuscles in contact with it to swell, then become strongly refractive, and finally dissolve.

*Muller-Hössly*\* neutralizes 500 to 1000 cc. of the sample and blows a current of air into it through a glass tube extending to the bottom of the container, collects the foam which froths over, and makes the test on the liquid obtained by the subsiding of the foam. The saponin in the foam is in much greater amount than in the original liquid.

**Determination of Caffein.**—*Fuller Method*.†—Weigh 50 grams or measure an equivalent volume into a small beaker, add 5 cc. of concentrated ammonium hydroxide, allow to digest overnight; then add 2 cc. more of ammonium hydroxide, heat for two hours, transfer to a large separatory funnel, dilute with 3 volumes of acid, add 5 cc. of ammonium hydroxide and shake out with four successive portions of chloroform, each of 50 cc. In case any dyestuff is removed by the chloroform, shake out with a saturated solution of sodium bisulphite, which will remove some of the color.

Distil off the bulk of the chloroform and evaporate the remainder in a porcelain dish. Dissolve the residue in 25 cc. of 2% sulphuric acid, shake out with five portions of 15 cc. each of chloroform, filter the combined chloroform solutions into a flask, distil off the bulk of the chloroform and evaporate in a tared dish; dry at 100° C. and weigh.

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\* Mitt. Lebensm. Hyg., 8, 1917, p. 113.

† U. S. Dept. of Agric., Bur. of Chem., Bul. 137, p. 191.



If the caffein is not pure, dissolve in 15 cc. of 10% hydrochloric acid, add an excess of a solution of 10 grams of iodine and 20 grams of potassium iodide in 100 cc. of water, allow to stand overnight, filter, and wash twice with 10 cc. of the iodine solution. Transfer filter and precipitate to the original precipitation flask, add sufficient sulphurous acid to dissolve the precipitate, heating gently, filter into a separatory funnel, wash three times with water, and add ammonium hydroxide in excess; shake out four times with 15 cc. portions of chloroform, and filter the chloroform extracts into a flask, using a 7 cm. filter and keeping the funnel covered with a watch glass. Wash the filter with 5 portions of 5 cc. of chloroform. If the chloroform extract is colored, concentrate, add a small amount of animal charcoal, rotate several times and filter. Distil off part of the solvent and evaporate the remainder in a tared dish, dry at 100° C., and weigh.

**Detection and Determination of Cocaine.**—*Fuller Method.\**—To 200 cc. of the sample in a large separatory funnel, add concentrated ammonium hydroxide to alkaline reaction, and shake out with three portions of 50 cc. each of Prolius mixture (4 parts ether, 1 part chloroform, 1 part alcohol), collecting the solvent in another separatory funnel. If desired the aqueous solution may be reserved for the detection of salicylic and benzoic acids and saccharin. Filter the combined Prolius extracts into an evaporating dish, and evaporate on a steam bath, removing the dish as the last traces of solvent disappear. Dissolve the residue in normal sulphuric acid, transfer to a separatory funnel and shake out four times with 15 cc. portions of chloroform; wash the combined chloroform solutions once with water, reject the chloroform, and add the water extract to the original acid solution. Add 10 cc. of petroleum ether, boiling at 40° to 50° C., and shake; draw off the acid layer, rejecting the petroleum ether, add concentrated ammonium hydroxide in excess and shake out three times with 15 cc. portions of petroleum ether, collecting the ethereal solutions in another separatory funnel. To the latter add 10 cc. of water and shake thoroughly; reject the water extract and filter the petroleum ether into a beaker, washing twice with 10 cc. portions of the solvent. Evaporate over a steam bath, using a fan. By this method, if coca alkaloids are present, a nearly colorless residue will be obtained, which will finally crystallize on standing.

Dissolve the residue in petroleum ether and divide into four portions, one of which may be small. Evaporate the solvent and to the small

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 137, p. 192.



portion add a few drops of normal sulphuric acid, warm gently, filter into a test-tube, and add a drop of potassium mercuric iodide test solution (Meyer's reagent). A precipitate indicates an alkaloid, but does not identify it as cocaine; if no precipitate forms, cocaine is not present and further test is unnecessary.

To another portion add a few drops of concentrated nitric acid, and evaporate on a steam bath until the acid is all driven off, then add a few drops of half normal alcoholic potash and note the first odor that comes off, which, if cocaine is present, is that of ethyl benzoate.

The residue of the third portion should be applied to the end of the tongue by rubbing with the finger. Cocaine will cause a numbness which is not apparent immediately, but develops gradually, and persists for a longer or shorter time according to the amount present.

Remove a portion of the fourth residue to a microscopic slide, add a drop or two of gold chloride test solution, and stir vigorously, noting the character of the crystals under the microscope.

All the above tests should be checked by controls on pure cocaine.

If a quantitative determination of coca alkaloids is desired the residue after evaporating the petroleum ether should be weighed, then, as a check on the gravimetric determination, warmed in 50 cc. of fiftieth-normal sulphuric acid until dissolved, cooled, and titrated with fiftieth-normal potassium or sodium hydroxide, using cochineal as indicator. The factor for cocaine is 0.006018.

**Determination of Caffein and Detection of Cocaine and Glycerol.—***Fuller Method.\**—Weigh 50 grams of the sample into an evaporating dish, add 5 cc. of concentrated ammonium hydroxide, cover with a watch glass and allow to stand twelve hours. Add 2 cc. more of ammonium hydroxide and evaporate on steam bath. Warm the residue with 25 cc. of 95% alcohol on the steam bath, cool, and pour off the alcohol into another evaporating dish, repeating the treatment four times. Evaporate the combined alcoholic extract, dissolve the residue at 25 cc. of 2% sulphuric acid, transfer to a separatory funnel and shake out 5 times with 15 cc. portions of chloroform.

Reserve the acid liquid for subsequent tests for cocaine and glycerol.

Distil off most of the chloroform, evaporate in a dish on a steam bath, dissolve the residue in 10% hydrochloric acid and transfer to a small flask. Add to the acid solution an excess of iodine solution (10 grams

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\* U. S. Dept. of Agric., Bur. of Chem., Bul. 137, p. 192.



iodine and 20 grams potassium iodide in 100 cc. of water), rotate flask, allow to settle overnight, filter, and wash flask and precipitate twice with the iodine solution, then transfer filter and precipitate to the flask. Heat gently with sufficient sulphurous acid solution to dissolve the precipitate, filter into a separatory funnel, cool, add excess of concentrated ammonium hydroxide, and shake out four times with 15 cc. portions of chloroform. Filter the chloroform extract into a flask, using a 7-cm. filter in a small funnel covered with a watch glass, or filter through cotton plugged in the stem of the separatory funnel. Decolorize the chloroform, if necessary, with animal charcoal, distil off most of the chloroform, then evaporate in a tared dish over steam, dry at  $100^{\circ}$  C. and weigh.

Add an excess of concentrated ammonium hydroxide to the solution from which the caffeine was extracted, shake out three times with petroleum ether, boiling at  $40^{\circ}$  to  $60^{\circ}$  C., filter ether solution, divide into four parts, evaporate, and test for cocaine as described in the preceding method.

Evaporate the aqueous solution from the cocaine extraction with milk of lime and proceed as in the determination of glycerol in wines (page 734). The glycerol thus obtained will be only an approximation to the true amount.



## CHAPTER XXII.

### DETERMINATION OF ACIDITY BY MEANS OF THE HYDROGEN ELECTRODE.

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THE usual methods of determining acid and alkali are frequently inapplicable in food analysis because the color change of the indicator, by means of which the end point is observed, is masked by the presence of colored substances or by turbidity. In such cases the use of the hydrogen-electrode method is essential. Furthermore, there are many titrations in which it is not so important to know the total quantity of acid present as it is to determine exactly the concentration of hydrogen ions in the original solution. That is, it is desirable to determine the actual acidity as distinct from the total available acidity. A large part of the latter may be present originally in the form of undissociated molecules, and therefore inactive as acid until alkali is added. The hydrogen electrode method offers a simple means for such a determination. Finally, there are many occasions when the analyst wishes to prepare a solution of definite acidity which may be far from the neutral point, and yet also far from being so acid or so alkaline as to be readily analysed. In such cases also the hydrogen electrode is of great convenience in that it indicates constantly the actual acidity of a solution at any moment, and acid or alkali need therefore to be added only until the instrument records the proper concentration. All three problems are frequently met with in courses of food analysis.

In addition, indicator titrations are occasionally impossible because the salts present in the material to be titrated exert such a strong "buffer" action—as in milk, for instance, or in rhubarb juice—that the color change of the indicator is so gradual as to be quite unreliable. Related to this is the fact that the choice of the proper indicator is exceedingly important in such cases. The common indicators may all change at a degree of acidity which wholly unsuits them for some particular titration. And, lastly, the usual titration can give no information as to which acid or which



alkali is present in the solution that is being analysed. Thus fruit acids are empirically reported as per cent citric, malic or sulphuric regardless of the acid or mixture of acids present. On the other hand, the electro-metric method frequently reveals the characteristics of the acid that is being determined. For all these reasons, then, a simple form of the hydrogen electrode apparatus is to be recommended as part of the equipment of any food laboratory.

**Principle of the Method.**—The first principle involved in this method of determining acidity is that every aqueous solution must have a definite concentration of hydrogen ions. Even pure water can be regarded as an acid, and equally well as an alkali, in the sense that water contains both hydrogen and hydroxide ions in definite concentration. The dissociation of water into its ions is weak, yet accurate measurements have shown that about one water molecule in five hundred million is dissociated into its ions. In other words, the concentration of hydrogen ions in water is very close to  $10^{-7}$  grams per liter. Since each molecule of water on dissociation gives rise to one hydrogen ion and one hydroxide ion, the concentration of hydroxide ion in water is also  $10^{-7}$  gram ions per liter, i.e., both are  $10^{-7}$  normal. The product of the two concentrations is consequently  $10^{-14}$ .

According to the simple principles of chemical equilibrium these two concentrations must bear a definite ratio toward each other at all times. That is, in the reaction represented by the equation  $\text{H}_2\text{O} \rightleftharpoons \text{H}^+ + \text{OH}^-$ , definite equilibrium concentrations of the reacting substances must always be obtained such that the velocity of the forward reaction is the same as that of the reverse action. This can be the case only if the concentration of hydrogen ions multiplied by the concentration of hydroxide ions be constant; i.e., if, using the usual chemical symbols,  $(\text{H}^+)(\text{OH}^-) = K = 10^{-14}$ . This constant is, of course, the dissociation constant of water, and represents the product of the concentrations of hydrogen and hydroxide ions expressed in normality.

This constant must hold good for every solution which contains water, and thus for all aqueous solutions, irrespective of the amount of hydrogen or hydroxide ions added from other sources. That is to say, in a normal solution of a strong acid, the hydrogen ion concentration is one, or  $10^0$ ; substituted in the above equation, the hydroxide concentration must be  $10^{-14}$  in order that their product remain  $10^{-14}$ . Similarly a hundredth normal solution of a strong acid would have a concentration of hydroxide equal to  $10^{-12}$ . Strong acids therefore contain definite concentrations of hydroxide ions. At the other end of the scale, alkalis similarly contain



definite concentrations of hydrogen ions. The variation of hydrogen with hydroxide is represented in the following table:

TABLE I.

If $(H^+) = 10^1$	$(OH^-) = 10^{-15}$	$P_H = -1$
$10^0$ (N)	$10^{-14}$	0
$10^{-1}$ (0.1N)	$10^{-13}$	1
$10^{-2}$ (0.01N)	$10^{-12}$	2
$10^{-3}$	$10^{-11}$	3
$10^{-4}$	$10^{-10}$	4
$10^{-5}$	$10^{-9}$	5
$10^{-6}$	$10^{-8}$	6
$10^{-7}$	$10^{-7}$	7
$10^{-8}$	$10^{-6}$	8
$10^{-9}$	$10^{-5}$	9
$10^{-10}$	$10^{-4}$	10
$10^{-11}$	$10^{-3}$	11
$10^{-12}$	$10^{-2}$ (0.01N)	12
$10^{-13}$	$10^{-1}$ (0.1N)	13
$10^{-14}$	$10^0$ (N)	14
$10^{-15}$	$10^1$	15

The negative exponent of 10 in these concentration figures for the hydrogen ion is the distinguishing factor for each case and has come to be used directly in this sense under the name of " $P_H$ ."

Pure water stands in the center of this scale in that its hydrogen ion concentration is exactly equal to its hydroxide concentration, both being  $10^{-7}$ . Water is therefore neutral, being as acid as it is basic. This is the exact neutral point, though it is seldom indicated by the indicators in general use. Most indicators will give either their "acid color" or their "alkali color" in water solution. Methyl orange, for instance, shows alkali in pure water, and phenolphthalein shows acid in pure water. Litmus and rosolic acid are two indicators that do change at the neutral point.

While each indicator is suited for the determination of some *one* value of  $P_H$ , it is obvious that no one indicator can be used to *follow the change* of hydrogen and hydroxide ion concentration when a solution is titrated, i.e., when the hydrogen ion concentration may vary all the way from  $10^{-1}$  to  $10^{-14}$ . Each indicator has its own definite changing point, and will mark only the point at which that concentration is attained. The changing points for methyl orange and for phenolphthalein are indicated in Fig. 3. The hydrogen electrode method, however, records on a scale the concentration of hydrogen ions at all times, and it is thus possible to follow continuously the change beginning with strong acid all the way to



strong alkali or vice versa. The actual hydrogen ion concentration is indicated at every instant.

**Theory of the Method.**—The theory of the hydrogen electrode is familiar. Its details need not be discussed here as they are available in any text-book of physical chemistry. Essentially the method consists in the measurement of the voltage between a platinum electrode saturated with hydrogen and the acid solution. The platinum electrode is coated with a layer of platinum black which is allowed to become saturated with hydrogen gas. Hydrogen is readily soluble in platinum black so that such an electrode is essentially a "hydrogen electrode." That is, for all practical purposes it acts like a rod or electrode of hydrogen inserted into the solution. The other electrode, which is to make direct contact with the solution, i.e., with the hydrogen ions, must be one in which the transition from solution to the metallic connecting wire is made under definite and constant electrical conditions. For this purpose a "calomel cell" is used. Thereby the potential of the hydrogen ions is communicated to the rest of the system by, first, a solution of potassium chloride, then mercurous chloride, "calomel" solution, then calomel paste in contact with metallic mercury, which in turn contains the connecting wire.

In such a system the potential between the coated platinum or hydrogen electrode and the mercury is given by the equation:

$$E = 0.058 \log \left( \frac{1}{c} \right) + .283,$$

where  $c$  represents the actual concentration of hydrogen ions in the solution.

Transposing,  $\log c = - \left( \frac{E - .283}{.058} \right),$   
and

$$P_H = \left( \frac{E - .283}{.058} \right).$$

$E$  may then be read directly in volts on a voltmeter and the equation needs only to be solved for  $c$  or for  $P_H$  in order to give the hydrogen ion concentration or "actual acidity" of any unknown solution. The relation between  $E$  and  $c$  is linear, and the variation of one with the other may be calculated once for all and embodied in a table or curve, as is shown in the figures of this chapter. Indeed the voltmeter itself may conveniently be graduated to read in values of  $c$  directly, as well as in values of  $E$ . Titration consists then in following the change in  $E$  as  $c$  is varied by the addition of acid or alkali to the solution.



**The Apparatus.**—The necessary apparatus consists of the hydrogen electrode, the calomel cell, and a potentiometer arrangement to measure

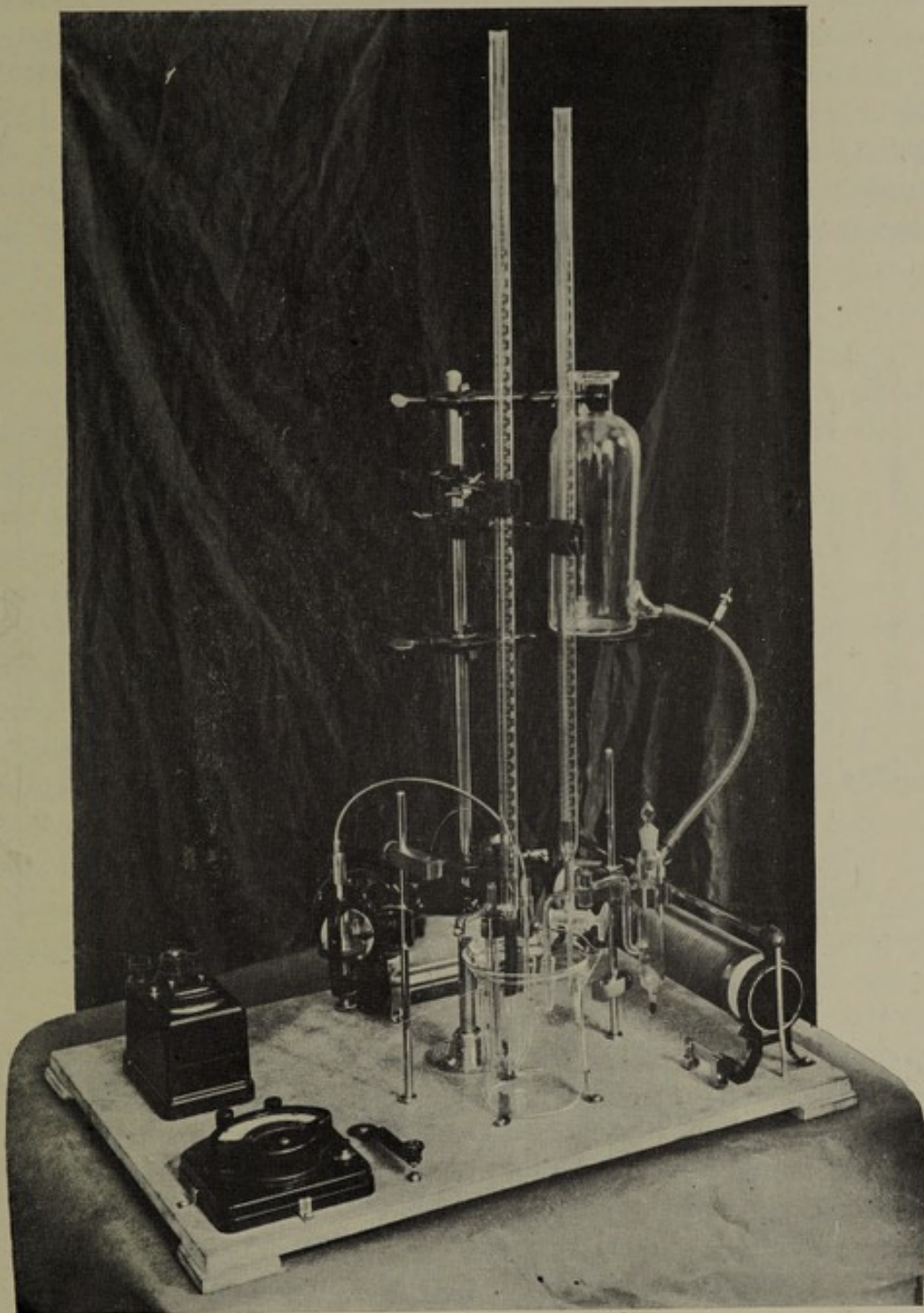
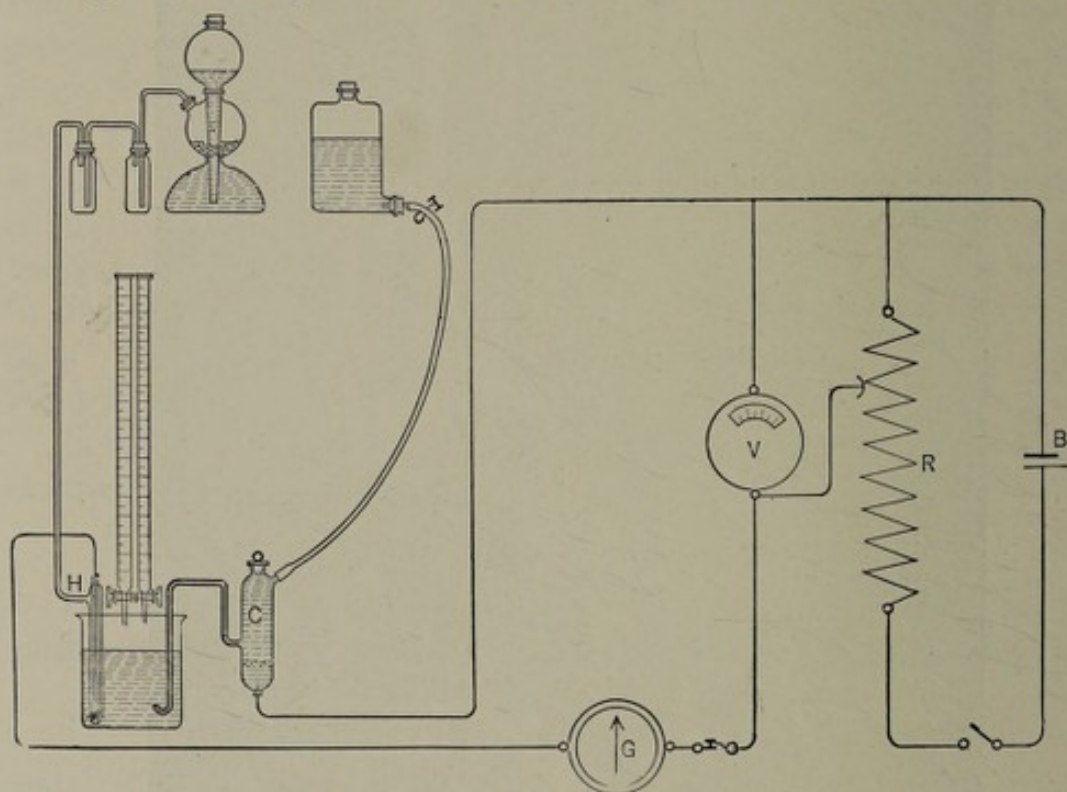


FIG. 1204.

the potential between them. For accurate work an accurate potentiometer is necessary, but for the usual laboratory determinations a potentiometer can



be readily built up from an ordinary dry cell, a 100-ohm variable resistance, a voltmeter, and a sensitive galvanometer which will determine when no current is passing. This galvanometer should have a sensitivity of one megohm. A diagram of the arrangement of these instruments is shown in Fig. 120*b* and a photograph of a typical assembly in Fig. 120*a*. *B* represents a dry cell, and is connected directly through the resistance *R*, and a knife switch to form a complete circuit which must be closed whenever the instrument is in use. By means of the variable contact on the resistance, various potentials may be drawn from this main circuit and sent through the

FIG. 120*b*.

side circuit, which comprises the calomel cell *C*, the solution to be titrated, the hydrogen electrode *H*, the galvanometer *G*, and a spring contact switch. The resistance is set at such a point that the galvanometer indicates the passage of no current. In that case, the potential being drawn from the main circuit is exactly equal and opposite to the potential from the hydrogen electrode-calomel electrode system. In order to measure this potential, a voltmeter *V* is placed in parallel with this side circuit, and at all times measures the potential. The procedure consists, therefore, simply in adjusting the resistance until on closing the contact key the galvanometer shows no current, and then reading the voltage. As suggested above, the



voltmeter may be graduated to read directly in units of hydrogen ion concentration rather than in volts.

*Details of the Apparatus.*—1. *The Hydrogen Electrode.*—This is a platinum wire 1 mm. in diameter, somewhat flattened at the lower end, which has been sealed into a small glass tube from which a copper wire leads to the rest of the circuit. This platinum wire must, when immersed in the solution, be about half covered by a larger tube which serves as a sort of bell to keep the upper half of the wire immersed in hydrogen gas while the lower half dips into the solution. The hydrogen is admitted to this bell-tube by a T-joint, and during operation bubbles continuously through the solution from under the bell at the rate of about two bubbles per second. The platinum wire must, before use and before insertion in the bell, be covered with a deposit of platinum black. This is done by first cleaning the wire thoroughly in chromic acid solution or in aqua regia if necessary. Platinum is then deposited on this wire by dipping it into a dilute solution of platinic chloride, and connecting the electrode to the negative pole of a dry cell, the positive being connected to another short piece of platinum wire which dips into the solution and forms the anode in the electrolysis. Deposition for fifteen minutes is ample, but it is desirable that the direction of the current be reversed frequently, as often as twice a minute, in order to give a smooth uniform coating of platinum, which should be black and velvety in appearance. The occluded chlorine may be removed by dipping the electrode into a ferrous or other reducing solution. The electrode is then washed with distilled water, and should thereafter always be kept moistened. When not in active use it should be kept in distilled water. If the electrode dries, the platinum deposit must be wiped with a dry cloth or more thoroughly removed, and a new layer of platinum black must be deposited. The electrode in this condition will absorb hydrogen from hydrogen gas and constitute in effect a hydrogen electrode. Time may be economized however, by saturating the electrode with hydrogen artificially by using this electrode as the cathode in an electrolysis of sulphuric acid. Hydrogen is thus evolved on the cathode, and serves to saturate it rapidly. This saturation should be repeated whenever the electrode is removed from its contact with pure hydrogen gas. The coating with platinum black should be serviceable for several weeks before it requires replacement. If solutions containing viscous materials or adhering precipitates are used, the platinum layer needs to be replaced more often.

This form of electrode may be purchased from supply houses. Other forms are also in use, particularly some in which the platinum is in the form



of a large foil or strip. This form is more stable in use but requires a much longer time to become saturated with hydrogen and is troublesome in solutions containing precipitates or other solid or viscous materials such as cream or fruit shreds. For such cases a fine platinum gauze may be attached to the glass bell that surrounds the electrode to prevent its clogging, particularly if some stirring device is used. A gold electrode coated with palladium black is probably the most effective form of hydrogen electrode.

2. *The Calomel Electrode.*—A calomel electrode may be made up by any of the various methods recommended in text-books of physical chemistry. The forms of cell used vary widely. The simplest is a test-tube with a two-hole rubber stopper, fitted with tubes, one of which leads to the solution to be titrated, and the other of which holds a glass tube forming electrical connection with the mercury in the bottom of the test-tube by means of a wire sealed through the glass. The form to be recommended is one in which electrical connection from the mercury is made by a platinum wire sealed directly through the glass where the latter holds the mercury. The tube should have a side-arm forming a bridge to the solution, and unless a fine capillary is used, this side-arm should contain a glass stop-cock which is kept loosely closed but not greased. The calomel electrode tube should have another side-arm placed somewhat higher through which additional potassium chloride solution can be introduced. Finally, it should have a wide opening through which it can be filled, but which should be tightly closed after filling by means of a well-fitting ground glass stopper.

To fill the cell it should be thoroughly cleaned and rinsed with a normal potassium chloride solution. About 3 cc. of carefully purified mercury are then placed in the bottom of the cell. Above this is put a layer of mercury-calomel paste. This is prepared by rubbing together in a mortar pure "calomel," mercurous chloride, and metallic mercury with a small amount of the potassium chloride solution. When this paste is in place it is covered with a normal solution of potassium chloride which has been saturated with calomel. A large quantity of accurately normal potassium chloride solution should be made up, and after preparation should be thoroughly shaken with calomel in order to saturate it with that substance. The calomel electrode tube should then be filled up with this solution, leaving only a small air bubble at the top. The tube should be well stoppered, and permanent connection should be made through the upper side-arm with a reservoir bottle containing the excess of potassium chloride



solution. Before each period of use a small quantity of the potassium chloride solution should be allowed to run through the calomel cell in order to wash out the lower side-arm, which constitutes the electrical connection with the solution to be titrated, and which will otherwise gradually become filled with materials from the latter solution by means of diffusion.

3. *The Electrical Instruments.*—Three electrical instruments are required, connected as shown in Fig. 120*b*, a variable resistance, a voltmeter, and a galvanometer. There are many varieties of resistances or rheostats on the market. Any form which permits the continuous variation of the resistance is satisfactory. The tubular wire rheostats of about 100 ohms total resistance are most convenient, but should be long enough to have at least 150 turns of wire in order to allow delicate adjustment of the end point. The voltmeter should have a total range of 1.25 volts, which is the maximum obtainable from a dry cell, and should be divided into hundredths of volts. The galvanometer, or other instrument used to detect the passage of current through the solution being titrated, is the only one of these instruments that needs to be sensitive. It should have a sensitivity of at least 1 megohm. There are several types of portable direct-reading galvanometers on the market with a sensitivity as great as this. By the use of the lamp and scale method, more sensitive and more expensive instruments may also be used with convenience. In very accurate work in which potential readings are to be made to millivolts, some form of electrometer is often used, such as the capillary electrometer or the quadrant electrometer. These are not required, however, for ordinary titration. The connections are shown in Fig. 120*b*, which is self-explanatory. It should be noted, however, that the short thick line of the battery, *B*, represents the positive or carbon pole of the dry cell. It is to this pole that the positive terminal of the voltmeter and the calomel cell connection are made.

Several complete assemblies of this apparatus are on the market. The most accurate, which can be depended upon to millivolts, is that designed by Dr. W. T. Bovie and manufactured by the Leeds & Northrup Co., of Philadelphia. This makes use of the quadrant electrometer, and is provided with a temperature compensating device. An apparatus designed by Dr. G. L. Kelley, can also be adapted to this purpose, and is sold by Arthur H. Thomas, of Philadelphia, Pa. The Central Scientific Co., of Chicago, has assembled the simplest forms of the various required instruments on a single board, the result being inexpensive and well suited for ordinary analytical work.



**The Titration.**—The solution to be titrated is placed in a sufficiently large beaker, the calomel electrode as above prepared is inserted, and the hydrogen electrode, platinized and saturated with hydrogen, is also inserted. A stream of hydrogen is allowed to pass over the hydrogen electrode and bubble through the solution continuously during the titration. The hydrogen should be pure, best made by electrolysis or generated from pure zinc and purified by passing through an alkaline solution. It is usually desirable to stir the solution by some extraneous stirring device. Electrical connections are made and preliminary observations of the voltage may be taken, although at the beginning of the titration the electrode is usually not saturated and does not give a constant voltage reading during the first few minutes. If the electrode has been saturated by means of the electrolysis of sulfuric acid, however, not more than a minute should be required to determine the original  $P_H$  or "actual acidity." If the solution is to be titrated acid or alkali may now be added, and readings of the voltage may be taken almost at once. The electrode action is most satisfactory when the potential is built upwards, that is, when alkali is added to acid. Beginning with a definite quantity of acid, the data to be recorded are the amounts of alkali added and the voltage at each addition. These data are best comprehended by plotting them graphically, recording voltage against cubic centimeters of alkali added. Several such curves are reproduced herewith.

**Typical Curves.**—Curve I, Fig. 1206, represents the titration of 25 cms. of tenth-normal hydrochloric acid solution with tenth-normal sodium hydroxide. Ordinates on this curve represent acidity. The voltage scale is represented on the left while the corresponding concentrations of hydrogen ions are given on the right. The voltage 0.69 is that of the neutral point, where  $(H^+) = (OH^-) = 10^{-7}$ . The higher the voltage, the lower the hydrogen ion concentration and the greater the alkalinity. The abscissæ represent volumes of the alkaline solution added.

The original voltage shown by Curve I is 0.35, which represents a tenth-normal hydrogen ion concentration. The curve shows that the first quantities of alkali added have little effect on the hydrogen ion concentration. The alkali is used up in the formation of sodium chloride and the fraction of the total acid used is so small that there is little change in the hydrogen ion concentration and in the voltage. As more and more acid is neutralized, however, every drop of alkali causes a correspondingly larger proportional change in the hydrogen ion concentration and the voltage rises more and more rapidly. When a voltage of .45 is attained and the acid



is less than one-thousandth normal the addition of a few drops of alkali causes so marked a change in the hydrogen ion concentration that the voltage rises rapidly and indeed abruptly. At 24.8 cc. of alkali, the potential rises to beyond the neutral point and this quantity of alkali therefore represents the total amount of acid originally present. This figure, 24.8, is the one that would be obtained by the usual indicator titration and represents the amount of tenth-normal alkali necessary to neutralize the acid originally present. The acid was therefore very slightly weaker than tenth-normal. Beyond this point the addition of alkali increases the hydroxide ion

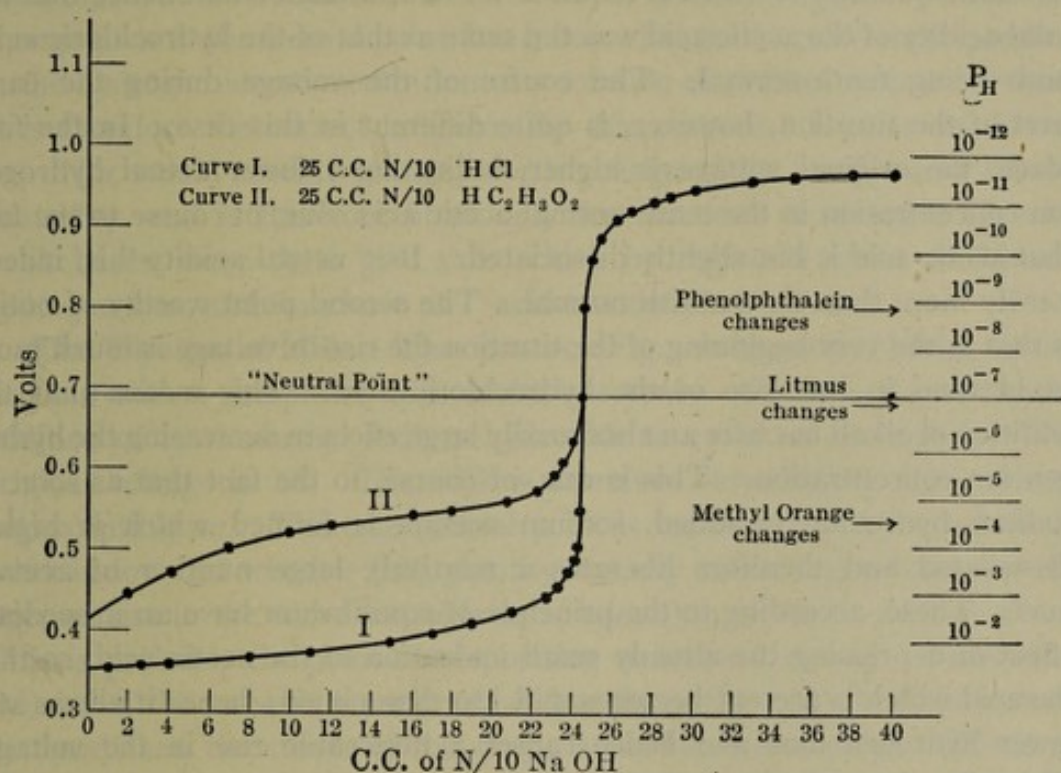


FIG. 120c.

concentration and decreases the hydrogen concentration in a lesser proportion and the voltage therefore rises more gradually, tending finally to reach the voltage of a tenth-normal alkali solution, which is slightly less than 1 volt. The further addition of alkali is, however, unnecessary, as the last part of the curve represents merely the dilution of tenth-normal alkali by the volume of solution present in the titrating vessel.

The center of the vertical rise in voltage at about 25 cc. of alkali needs further attention. It will be seen that the central point of the vertical line lies at a voltage of about .69. This is the voltage given by a strictly neutral solution and it indicates the point at which hydrogen and hydroxide



ions are in equal concentration. Since the central part of the vertical rise of this curve here falls exactly at the true neutral point it follows that the products of the reaction here used are such that they do not react with water to give a product which is itself acid or alkaline. The products of the particular reaction shown by this curve are, of course, sodium chloride and water, and it is obvious that sodium chloride does not hydrolyze to give an acid or a basic product.

Curve 2, Fig. 120c, shows the titration of the same quantity of tenth-normal acetic acid with the same alkaline solution. The curve shows that the same quantity of alkali is required for neutralization and hence that the total acidity of the acetic acid was the same as that of the hydrochloric acid, both being tenth-normal. The course of the voltage during the early part of the titration, however, is quite different in this case. In the first place, the original voltage is higher, indicating a lower actual hydrogen ion concentration in the tenth-normal acetic acid, due, of course to the fact that acetic acid is but slightly dissociated. Its "actual acidity" is, indeed hardly more than thousandth normal. The second point worthy of notice is that in the very beginning of the titration the rise in voltage is much more rapid than in the case of the hydrochloric acid. This means that the addition of alkali has here an abnormally large effect in decreasing the hydrogen ion concentration. This is due, of course, to the fact that as soon as sodium hydroxide is added, sodium acetate is formed which is highly dissociated and therefore liberates a relatively large number of acetate ions. These, according to the principle of equilibrium have an immediate effect in depressing the already small ionization of the acetic acid, so that the acid which is present becomes still less dissociated; hence it allows still fewer hydrogen ions and hence causes a noticeable rise in the voltage. This effect is marked only during the first 8 cc. and thereafter the trend of the curve is about the same as that in the titration of hydrochloric acid. Complete neutralization occurs at the same point, and the last part of the curve coincides with that of the hydrochloric acid since it represents only the dilution of the tenth-normal sodium hydroxide by the sodium acetate solution.

A third point to be noted is that the length of the vertical portion of this curve is much less than in the other case with the consequence that the center of this vertical portion lies not at a voltage of .69 but rather at about .76 volt. Now the center of the vertical portion represents the hydrogen ion concentration when exactly equivalent quantities of sodium hydroxide and of acetic acid are present; that is, it represents the conditions when only



water and sodium acetate are present. But sodium acetate hydrolyzes to some extent in water solution giving rise to undissociated acetic acid and thus allowing freedom to an excess number of hydroxide ions. In common terms, sodium acetate gives a basic reaction. This reaction is indicated on this curve by the fact that the center of the vertical position of the curve is at .76 volt, corresponding to a hydrogen ion concentration between  $10^{-8}$  and  $10^{-9}$ .

For this titration phenolphthalein is usually employed as indicator. The concentration of hydrogen ion, at which this indicator changes, is seen from Fig. 120c to be such that it is well suited for this purpose. It does not change color at the neutral point, but it does change at a point corresponding to the hydrogen ion concentration of a solution of sodium acetate in water. It is therefore the correct indicator for this purpose. Litmus would not do. Nor would methyl orange, which changes quite on the acid side of neutrality, and is therefore fitted only for titrations of strong acids with weak bases, since these give salts which hydrolyze in water to liberate hydrogen ions, and thus have an "acid reaction."

**The Titration of Milk.**—The curves on Fig. 120d represent titrations carried out with a sample of milk at various times. Curve 1 represents the addition of tenth-normal sodium hydroxide to 25 cc. of fresh milk. The original voltage is .68. The milk is therefore very slightly acid—almost imperceptibly so. The true neutral point is reached with 1.5 cc. of alkali, but it is doubtful whether this quantity has any actual significance, for the steepest rise in the curve comes later at about 5.5 cc. and a voltage of 0.8. This is probably the point corresponding to sodium lactate. Thereafter, the rise of the curve is more gradual, but the entire curve is notably more flattened than the curves of the strong acids heretofore used. This is no doubt due to the presence of salts, especially the salts of weak alkalis and weak acids like the calcium phosphates. The data obtained from this curve are more reliable than those obtained by the usual indicator titrations used in commercial laboratories. The first point, for instance, is usually determined by the use of litmus, which changes very gradually, and gives a much less accurate determination of the actual hydrogen ion concentration. The second point is determined by the use of phenolphthalein which changes at an acidity corresponding to a voltage of .8. The opacity of milk hinders an accurate determination of the color change.

The same sample of milk was kept on ice and its actual hydrogen ion concentration was determined from day to day. The voltage varied as follows:



On June 18. ....	.68
" 19. ....	.66
" 20. ....	.60
" 21. ....	.58
" 23. ....	.56
" 24. ....	.54

After a week, therefore, the acidity had increased so that the milk showed a hydrogen ion concentration of about  $10^{-4}$ , that is, the milk was ten-

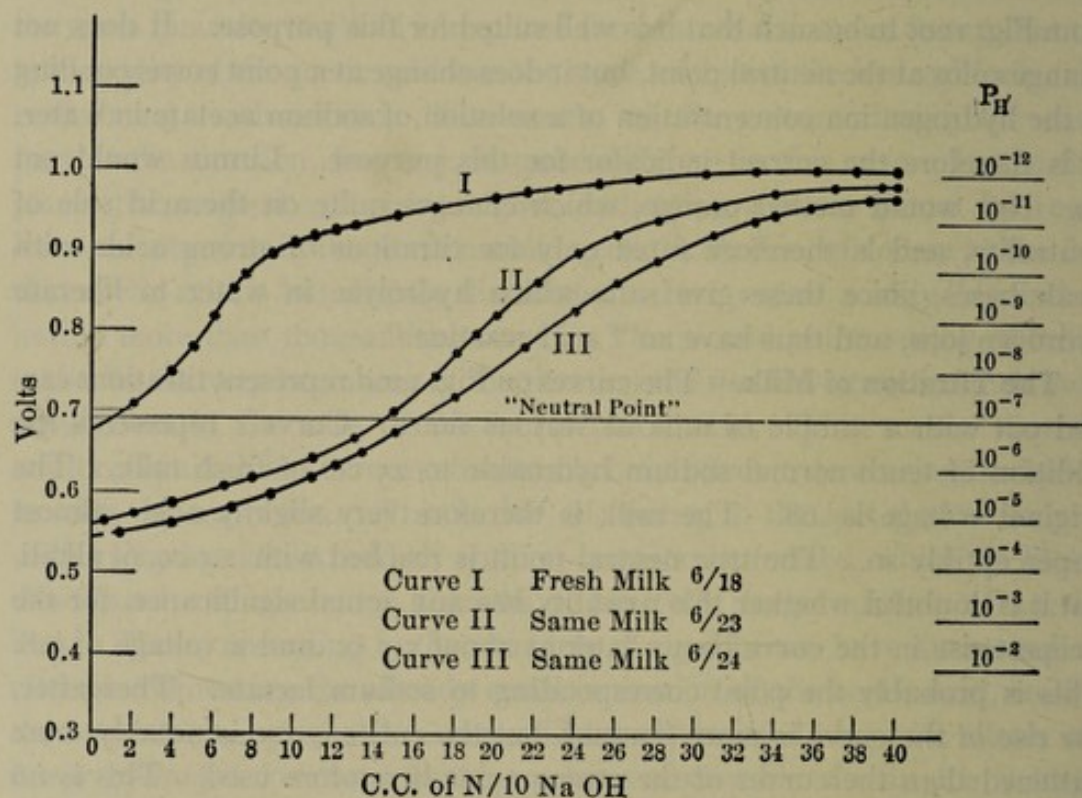


FIG. 120d.

thousandth normal in actual hydrogen ion concentration. This acidity, however, is due to lactic acid which is but slightly dissociated and the total acid present is undoubtedly more than this quantity. That this is true is shown by curves 2 and 3 of Fig. 120d, which represent titrations carried out on this sample on June 23 and June 24 respectively. The general form of these curves is the same as that of the first titration on this sample, being, however, somewhat more flattened. Total neutralization of the acid is reached again at about .8 volt corresponding to 18.6 cc. of alkali on June 23d. By the next day 22.3 cc. were required to reach the same voltage. The difference is a measure of the growth of lactic acid during the twenty-



four hours that had elapsed. The original voltage or "actual acidity" is, however, an equally good measure of the souring of the milk, and can be readily and speedily determined.

When milk has become sour or when it is rich in cream, the electrode tends to become clogged with the solid materials unless it is protected by a gauze as suggested above.

**Tea and Coffee.**—Fig. 120e represents the titration of samples of tea and coffee brews. Coffee is obviously more acid both in its actual hydrogen ion concentration, which is fairly high, and in its total acid. The curve

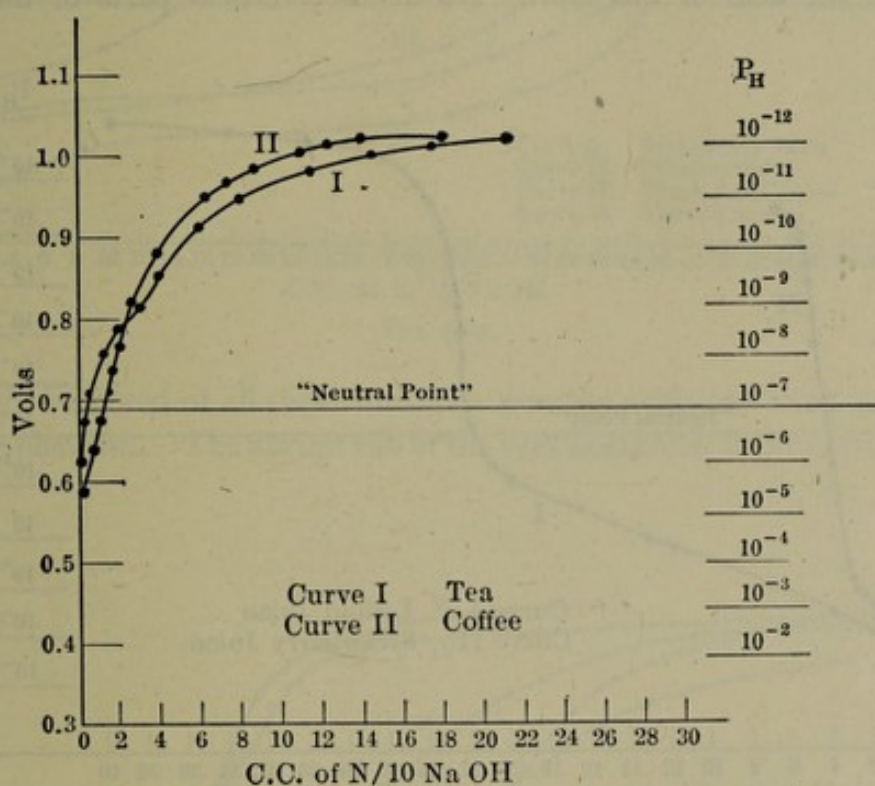


FIG. 120e.

for tea, on the other hand, is much flatter and indicates the presence of weaker acids and of more basic salts. The interesting portions of these titrations are in the voltages lying between 0.6 and 0.8 and the titration should be carried on with hundredth-normal alkali instead of with tenth-normal.

**The Acidity of Fruit Juices.**—For the titration of fruit juices, these are prepared in the usual way by pressing out the juice and straining through a fine cloth or filter. In all cases represented here by curves 25 cc. of fruit juice were used, though it is possible and often necessary to use a smaller quantity. *The analyses represented are single instances chosen at random and make no claim to being representative for the different varieties of fruit.*



*Lemon and Strawberry.*—Fig. 120f represents the titration of two common acid fruits, the lemon and the strawberry. It is noticeable that the actual acidity of the strawberry is greater than that of the lemon, being more than hundredth-normal. Yet the total acidity of the lemon is almost five times that of the strawberry. Twenty-five cc. of fruit juice were taken; hence the lemon is almost normal in total acid in that 22 cc. of normal alkali were required to neutralize it. The curve for the lemon is a typical curve for citric acid. The long sloping portion of the curve running from zero to 20 cc. represents the gradual neutralization of the three hydrogens that comprise the acid of this fruit. No distinct vertical parts of the curve

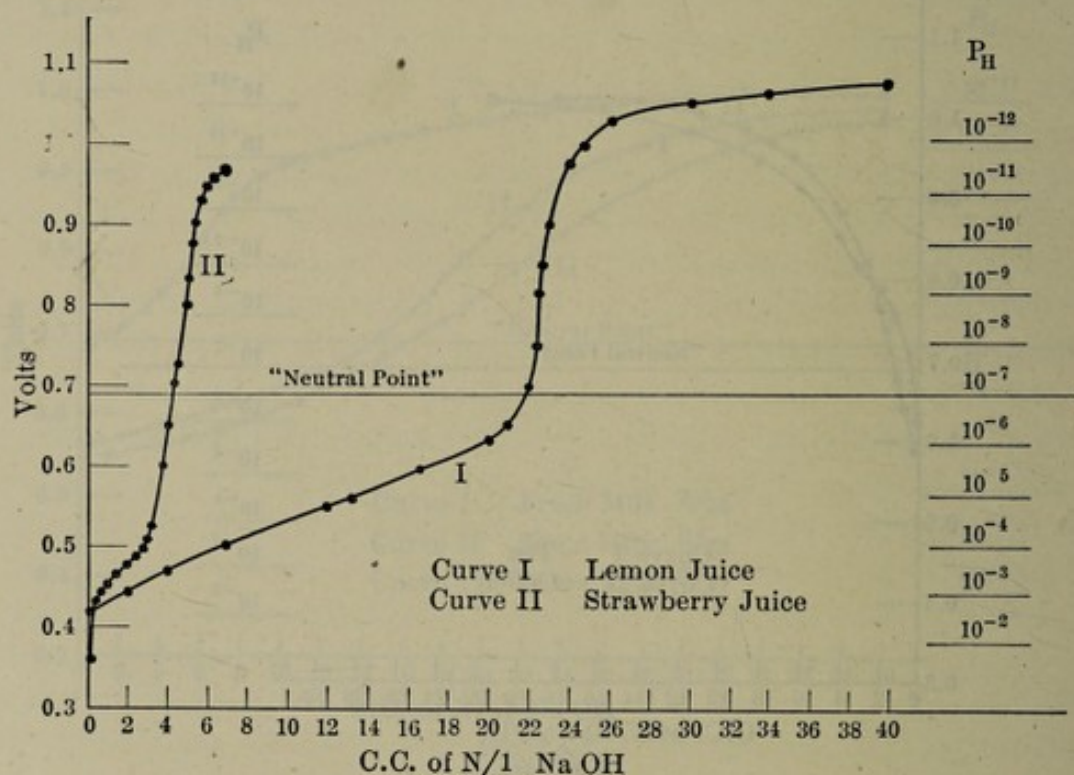


FIG. 120f.

are noticed because the reaction of the second hydrogen begins before that of the first is complete, and that of the third also begins very soon.

*The Citric Fruits.*—Fig. 120g shows this same titration of the strawberry as executed with one-tenth normal alkali. On the same figure appear the curves representing the orange, the grape fruit and the tomato. The strawberry is the most acid of these four fruits, especially in its actual acidity. Citric, salicylic and malic acids are present. The total acid of the orange is greater than that of the grapefruit though its actual acidity is less. The flat appearance of the orange curve marks the presence of other salts. The



general slanting appearance of the tomato curve is explained by the complexity of its acid constituents.

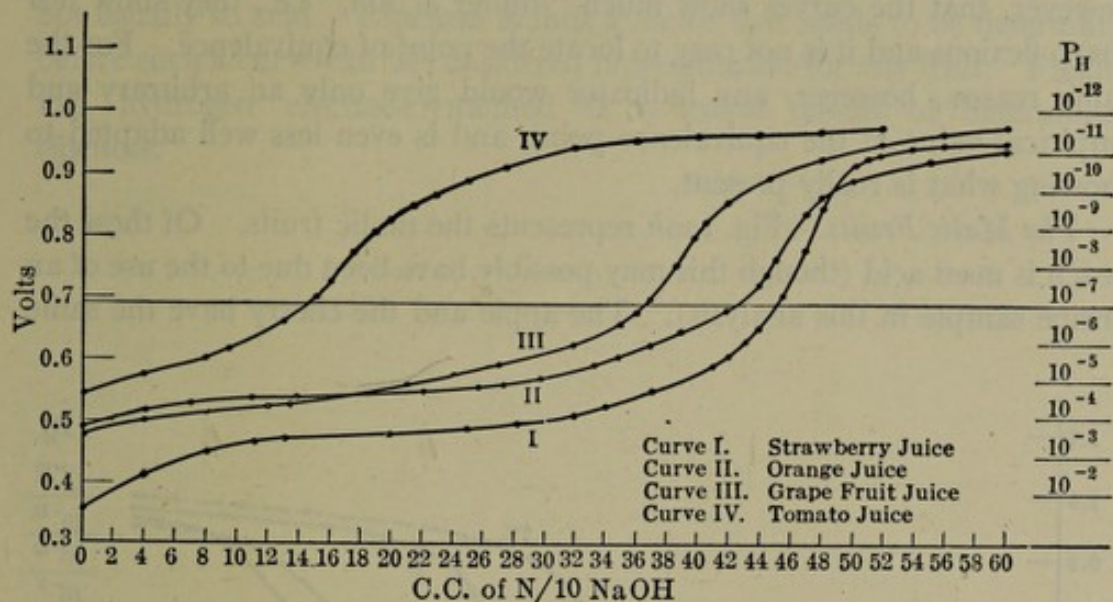


FIG. 120g.

It will be noted of all these titrations that the acids are weak and other salts are plentiful. The abrupt rise at the very beginning of the curve always

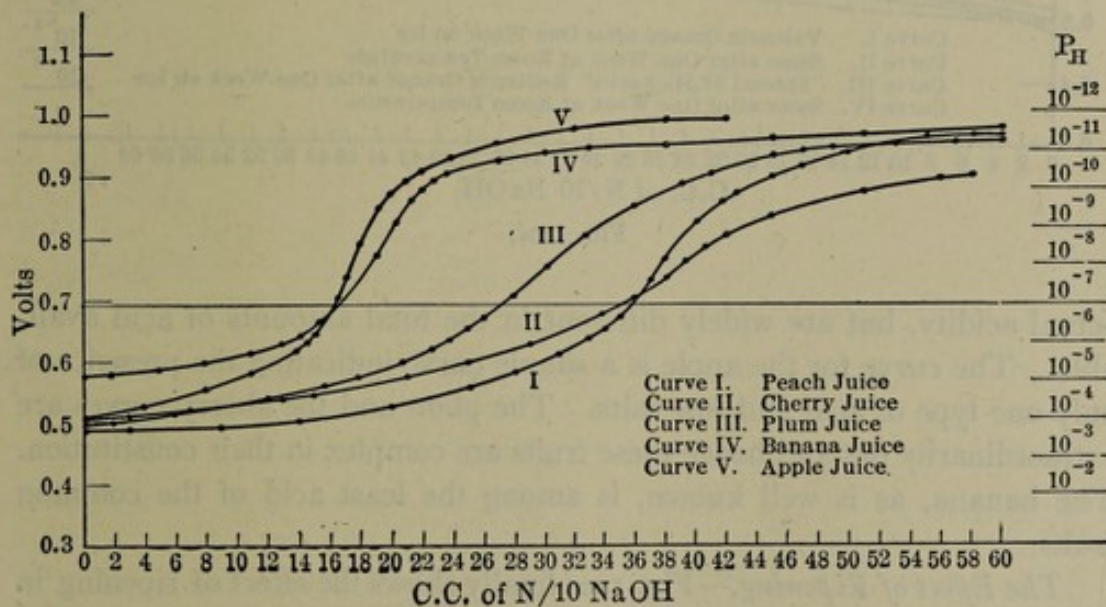


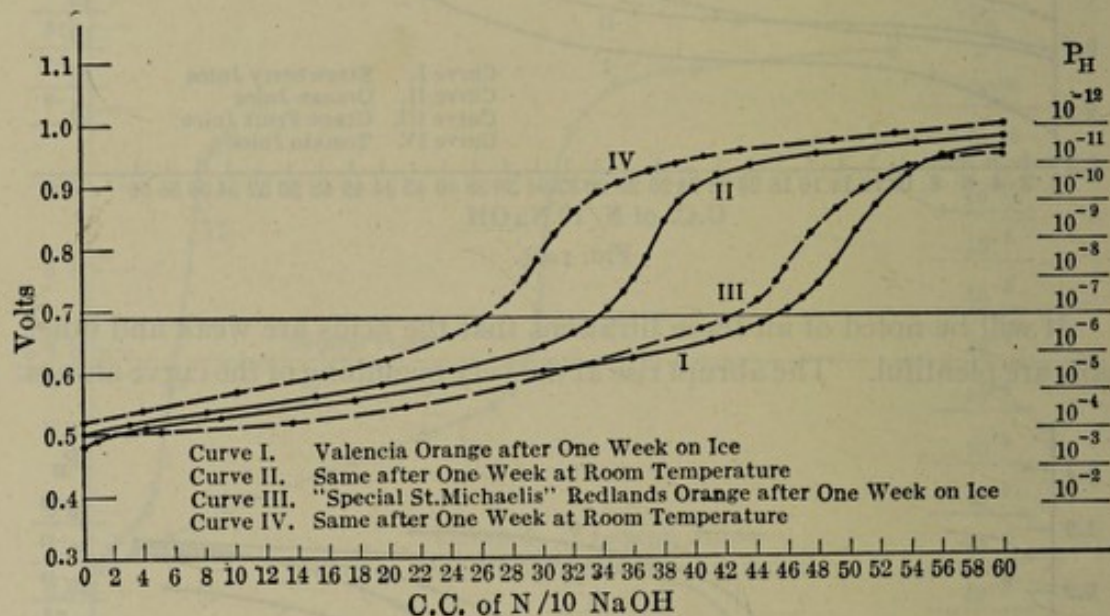
FIG. 120h.

points to the presence of an undissociated acid. In every case the center of the vertical portion of the curve lies on the alkaline side of the neutrality line. The exact position of this point would be a means of identifying the



acid present, for the weaker the acid the higher is the voltage given by the sodium salt in water. So many acids and other salts are present, however, that the curves show much "buffer action," i.e., they show few sharp flexions and it is not easy to locate the point of equivalence. For the same reason, however, any indicator would give only an arbitrary and empirical value of the equivalence point, and is even less well adapted to showing what is really present.

*The Malic Fruits.*—Fig. 120*h* represents the malic fruits. Of these the peach is most acid (though this may possibly have been due to the use of an unripe sample in this analysis). The apple and the cherry have the same

FIG. 120*i*.

actual acidity, but are widely different in the total amounts of acid available. The curve for the apple is a simple curve indicating the presence of only one type of acid and few salts. The plum and the cherry curves are extraordinarily flat and hence these fruits are complex in their constitution. The banana, as is well known, is among the least acid of the common fruits.

*The Effect of Ripening.*—Fig. 120*i* finally shows the effect of ripening in decreasing the fruit acidity. Here Curves 1 and 2 represent juice from two samples of Valencia orange, one of which was kept on ice for a week while the other was kept at room temperature. The marked change in acidity shows an effectual ripening in the second case. Precisely the same is shown by Curves 3 and 4 of this figure, which represents a similar experiment with

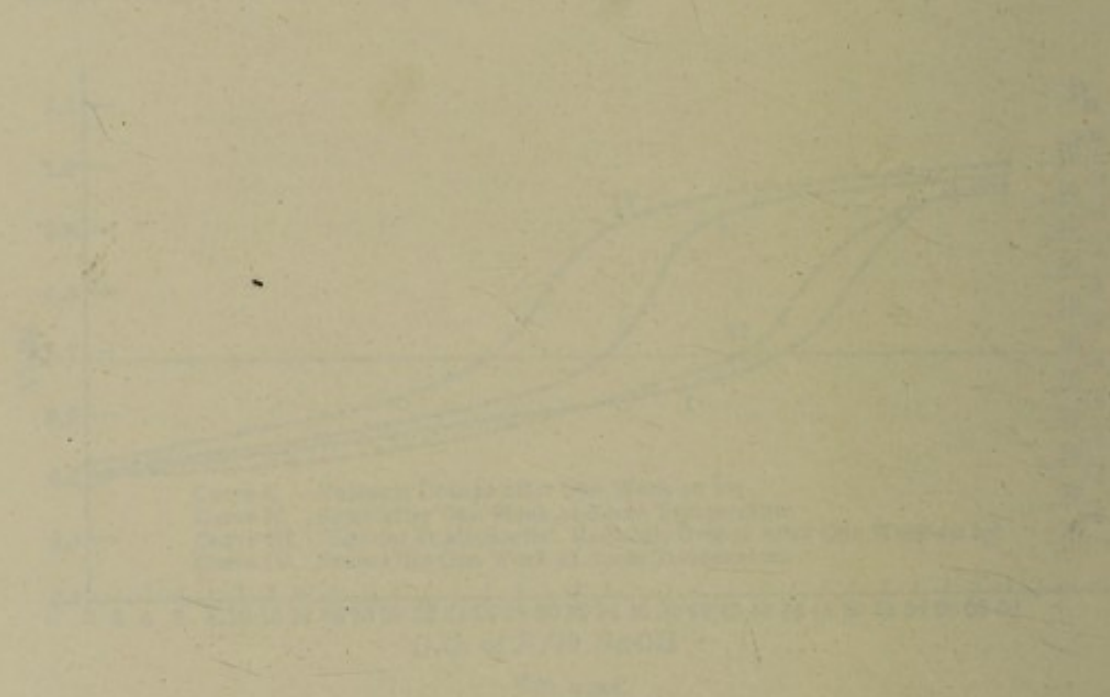


two samples of Redlands orange. The peaches represented in Fig. 120*h* appeared to be ripe but the curve indicates unripeness since this fruit is not usually so acid. Ripeness is thus a factor that needs to be determined before such a curve can be considered representative for any fruit. Finally, this hydrogen electrode method is a useful means of determining ripeness.



Two numbers of different signs. The product of two numbers of different signs is negative. The product of two numbers of the same sign is positive. The product of a number and its reciprocal is 1. The product of a number and its additive inverse is 0. The product of a number and its multiplicative inverse is 1. The product of a number and its additive inverse is 0. The product of a number and its multiplicative inverse is 1. The product of a number and its additive inverse is 0. The product of a number and its multiplicative inverse is 1.

The product of two numbers of different signs is negative. The product of two numbers of the same sign is positive. The product of a number and its reciprocal is 1. The product of a number and its additive inverse is 0. The product of a number and its multiplicative inverse is 1. The product of a number and its additive inverse is 0. The product of a number and its multiplicative inverse is 1.



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The product of two numbers of different signs is negative. The product of two numbers of the same sign is positive. The product of a number and its reciprocal is 1. The product of a number and its additive inverse is 0. The product of a number and its multiplicative inverse is 1. The product of a number and its additive inverse is 0. The product of a number and its multiplicative inverse is 1.



## APPENDIX.

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### THE FOOD AND DRUGS ACT, JUNE 30, 1906, AS AMENDED AUGUST 23, 1912 AND MARCH 3, 1913.

AN ACT FOR PREVENTING THE MANUFACTURE, SALE, OR TRANSPORTATION OF ADULTERATED OR MISBRANDED OR POISONOUS OR DELETERIOUS FOODS, DRUGS, MEDICINES, AND LIQUORS, AND FOR REGULATING TRAFFIC THEREIN, AND FOR OTHER PURPOSES.

*Be it enacted by the Senate and House of Representatives of the United States of America in Congress assembled,* That it shall be unlawful for any person to manufacture within any Territory or the District of Columbia any article of food or drug which is adulterated or misbranded, within the meaning of this Act; and any person who shall violate any of the provisions of this section shall be guilty of a misdemeanor, and for each offense shall, upon conviction thereof, be fined not to exceed five hundred dollars or shall be sentenced to one year's imprisonment, or both such fine and imprisonment, in the discretion of the court, and for each subsequent offense and conviction thereof shall be fined not less than one thousand dollars or sentenced to one year's imprisonment, or both such fine and imprisonment, in the discretion of the Court.

SEC. 2. That the introduction into any State or Territory or the District of Columbia from any other State or Territory or the District of Columbia, or from any foreign country, or shipment to any foreign country of any article of food or drugs which is adulterated or misbranded, within the meaning of this Act, is hereby prohibited; and any person who shall ship or deliver for shipment from any State or Territory or the District of Columbia to any other State or Territory or the District of Columbia, or to a foreign country, or who shall receive in any State or Territory or the District of Columbia from any other State or Territory or the District of Columbia, or foreign country, and having so received, shall deliver, in original unbroken packages, for pay or otherwise, or offer to deliver to any other person, any such article so adulterated or misbranded within the meaning of this Act, or any person who shall sell or offer for sale in the District of Columbia or the Territories of the United States any such adulterated or misbranded foods or drugs, or export or offer to export the same to any foreign country, shall be guilty of a misdemeanor, and for such offense be fined not exceeding two hundred dollars for the first offense, and upon conviction for each subsequent offense not exceeding three hundred dollars or be imprisoned not exceeding one year, or both, in the discretion of the court: *Provided*, That no article shall be deemed misbranded or adulterated within the provisions of this Act when intended for export to any foreign country and prepared or packed according to the specifications or directions of the foreign purchaser when no substance is used in the preparation or packing thereof in conflict with the laws of the foreign country to which said article is intended to be shipped; but if said article shall be in fact sold or offered for sale for domestic use or consumption, then this proviso shall not exempt said article from the operation of any of the other provisions of this Act.

SEC. 3. That the Secretary of the Treasury, the Secretary of Agriculture, and the Secretary of Commerce and Labor shall make uniform rules and regulations for carrying out the provisions of this act, including the collection and examination of specimens of foods and drugs manufactured or offered for sale in the District of Columbia, or in any



Territory of the United States, or which shall be offered for sale in unbroken packages in any State other than that in which they shall have been respectively manufactured or produced, or which shall be received from any foreign country, or intended for shipment to any foreign country, or which may be submitted for examination by the chief health, food, or drug officer of any State, Territory, or the District of Columbia, or at any domestic or foreign port through which such product is offered for interstate commerce, or for export or import between the United States and any foreign port or country.

SEC. 4. That the examinations of specimens of foods and drugs shall be made in the Bureau of Chemistry of the Department of Agriculture, or under the direction and supervision of such Bureau, for the purpose of determining from such examinations whether such articles are adulterated or misbranded within the meaning of this Act; and if it shall appear from any such examination that any of such specimens is adulterated or misbranded within the meaning of this Act, the Secretary of Agriculture shall cause notice thereof to be given to the party from whom such sample was obtained. Any party so notified shall be given an opportunity to be heard, under such rules and regulations as may be prescribed as aforesaid, and if it appears that any of the provisions of this Act have been violated by such party, then the Secretary of Agriculture shall at once certify the facts to the proper United States district attorney, with a copy of the results of the analysis or the examination of such article duly authenticated by the analyst or officer making such examination, under the oath of such officer. After judgment of the court, notice shall be given by publication in such manner as may be prescribed by the rules and regulations aforesaid.

SEC. 5. That it shall be the duty of each district attorney to whom the Secretary of Agriculture shall report any violation of this Act, or to whom any health or food or drug officer or agent of any State, Territory, or the District of Columbia shall present satisfactory evidence of any such violation, to cause appropriate proceedings to be commenced and prosecuted in the proper courts of the United States, without delay, for the enforcement of the penalties as in such case herein provided.

SEC. 6. That the term "drug," as used in this Act, shall include all medicines and preparations recognized in the United States Pharmacopœia or National Formulary for internal or external use, and any substance or mixture of substances intended to be used for the cure, mitigation, or prevention of disease of either man or other animals. The term "food," as used herein, shall include all articles used for food, drink, confectionery, or condiment by man or other animals, whether simple, mixed, or compound.

SEC. 7. That for the purposes of this Act an article shall be deemed to be adulterated:  
In case of drugs:

First. If, when a drug is sold under or by a name recognized in the United States Pharmacopœia or National Formulary, it differs from the standard of strength, quality, or purity, as determined by the test laid down in the United States Pharmacopœia or National Formulary official at the time of investigation: *Provided*, That no drug defined in the United States Pharmacopœia or National Formulary shall be deemed to be adulterated under this provision if the standard of strength, quality, or purity be plainly stated upon the bottle, box, or other container thereof although the standard may differ from that determined by the test laid down in the United States Pharmacopœia or National Formulary.

Second. If its strength or purity fall below the professed standard or quality under which it is sold.

In the case of confectionery:

If it contain terra alba, barytes, talc, chrome yellow, or other mineral substance or poisonous color or flavor, or other ingredient deleterious or detrimental to health, or any vinous, malt, or spirituous liquor or compound or narcotic drug.

In the case of food:



First. If any substance has been mixed and packed with it so as to reduce or lower or injuriously affect its quality or strength.

Second. If any substance has been substituted wholly or in part for the article.

Third. If any valuable constituent of the article has been wholly or in part abstracted.

Fourth. If it be mixed, colored, powdered, coated, or stained in a manner whereby damage or inferiority is concealed.

Fifth. If it contain any added poisonous or other added deleterious ingredient which may render such article injurious to health: *Provided*, That when in the preparation of food products for shipment they are preserved by any external application applied in such manner that the preservative is necessarily removed mechanically, or by maceration in water, or otherwise, and directions for the removal of said preservative shall be printed on the covering or the package, the provisions of this Act shall be construed as applying only when said products are ready for consumption.

Sixth. If it consists in whole or in part of a filthy, decomposed, or putrid animal or vegetable substance, or any portion of an animal unfit for food, whether manufactured or not, or if it is the product of a diseased animal, or one that has died otherwise than by slaughter.

SEC. 8. That the term "misbranded," as used herein, shall apply to all drugs, or articles of food, or articles which enter into the composition of food, the package or label of which shall bear any statement, design, or device regarding such article, or the ingredients or substances contained therein which shall be false or misleading in any particular, and to any food or drug product which is falsely branded as to the State, Territory, or country in which it is manufactured or produced.

That for the purposes of this Act an article shall also be deemed to be misbranded:

In case of drugs:

First. If it be an imitation of or offered for sale under the name of another article.

Second. If the contents of the package as originally put up shall have been removed, in whole or in part, and other contents shall have been placed in such package, or if the package fail to bear a statement on the label of the quantity or proportion of any alcohol, morphine, opium, cocaine, heroin, alpha or beta eucaine, chloroform, cannabis indica, chloral hydrate, or acetanilide, or any derivative or preparation of any such substances contained therein.

Third.\* If its package or label shall bear or contain any statement, design, or device regarding the curative or therapeutic effect of such article or any of the ingredients or substances contained therein, which is false and fraudulent.

In the case of food:

First. If it be an imitation of or offered for sale under the distinctive name of another article.

Second. If it be labeled or branded so as to deceive or mislead the purchaser, or purport to be a foreign product when not so, or if the contents of the package as originally put up shall have been removed in whole or in part and other contents shall have been placed in such package, or if it fail to bear a statement on the label of the quantity or proportion of any morphine, opium, cocaine, heroin, alpha or beta eucaine, chloroform, cannabis indica, chloral hydrate, or acetanilide, or any derivative or preparation of any of such substances contained therein.

Third.† If in package form, the quantity of the contents be not plainly and conspicuously marked on the outside of the package in terms of weight, measure, or numerical count: *Provided*, however, That reasonable variations shall be permitted, and tolerances and also exemptions as to small packages shall be established by rules and

\* This paragraph constitutes the amendment of August 23, 1912.

† This paragraph is as amended March 3, 1913.



regulations made in accordance with the provisions of Section three of this Act. That this Act shall take effect and be in force from and after its passage: *Provided*, however, That no penalty of fine, imprisonment, or confiscation shall be enforced for any violation of its provisions as to domestic products prepared or foreign products imported prior to eighteen months after its passage.

Fourth. If the package containing it or its label shall bear any statement, design, or device regarding the ingredients or the substances contained therein, which statement, design, or device shall be false or misleading in any particular: *Provided*, That an article of food which does not contain any added poisonous or deleterious ingredients shall not be deemed to be adulterated or misbranded in the following cases:

First. In the case of mixtures or compounds which may be now or from time to time hereafter known as articles of food, under their own distinctive names, and not an imitation of or offered for sale under the distinctive name of another article, if the name be accompanied on the same label or brand with a statement of the place where said article has been manufactured or produced.

Second. In the case of articles labeled, branded, or tagged so as to plainly indicate that they are compounds, imitations, or blends, and the word "compound," "imitation," or "blend," as the case may be, is plainly stated on the package in which it is offered for sale: *Provided*, That the term blend as used herein shall be construed to mean a mixture of like substances, not excluding harmless coloring or flavoring ingredients used for the purpose of coloring and flavoring only: *And provided further*, That nothing in this Act shall be construed as requiring or compelling proprietors or manufacturers of proprietary foods which contain no unwholesome added ingredient to disclose their trade formulas, except in so far as the provisions of this Act may require to secure freedom from adulteration or misbranding.

SEC. 9. That no dealer shall be prosecuted under the provisions of this Act when he can establish a guaranty signed by the wholesaler, jobber, manufacturer, or other party residing in the United States, from whom he purchases such articles, to the effect that the same is not adulterated or misbranded within the meaning of this Act, designating it. Said guaranty, to afford protection, shall contain the name and address of the party or parties making the sale of such articles to such dealer, and in such case said party or parties shall be amenable to the prosecutions, fines, and other penalties which would attach, in due course, to the dealer under the provisions of this Act.

SEC. 10. That any article of food, drug, or liquor that is adulterated or misbranded within the meaning of this Act, and is being transported from one State, Territory, District, or insular possession to another for sale, or, having been transported, remains unloaded, unsold, or in original unbroken packages, or if it be sold or offered for sale in the District of Columbia or the Territories, or insular possessions of the United States, or if it be imported from a foreign country for sale, or if it is intended for export to a foreign country, shall be liable to be proceeded against in any district court of the United States within the district where the same is found, and seized for confiscation by a process of libel for condemnation. And if such article is condemned as being adulterated or misbranded, or of a poisonous or deleterious character, within the meaning of this Act, the same shall be disposed of by destruction or sale, as the said court may direct, and the proceeds thereof, if sold, less the legal costs and charges, shall be paid into the Treasury of the United States, but such goods shall not be sold in any jurisdiction contrary to the provisions of this Act or the laws of that jurisdiction: *Provided however*, That upon the payment of the costs of such libel proceedings and the execution and delivery of a good and sufficient bond to the effect that such articles shall not be sold or otherwise disposed of contrary to the provisions of this Act, or the laws of any State, Territory, District, or insular possession, the court may by order direct that such articles be delivered to the owner thereof. The proceedings of such libel



cases shall conform, as near as may be, to the proceedings in admiralty, except that either party may demand trial by jury of any issue of fact joined in any such case, and all such proceedings shall be at the suit of and in the name of the United States.

SEC. 11. The Secretary of the Treasury shall deliver to the Secretary of Agriculture, upon his request from time to time, samples of foods and drugs which are being imported into the United States or offered for import, giving notice thereof to the owner or consignee, who may appear before the Secretary of Agriculture, and have the right to introduce testimony, and if it appear from the examination of such samples that any article of food or drug offered to be imported into the United States is adulterated or misbranded within the meaning of this Act, or is otherwise dangerous to the health of the people of the United States, or is of a kind forbidden entry into, or forbidden to be sold or restricted in sale in the country in which it is made or from which it is exported, or is otherwise falsely labeled in any respect, the said article shall be refused admission, and the Secretary of the Treasury shall refuse delivery to the consignee and shall cause the destruction of any goods refused delivery which shall not be exported by the consignee within three months from the date of notice of such refusal under such regulations as the Secretary of the Treasury may prescribe: *Provided*, That the Secretary of the Treasury may deliver to the consignee such goods pending examination and decision in the matter on execution of a penal bond for the amount of the full invoice value of such goods, together with the duty thereon, and on refusal to return such goods for any cause to the custody of the Secretary of the Treasury, when demanded, for the purpose of excluding them from the country, or for any other purpose, said consignee shall forfeit the full amount of the bond: *And provided further*, That all charges for storage, cartage, and labor on goods which are refused admission or delivery shall be paid by the owner or consignee, and in default of such payment shall constitute a lien against any future importation made by such owner or consignee.

SEC. 12. That the term "Territory" as used in this Act shall include the insular possessions of the United States. The word "person" as used in this Act shall be construed to import both the plural and the singular, as the case demands, and shall include corporations, companies, societies and associations. When construing and enforcing the provisions of this Act, the act, omission, or failure of any officer, agent, or other person acting for or employed by any corporation, company, society, or association, within the scope of his employment or office, shall in every case be also deemed to be the act, omission, or failure of such corporation, company, society, or association as well as that of the person.

SEC. 13. That this Act shall be in force and effect from and after the first day of January, nineteen hundred and seven.

#### THE MEAT-INSPECTION LAW.

EXTRACT FROM AN ACT OF CONGRESS ENTITLED "AN ACT MAKING APPROPRIATIONS FOR THE DEPARTMENT OF AGRICULTURE FOR THE FISCAL YEAR ENDING JUNE THIRTIETH, NINETEEN HUNDRED AND SEVEN," APPROVED JUNE 30, 1906.

That for the purpose of preventing the use in interstate or foreign commerce, as herein-after provided, of meat and meat food products which are unsound, unhealthful, unwholesome, or otherwise unfit for human food, the Secretary of Agriculture, at his discretion, may cause to be made, by inspectors appointed for that purpose, an examination and inspection of all cattle, sheep, swine, and goats before they shall be allowed to enter into any slaughtering, packing, meat-canning, rendering, or similar establishment, in which they are to be slaughtered and the meat and meat food products thereof are to be used in interstate or foreign commerce; and all cattle, swine, sheep, and goats found on such inspection to



show symptoms of disease shall be set apart and slaughtered separately from all other cattle, sheep, swine, or goats, and when so slaughtered the carcasses of said cattle, sheep, swine, or goats shall be subject to a careful examination and inspection, all as provided by the rules and regulations to be prescribed by the Secretary of Agriculture as herein provided for.

That for the purposes hereinbefore set forth the Secretary of Agriculture shall cause to be made by inspectors appointed for that purpose, as hereinafter provided, a post-mortem examination and inspection of the carcasses and parts thereof of all cattle, sheep, swine, and goats to be prepared for human consumption at any slaughtering, meat-canning, salting, packing, rendering, or similar establishment in any State, Territory, or the District of Columbia for transportation or sale as articles of interstate or foreign commerce, and the carcasses and parts thereof of all such animals found to be sound, healthful, wholesome, and fit for human food shall be marked, stamped, tagged, or labeled as "Inspected and Passed;" and said inspectors shall label, mark, stamp, or tag as "Inspected and Condemned," all carcasses and parts thereof of animals found to be unsound, unhealthful, unwholesome, or otherwise unfit for human food; and all carcasses and parts thereof thus inspected and condemned shall be destroyed for food purposes by the said establishment in the presence of an inspector, and the Secretary of Agriculture may remove inspectors from any such establishment which fails to so destroy any such condemned carcass or part thereof, and said inspectors, after said first inspection shall, when they deem it necessary, reinspect said carcasses or parts thereof to determine whether since the first inspection the same have become unsound, unhealthful, unwholesome, or in any way unfit for human food, and if any carcass or any part thereof shall, upon examination and inspection subsequent to the first examination and inspection, be found to be unsound, unhealthful, unwholesome, or otherwise unfit for human food, it shall be destroyed for food purposes by the said establishment in the presence of an inspector, and the Secretary of Agriculture may remove inspectors from any establishment which fails to so destroy any such condemned carcass or part thereof.

The foregoing provisions shall apply to all carcasses or parts of carcasses of cattle, sheep, swine, and goats, or the meat or meat products thereof which may be brought into any slaughtering, meat-canning, salting, packing, rendering, or similar establishment, and such examination and inspection shall be had before the said carcasses or parts thereof shall be allowed to enter into any department wherein the same are to be treated and prepared for meat food products; and the foregoing provisions shall also apply to all such products which, after having been issued from any slaughtering, meat-canning, salting, packing, rendering, or similar establishment, shall be returned to the same or to any similar establishment where such inspection is maintained.

That for the purposes hereinbefore set forth the Secretary of Agriculture shall cause to be made by inspectors appointed for that purpose an examination and inspection of all meat food products prepared for interstate or foreign commerce in any slaughtering, meat-canning, salting, packing, rendering, or similar establishment, and for the purposes or any examination and inspection said inspectors shall have access at all times, by day or night, whether the establishment be operated or not, to every part of said establishment; and said inspectors shall mark, stamp, tag, or label as "Inspected and Passed" all such products found to be sound, healthful, and wholesome, and which contain no dyes, chemicals, preservatives, or ingredients which render such meat or meat food products unsound, unhealthful, unwholesome, or unfit for human food; and said inspectors shall label, mark, stamp, or tag as "Inspected and Condemned" all such products found unsound, unhealthful, and unwholesome, or which contain dyes, chemicals, preservatives, or ingredients which render such meat or meat food products unsound, unhealthful, unwholesome, or unfit for human food, and all such condemned meat food products shall be destroyed for food pur-



poses, as hereinbefore provided, and the Secretary of Agriculture may remove inspectors from any establishment which fails to so destroy such condemned meat food products: *Provided*, That, subject to the rules and regulations of the Secretary of Agriculture, the provisions hereof in regard to preservatives shall not apply to meat food products for export to any foreign country and which are prepared or packed according to the specifications or directions of the foreign purchaser, when no substance is used in the preparation or packing thereof in conflict with the laws of the foreign country to which said article is to be exported; but if said article shall be in fact sold or offered for sale for domestic use or consumption, then this proviso shall not exempt said article from the operation of all the other provisions of this act.

That when any meat or meat food product prepared for interstate or foreign commerce which has been inspected as hereinbefore provided and marked "Inspected and Passed" shall be placed or packed in any can, pot, tin, canvas, or other receptacle or covering in any establishment where inspection under the provisions of this act is maintained, the person, firm, or corporation preparing said product shall cause a label to be attached to said can, pot, tin, canvas, or other receptacle or covering, under the supervision of an inspector, which label shall state that the contents thereof have been "Inspected and Passed" under the provisions of this act; and no inspection and examination of meat or meat food products deposited or inclosed in cans, tins, pots, canvas, or other receptacle or covering in any establishment where inspection under the provisions of this act is maintained shall be deemed to be complete until such meat or meat food products have been sealed or inclosed in said can, tin, pot, canvas, or other receptacle or covering under the supervision of an inspector, and no such meat or meat food products shall be sold or offered for sale by any person, firm, or corporation in interstate or foreign commerce under any false or deceptive name; but established trade name or names which are usual to such products and which are not false and deceptive and which shall be approved by the Secretary of Agriculture are permitted.

The Secretary of Agriculture shall cause to be made, by experts in sanitation or by other competent inspectors, such inspection of all slaughtering, meat-canning, salting, packing, rendering, or similar establishments in which cattle, sheep, swine, and goats are slaughtered and the meat and meat food products thereof are prepared for interstate or foreign commerce as may be necessary to inform himself concerning the sanitary conditions of the same, and to prescribe the rules and regulations of sanitation under which such establishments shall be maintained; and where the sanitary conditions of any such establishment are such that the meat or meat food products are rendered unclean, unsound, unhealthful, unwholesome, or otherwise unfit for human food, he shall refuse to allow said meat or meat food products to be labeled, marked, stamped, or tagged as "Inspected and Passed."

That the Secretary of Agriculture shall cause an examination and inspection of all cattle, sheep, swine, and goats, and the food products thereof, slaughtered and prepared in the establishments hereinbefore described for the purposes of interstate or foreign commerce to be made during the nighttime as well as during the daytime when the slaughtering of said cattle, sheep, swine, and goats, or the preparation of said food products is conducted during the nighttime.

That on and after October first, nineteen hundred and six, no person, firm, or corporation shall transport or offer for transportation, and no carrier of interstate or foreign commerce shall transport or receive for transportation from one State or Territory or the District of Columbia to any other State or Territory or the District of Columbia, or to any place under the jurisdiction of the United States, or to any foreign country, any carcasses or parts thereof, meat, or meat food products thereof which have not been inspected, examined, and marked as "Inspected and Passed," in accordance with the terms of this act and with the rules and



regulations prescribed by the Secretary of Agriculture: *Provided*, That all meat and meat food products on hand on October first, nineteen hundred and six, at establishments where inspection has not been maintained, or which have been inspected under existing law, shall be examined and labeled under such rules and regulations as the Secretary of Agriculture shall prescribe, and then shall be allowed to be sold in interstate or foreign commerce.

That no person, firm, or corporation, or officer, agent, or employee thereof, shall forge, counterfeit, simulate, or falsely represent, or shall without proper authority use, fail to use, or detach, or shall knowingly or wrongfully alter, deface, or destroy, or fail to deface or destroy, any of the marks, stamps, tags, labels, or other identification devices provided for in this act, or in and as directed by the rules and regulations prescribed hereunder by the Secretary of Agriculture, on any carcasses, parts of carcasses, or the food product, or containers thereof, subject to the provisions of this act, or any certificate in relation thereto, authorized or required by this act or by the said rules and regulations of the Secretary of Agriculture.

That the Secretary of Agriculture shall cause to be made a careful inspection of all cattle, sheep, swine, and goats intended and offered for export to foreign countries at such times and places, and in such manner as he may deem proper, to ascertain whether such cattle, sheep, swine, and goats are free from disease.

And for this purpose he may appoint inspectors who shall be authorized to give an official certificate clearly stating the condition in which such cattle, sheep, swine, and goats are found.

And no clearance shall be given to any vessel having on board cattle, sheep, swine, or goats for export to a foreign country until the owner or shipper of such cattle, sheep, swine, or goats has a certificate from the inspector herein authorized to be appointed, stating that the said cattle, sheep, swine, or goats are sound and healthy, or unless the Secretary of Agriculture shall have waived the requirement of such certificate for export to the particular country to which such cattle, sheep, swine, or goats are to be exported.

That the Secretary of Agriculture shall also cause to be made a careful inspection of the carcasses and parts thereof of all cattle, sheep, swine, and goats, the meat of which, fresh, salted, canned, corned, packed, cured, or otherwise prepared, is intended and offered for export to any foreign country, at such times and places and in such manner as he may deem proper.

And for this purpose he may appoint inspectors who shall be authorized to give an official certificate stating the condition in which said cattle, sheep, swine, or goats, and the meat thereof, are found.

And no clearance shall be given to any vessel having on board any fresh, salted, canned, corned, or packed beef, mutton, pork, or goat meat, being the meat of animals killed after the passage of this act, or except as hereinbefore provided for export to and sale in a foreign country from any port in the United States, until the owner or shipper thereof shall obtain from an inspector appointed under the provisions of this act a certificate that the said cattle, sheep, swine, and goats were sound and healthy at the time of inspection, and that their meat is sound and wholesome, unless the Secretary of Agriculture shall have waived the requirements of such certificate for the country to which said cattle, sheep, swine and goats or meats are to be exported.

That the inspectors provided for herein shall be authorized to give official certificates of the sound and wholesome condition of the cattle, sheep, swine, and goats, their carcasses and products as herein described, and one copy of every certificate granted under the provisions of this act shall be filed in the Department of Agriculture, another copy shall be delivered to the owner or shipper, and when the cattle, sheep, swine, and goats or their carcasses and products are sent abroad, a third copy shall be delivered to the chief officer of the vessel on which the shipment shall be made.



That no person, firm, or corporation engaged in the interstate commerce of meat or meat food products shall transport or offer for transportation, sell or offer to sell any such meat or meat food products in any State or Territory or in the District of Columbia or any place under the jurisdiction of the United States, other than in the State or Territory or in the District of Columbia or any place under the jurisdiction of the United States in which the slaughtering, packing, canning, rendering, or other similar establishment owned, leased, operated by said firm, person, or corporation is located unless and until said person, firm, or corporation shall have complied with all of the provisions of this act.

That any person, firm, or corporation, or any officer or agent of any such person, firm, or corporation, who shall violate any of the provisions of this act shall be deemed guilty of a misdemeanor, and shall be punished on conviction thereof by a fine of not exceeding ten thousand dollars or imprisonment for a period not more than two years, or by both such fine and imprisonment, in the discretion of the court.

That the Secretary of Agriculture shall appoint from time to time inspectors to make examination and inspection of all cattle, sheep, swine, and goats, the inspection of which is hereby provided for, and of all carcasses and parts thereof, and of all meats and meat food products thereof, and of the sanitary conditions of all establishments in which such meat and meat food products hereinbefore described are prepared; and said inspectors shall refuse to stamp, mark, tag, or label any carcass or any part thereof, or meat food product therefrom, prepared in any establishment hereinbefore mentioned, until the same shall have actually been inspected and found to be sound, healthful, wholesome, and fit for human food, and to contain no dyes, chemicals, preservatives, or ingredients which render such meat food product unsound, unhealthful, unwholesome, or unfit for human food; and to have been prepared under proper sanitary conditions, hereinbefore provided for; and shall perform such other duties as are provided by this act and by the rules and regulations to be prescribed by said Secretary of Agriculture; and said Secretary of Agriculture shall, from time to time, make such rules and regulations as are necessary for the efficient execution of the provisions of this act, and all inspections and examinations made under this act shall be such and made in such manner as described in the rules and regulations prescribed by said Secretary of Agriculture not inconsistent with the provisions of this act.

That any person, firm, or corporation, or any agent or employee of any person, firm, or corporation, who shall give, pay, or offer, directly or indirectly, to any inspector, deputy inspector, chief inspector, or any other officer or employee of the United States authorized to perform any of the duties prescribed by this act or by the rules and regulations of the Secretary of Agriculture any money or other thing of value, with intent to influence said inspector, deputy inspector, chief inspector, or other officer or employee of the United States in the discharge of any duty herein provided for, shall be deemed guilty of a felony and, upon conviction thereof, shall be punished by a fine not less than five thousand dollars nor more than ten thousand dollars and by imprisonment not less than one year nor more than three years; and any inspector, deputy inspector, chief inspector, or other officer or employee of the United States authorized to perform any of the duties prescribed by this act who shall accept any money, gift, or other thing of value from any person, firm, or corporation, or officers, agents, or employees thereof, given with intent to influence his official action, or who shall receive or accept from any person, firm, or corporation engaged in interstate or foreign commerce any gift, money, or other thing of value given with any purpose or intent whatsoever, shall be deemed guilty of a felony and shall, upon conviction thereof, be summarily discharged from office and shall be punished by a fine not less than one thousand dollars nor more than ten thousand dollars and by imprisonment not less than one year nor more than three years.

That the provisions of this act requiring inspection to be made by the Secretary of



Agriculture shall not apply to animals slaughtered by any farmer on the farm and sold and transported as interstate or foreign commerce, nor to retail butchers and retail dealers in meat and meat food products, supplying their customers: *Provided*, That if any person shall sell or offer for sale or transportation for interstate or foreign commerce any meat or meat food products which are diseased, unsound, unhealthful, unwholesome, or otherwise unfit for human food, knowing that such meat food products are intended for human consumption, he shall be guilty of a misdemeanor, and on conviction thereof shall be punished by a fine not exceeding one thousand dollars or by imprisonment for a period of not exceeding one year, or by both such fine and imprisonment: *Provided also*, That the Secretary of Agriculture is authorized to maintain the inspection in this act provided for at any slaughtering, meat canning, salting, packing, rendering, or similar establishment notwithstanding this exception, and that the persons operating the same may be retail butchers and retail dealers or farmers; and where the Secretary of Agriculture shall establish such inspection then the provisions of this act shall apply notwithstanding this exception.

That there is permanently appropriated, out of any money in the Treasury not otherwise appropriated, the sum of three million dollars, for the expenses of the inspection of cattle, sheep, swine, and goats and the meat and meat food products thereof which enter into interstate or foreign commerce and for all expenses necessary to carry into effect the provisions of this act relating to meat inspection, including rent and the employment of labor in Washington and elsewhere, for each year. And the Secretary of Agriculture shall, in his annual estimates made to Congress, submit a statement in detail, showing the number of persons employed in such inspections and the salary or per diem paid to each, together with the contingent expenses of such inspectors and where they have been and are employed.



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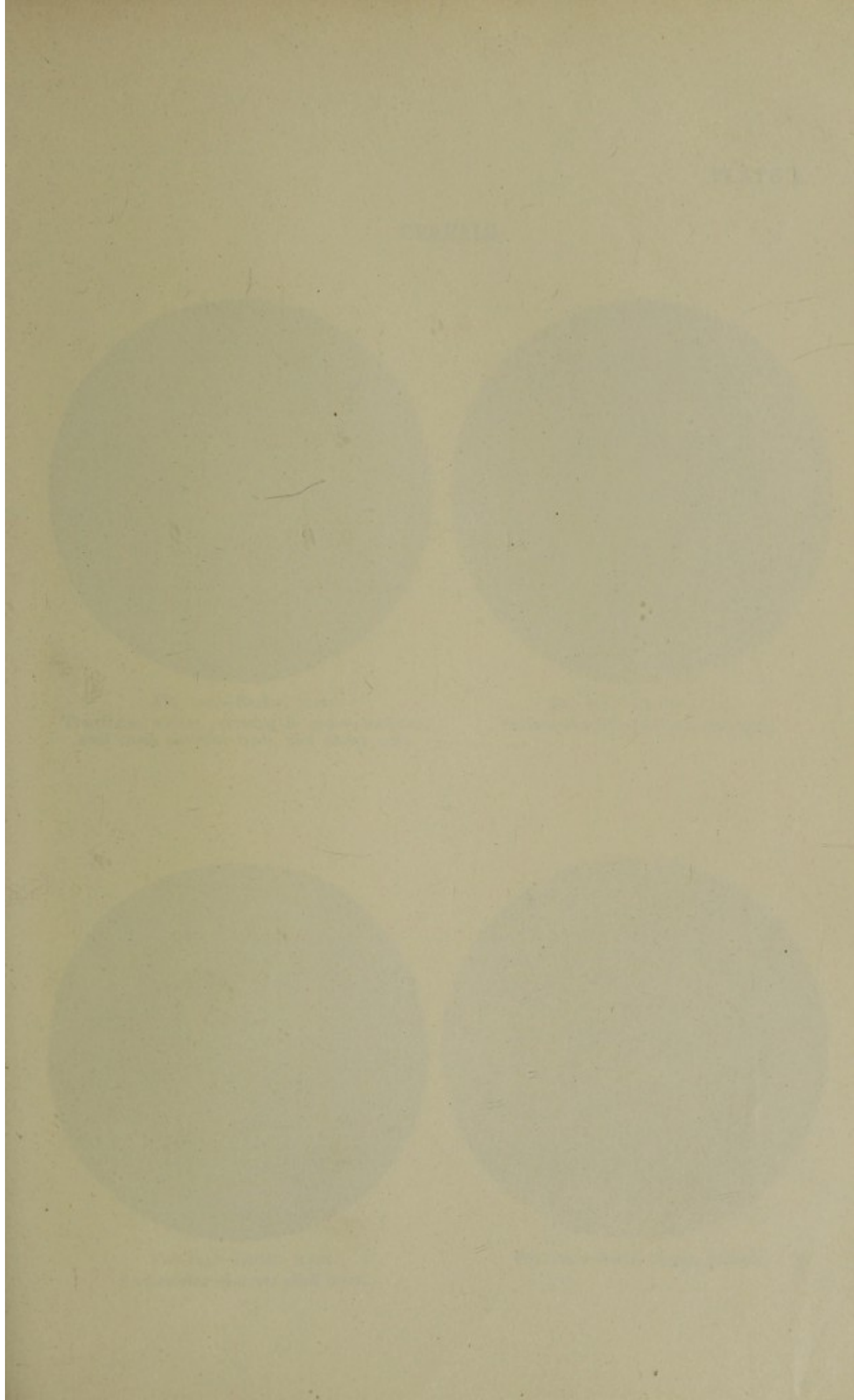
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CEREALS.

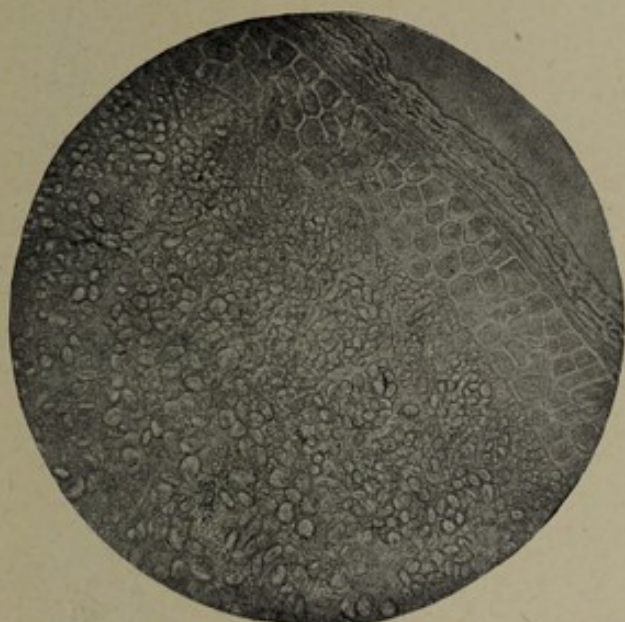


FIG. 121.—Barley,  $\times 110$ .  
Transverse section, showing in order, pericarp,  
seed coats, aleurone layer, and starch cells.

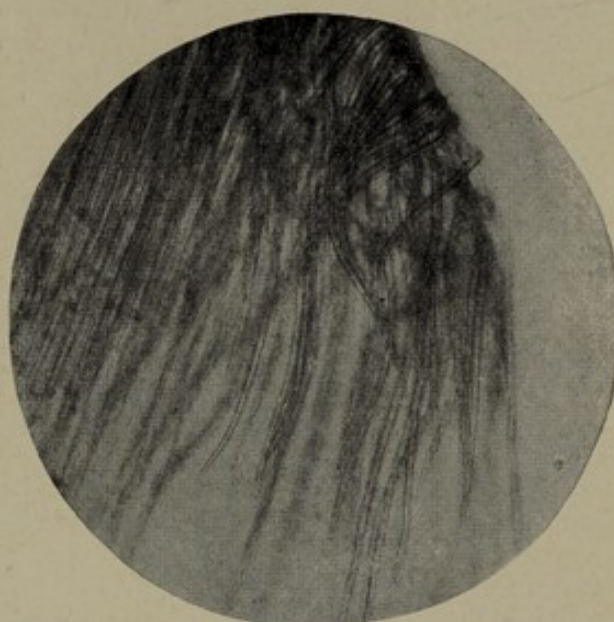


FIG. 122.—Barley,  $\times 55$ .  
Surface view of epidermis with hairs.

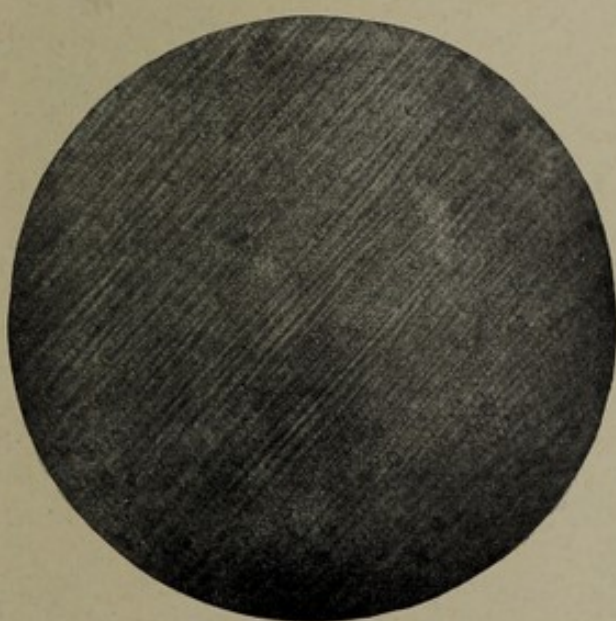


FIG. 123.—Barley,  $\times 125$ .  
Surface view of upper chaff layer.

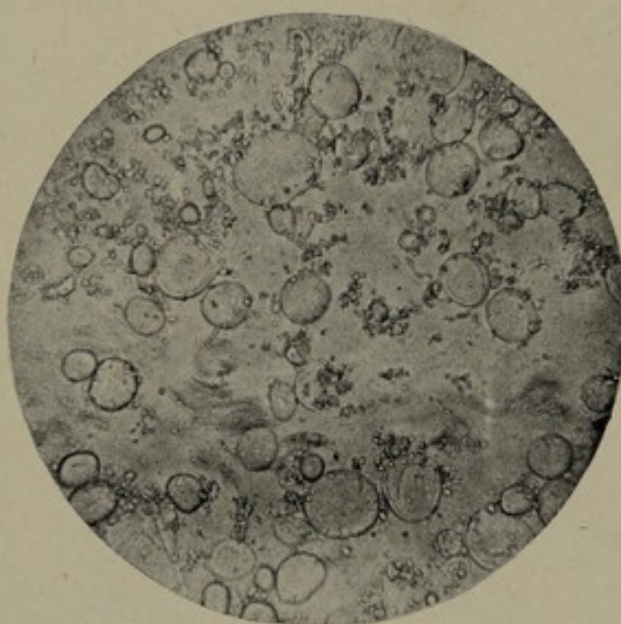
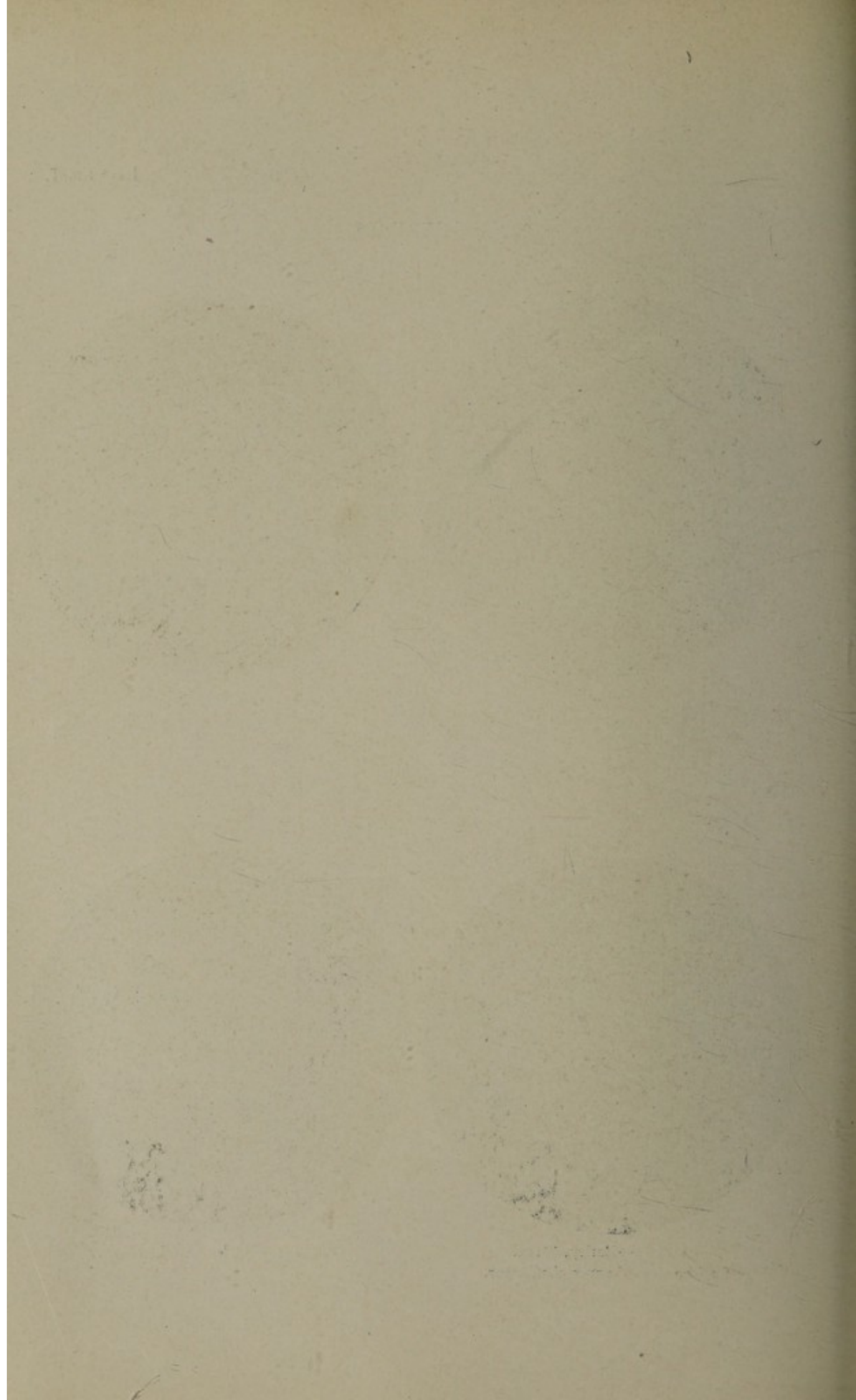


FIG. 124.—Barley Starch,  $\times 220$ .







CEREALS.

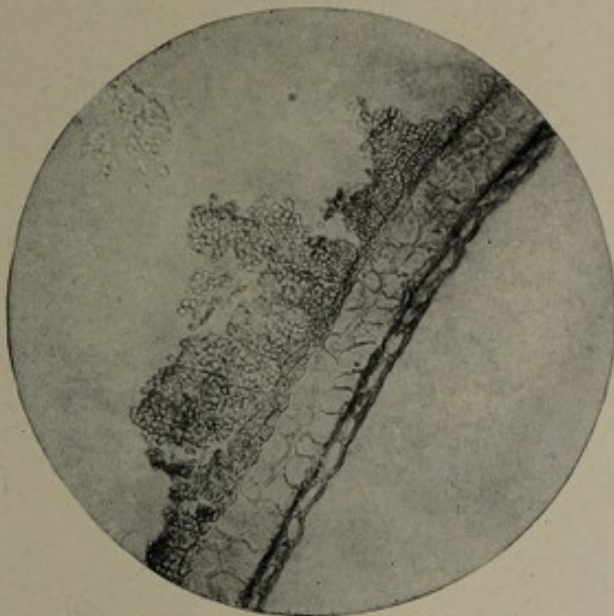


FIG. 125.—Buckwheat,  $\times 110$ .  
Transverse section through part of pericarp, seed  
coat, and part of endosperm.

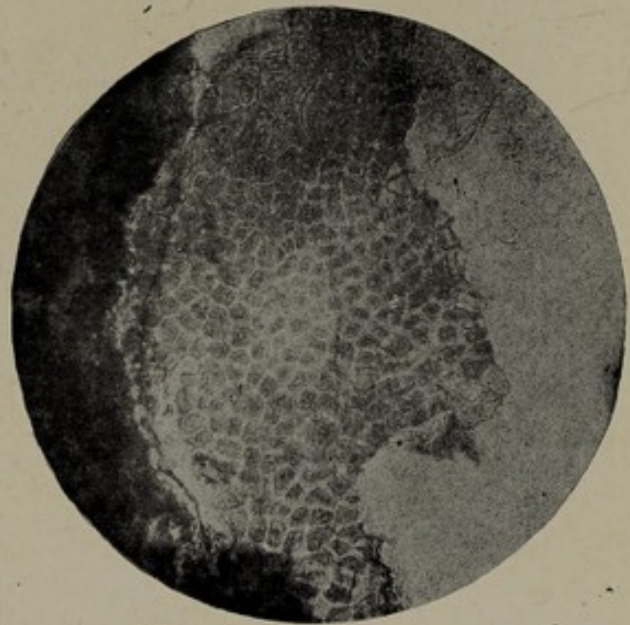


FIG. 126.—Buckwheat,  $\times 110$ .  
Surface view of scutellum.

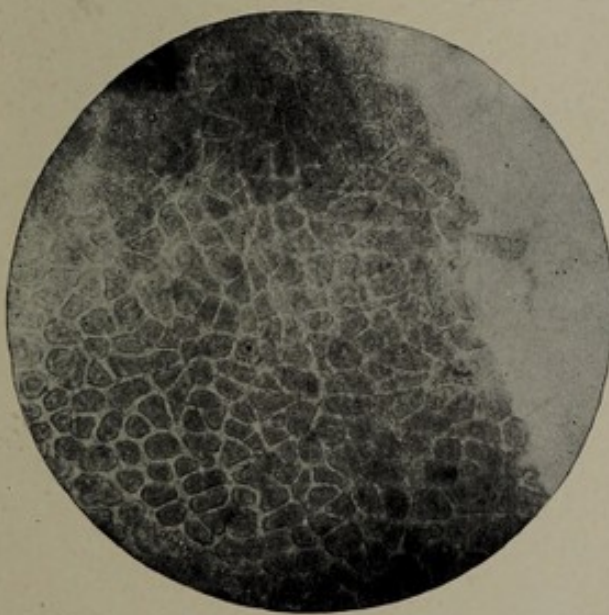


FIG. 127.—Buckwheat,  $\times 110$ .  
Surface section. Aleurone or proteid layer.

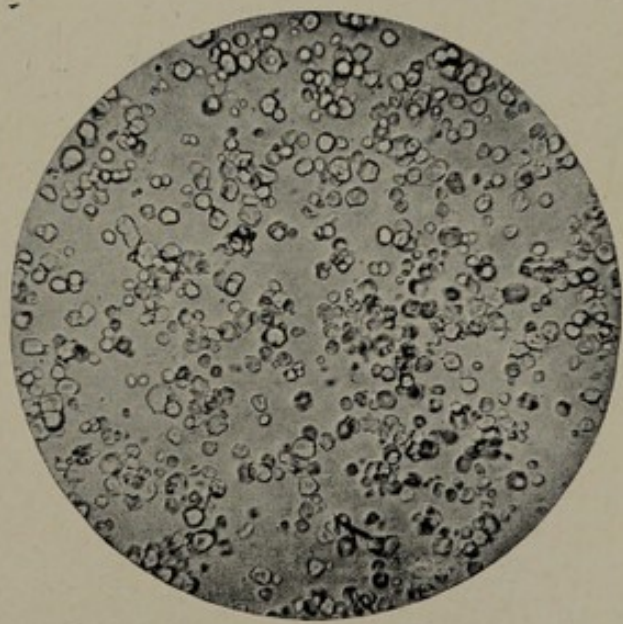
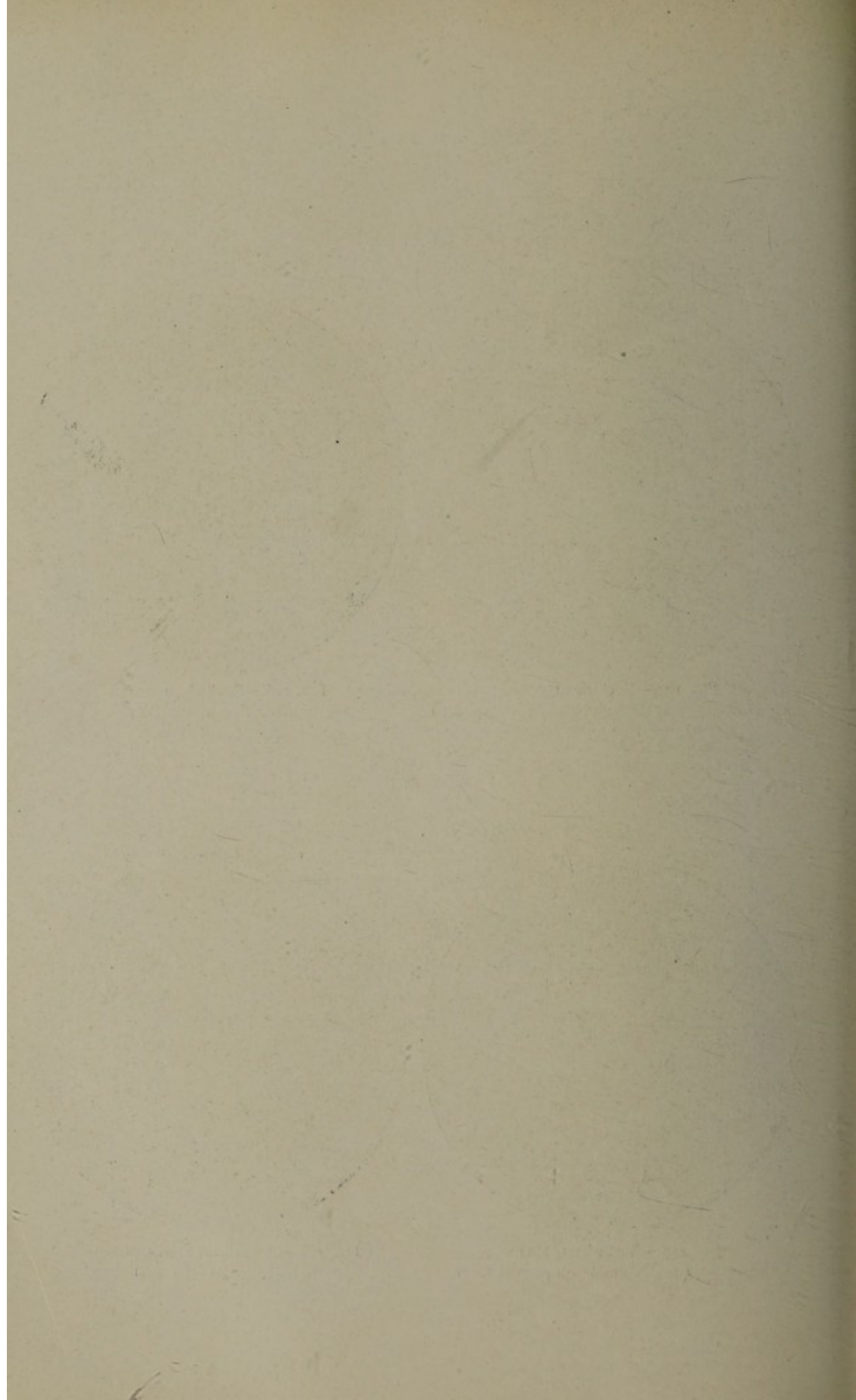


FIG. 128.—Buckwheat Starch,  $\times 220$ .  
Starch granules separated.







CEREALS.

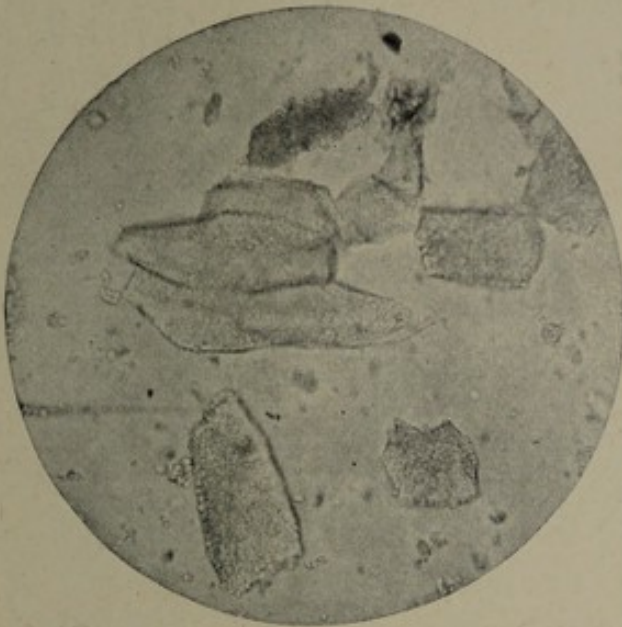


FIG. 129.—Buckwheat Starch,  $\times 110$ .  
Starch grains in masses.



FIG. 130.—Corn,  $\times 110$ .  
Transverse section through pericarp, seed coat, proteid layer, and part of endosperm, showing starch cells.

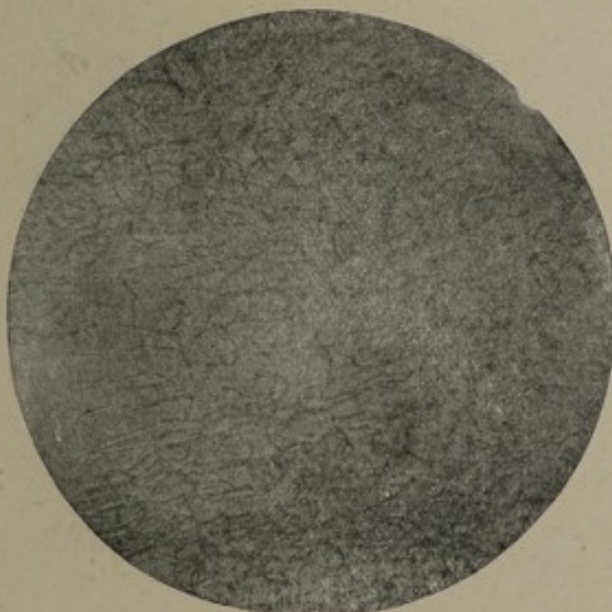


FIG. 131.—Corn,  $\times 110$ .  
Surface view showing two layers of the mesocarp.

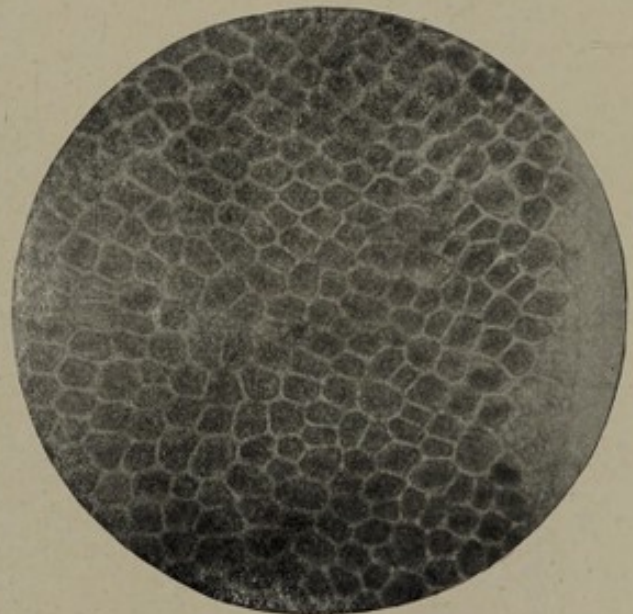
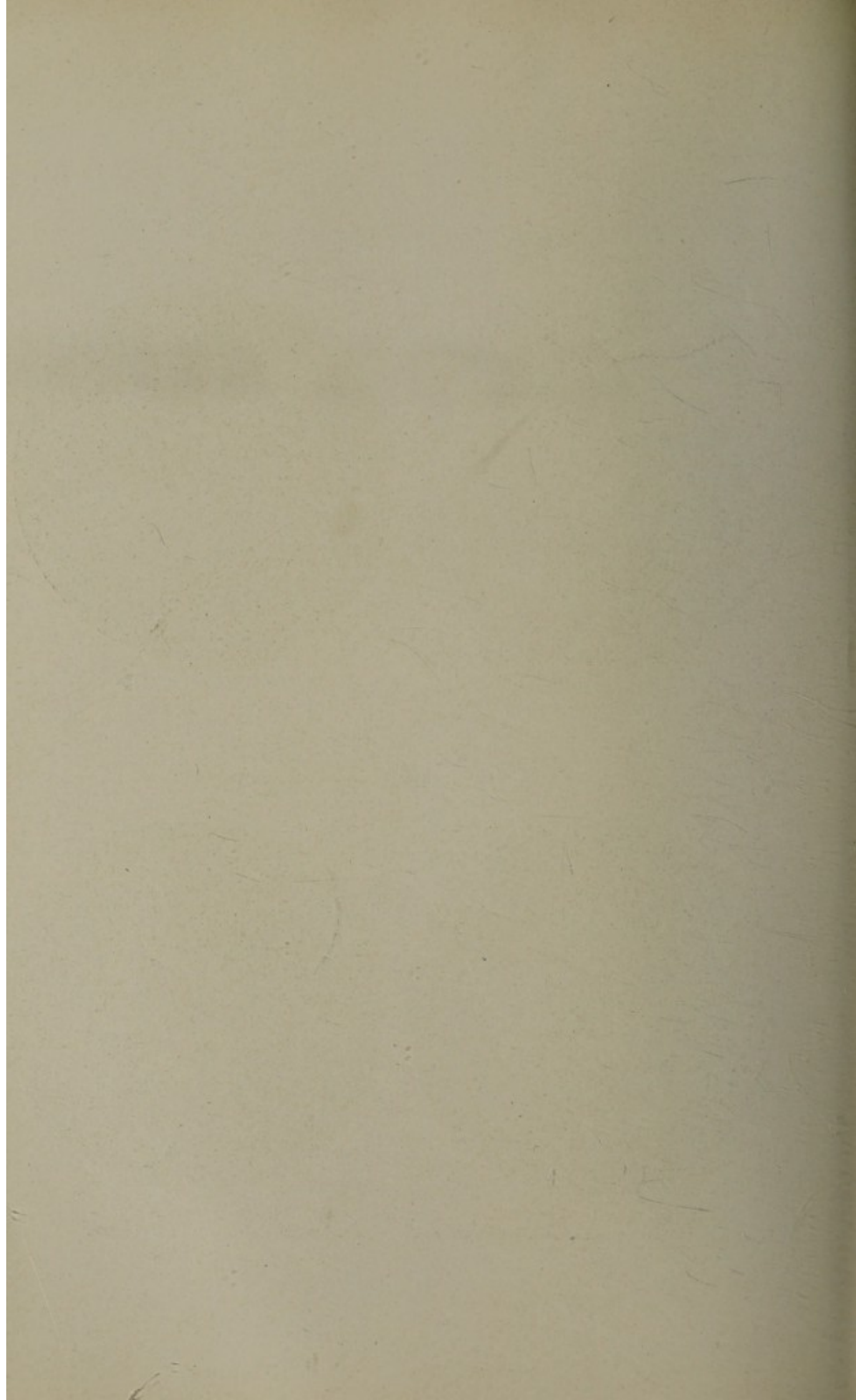


FIG. 132.—Corn,  $\times 110$ .  
Surface section. Proteid layer.







CEREALS.

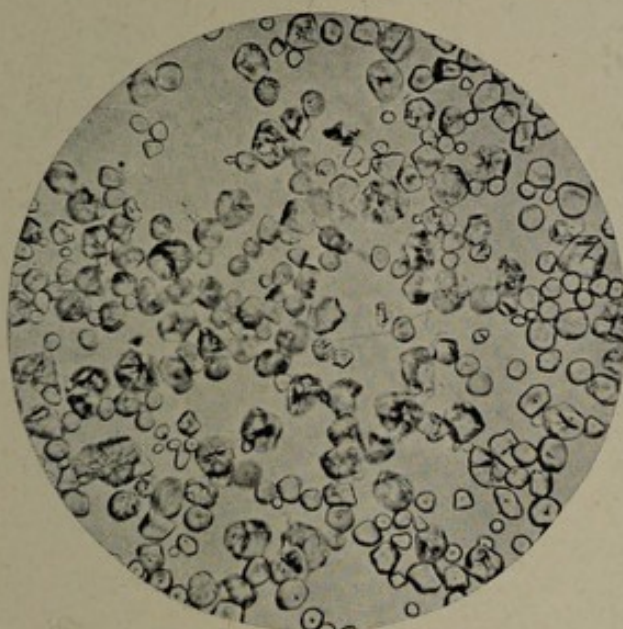


FIG. 133.—Corn Starch,  $\times 220$ .

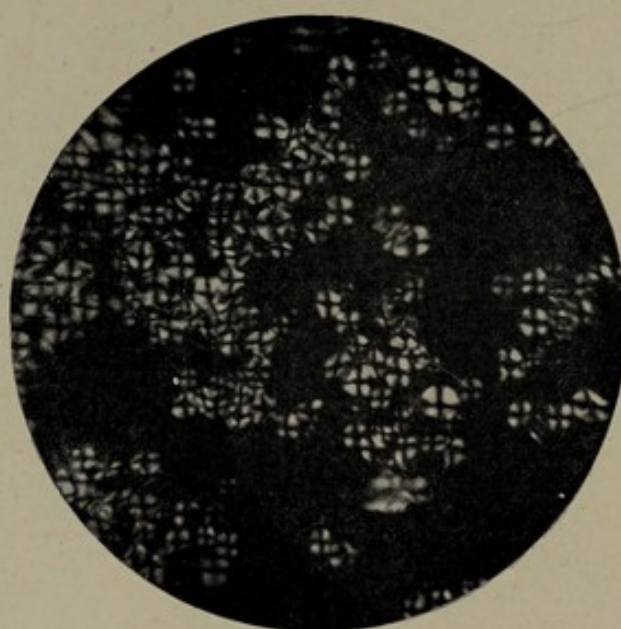


FIG. 134.—Corn Starch,  $\times 220$ .  
With polarized light.

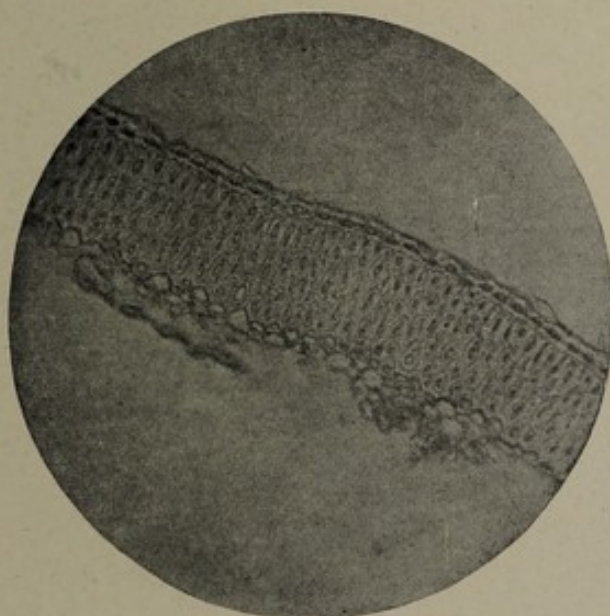


FIG. 135.—Oat,  $\times 110$ .  
Transverse section through chaff.

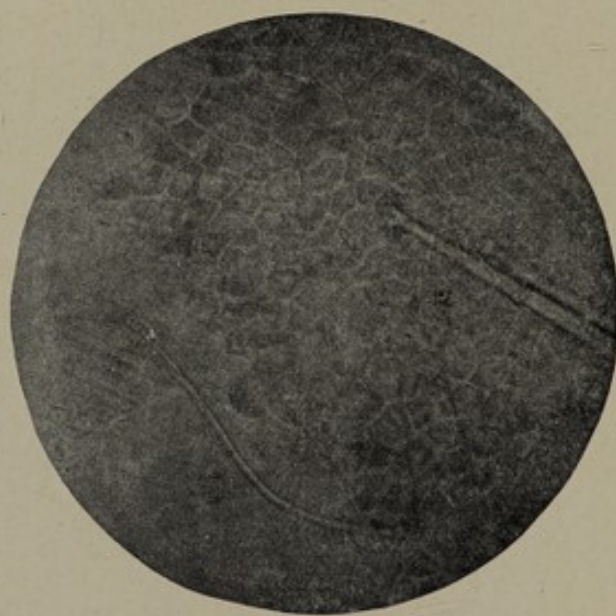
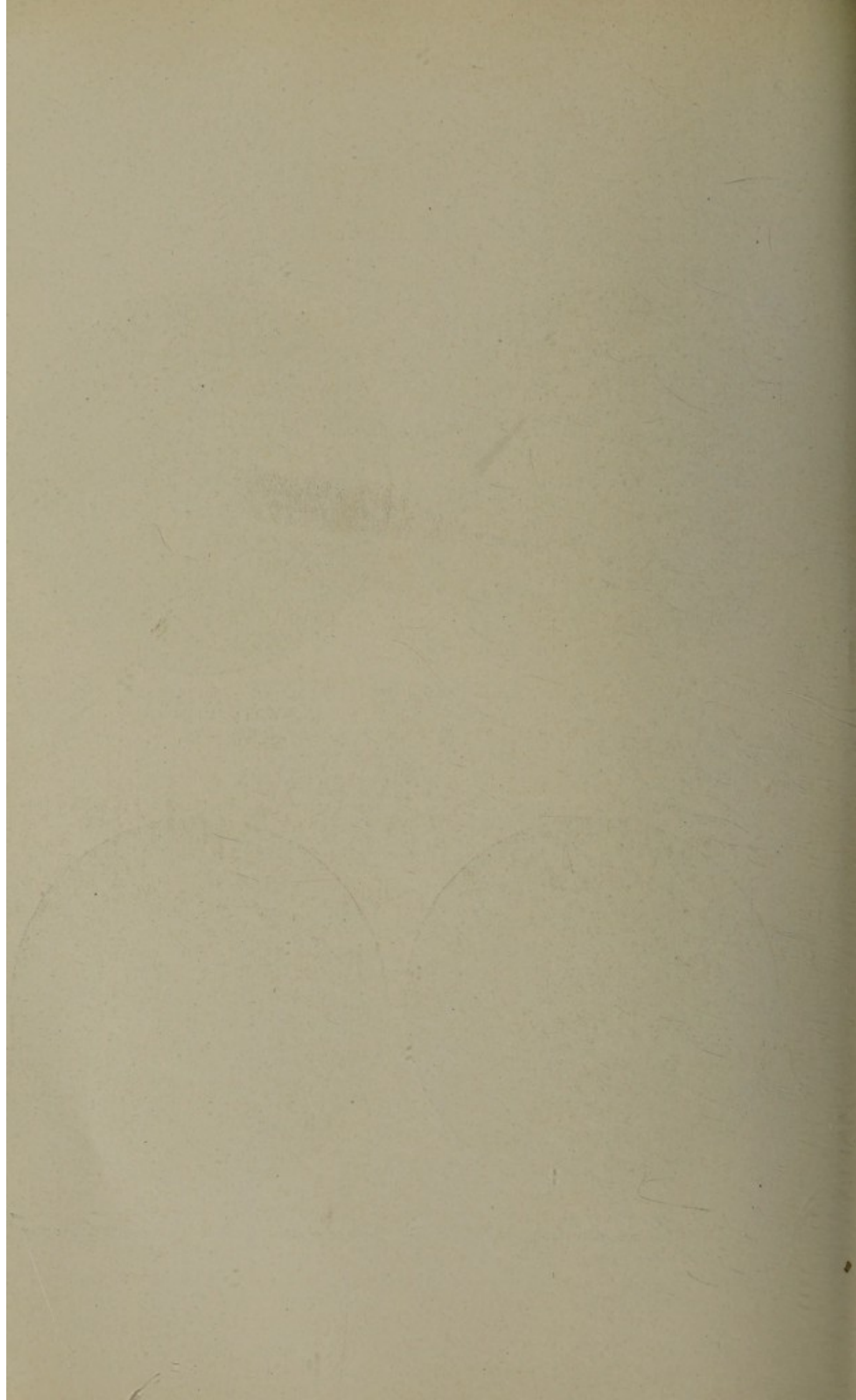


FIG. 136.—Oat,  $\times 110$ .  
Surface section. Proteid layer with fragments of  
epidermis and hairs.







CEREALS.

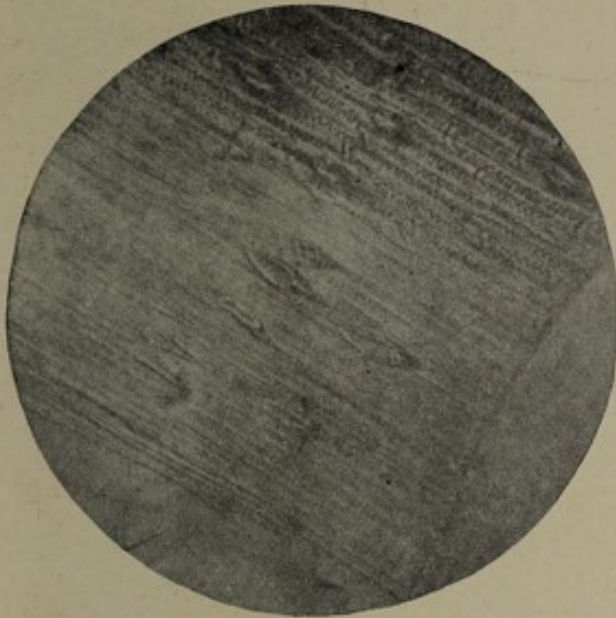


FIG. 137.—Oat,  $\times 110$ .  
Surface view of upper chaff layer.

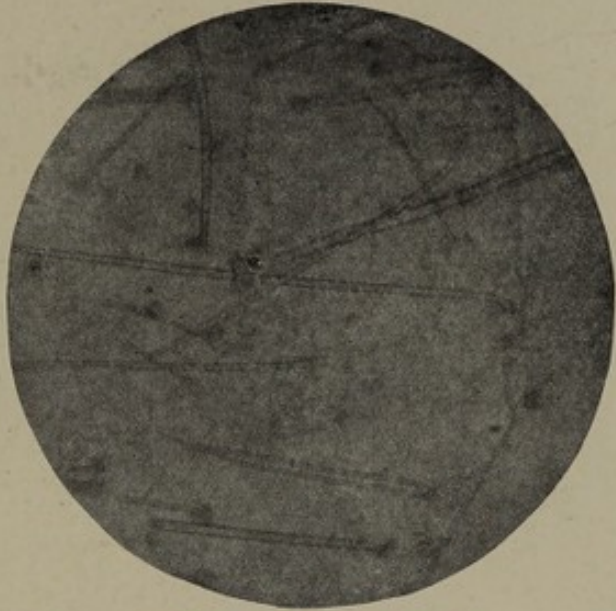


FIG. 138.—Oat,  $\times 55$ .  
Surface view of epidermis and hairs.

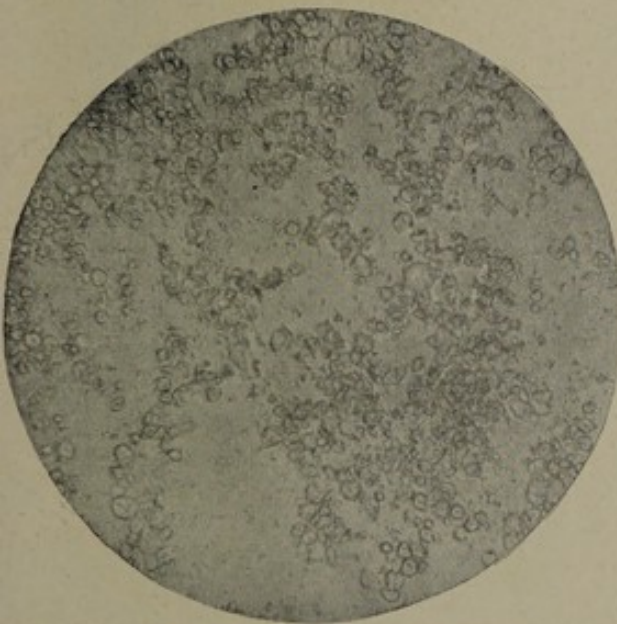


FIG. 139.—Oat Starch,  $\times 220$ .

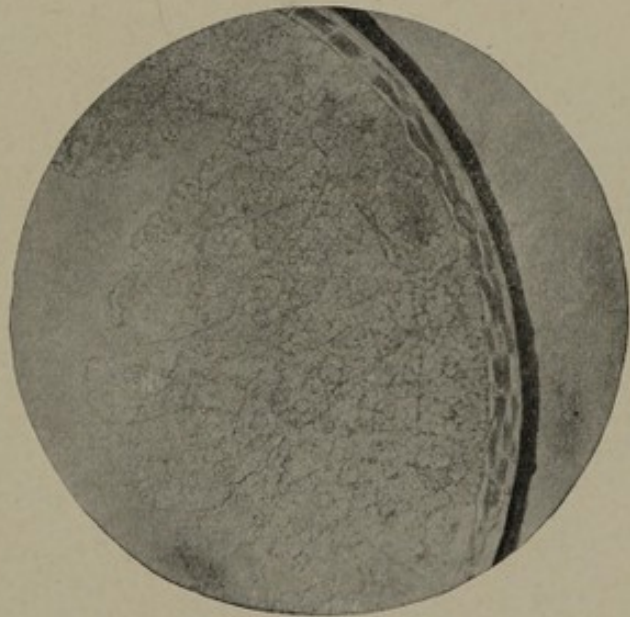
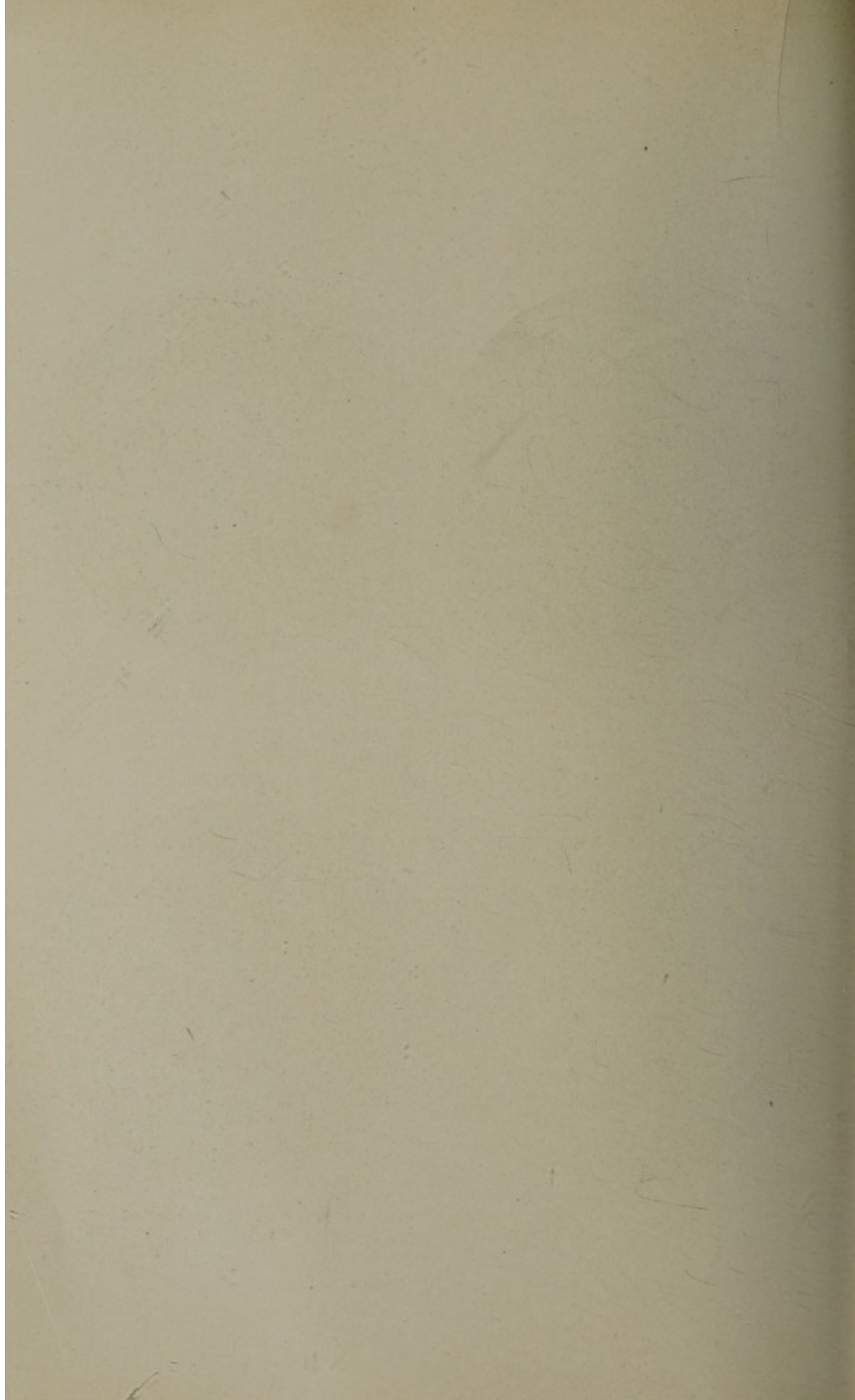


FIG. 140.—Rice,  $\times 110$   
Transverse section through seed coat and part of  
endosperm.







CEREALS.

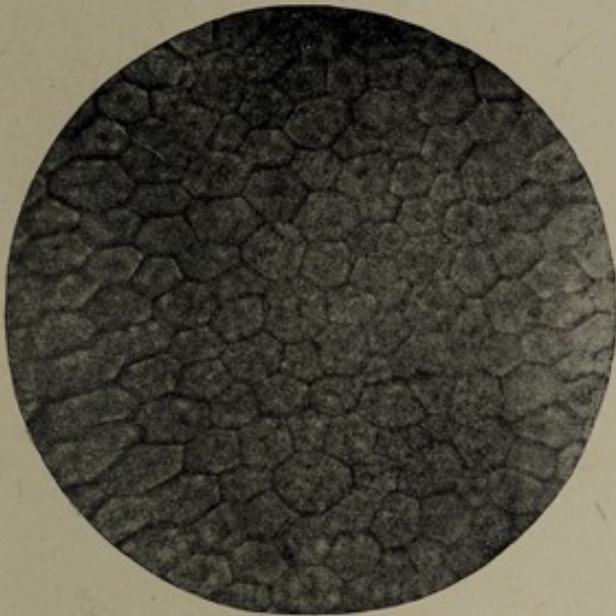


FIG. 141.—Rice,  $\times 110$ .  
Surface section through starch cells.

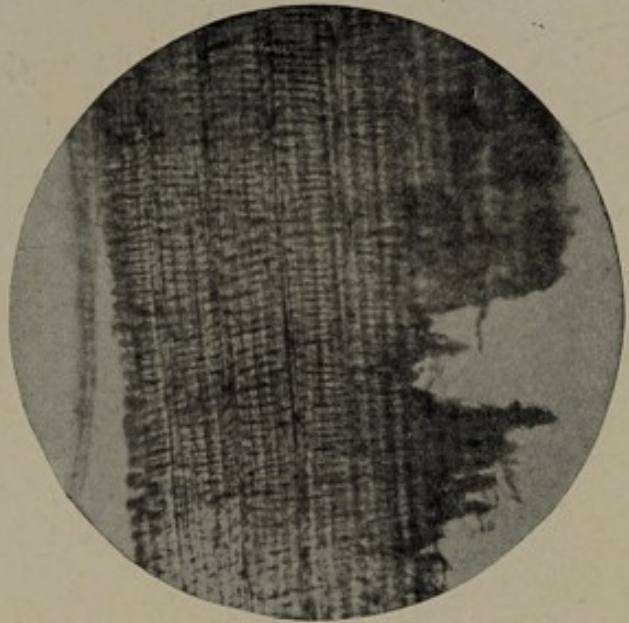


FIG. 142.—Rice,  $\times 110$ .  
Surface view of upper chaff layer.

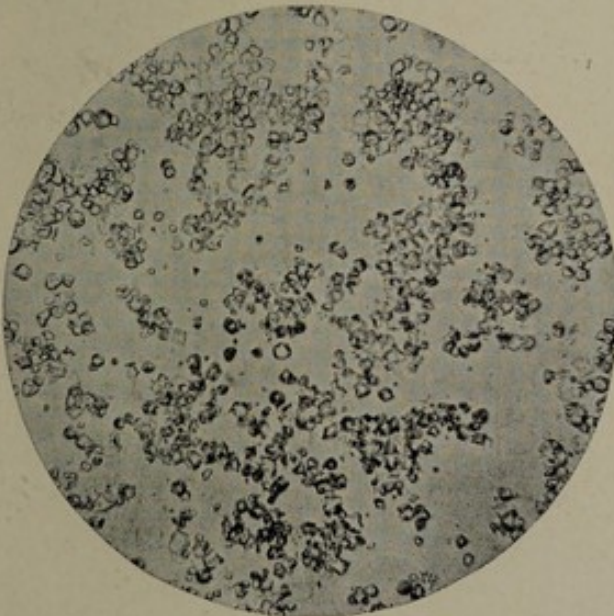
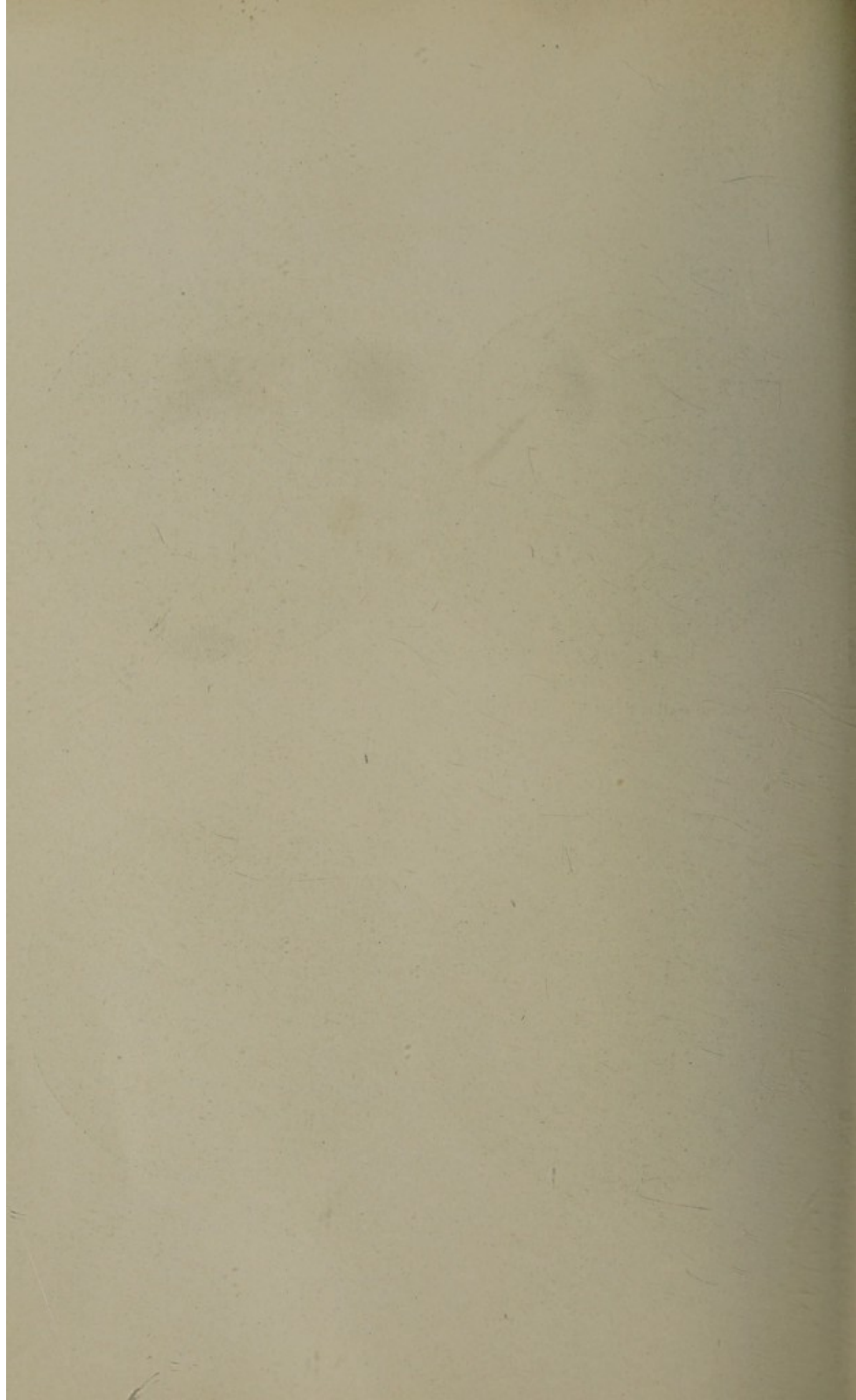


FIG. 143.—Rice Starch,  $\times 220$ .



FIG. 144.—Rye,  $\times 18$   
Transverse section through the entire grain.







CEREALS.



FIG. 145.—Rye,  $\times 110$ .  
Transverse section through pericarp, seed coat, aleurone layer, and starch cells of endosperm.

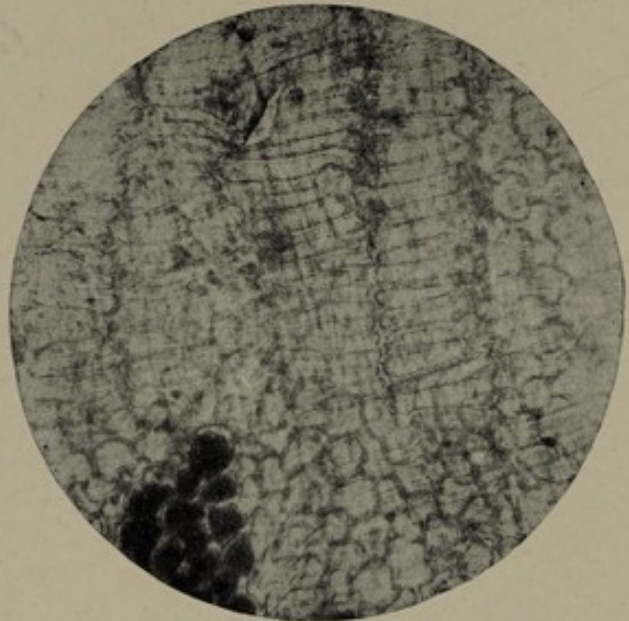


FIG. 146.—Rye,  $\times 110$ .  
Surface view of epidermis and underlying layers.

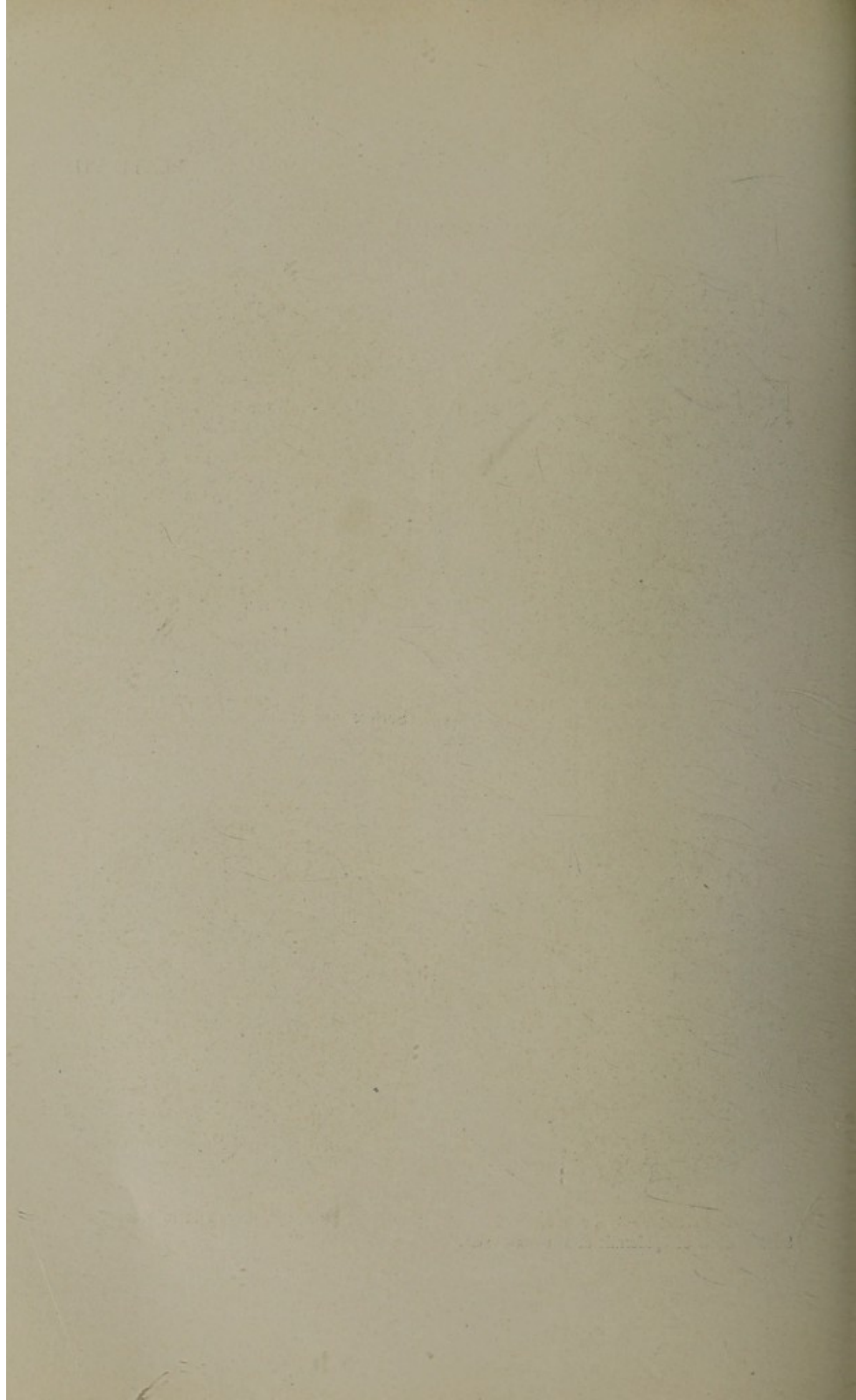


FIG. 147.—Rye,  $\times 110$ .  
Surface view of epidermis and of seed coat.



FIG. 148.—Rye Starch,  $\times 220$ .







CEREALS.

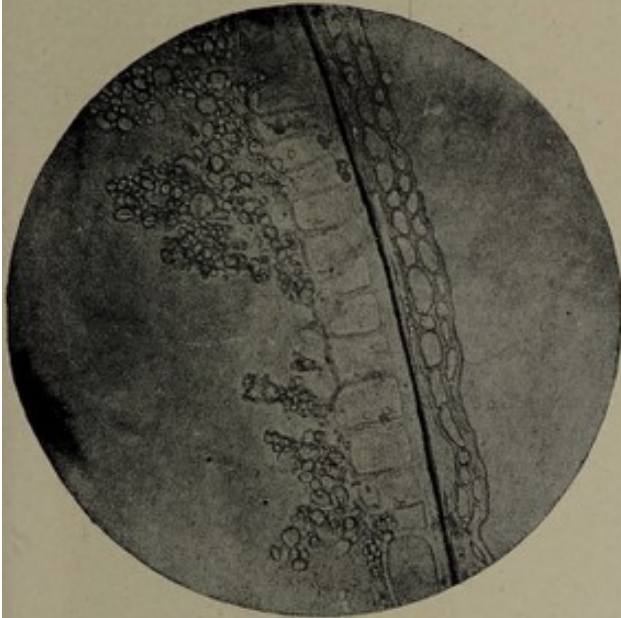


FIG. 149.—Wheat,  $\times 110$ .  
Transverse section through pericarp, seed coat,  
proteid layer, and starch cells of endosperm.



FIG. 150.—Wheat,  $\times 110$ .  
Surface view of outer and inner epidermis    Also  
showing proteid layer.

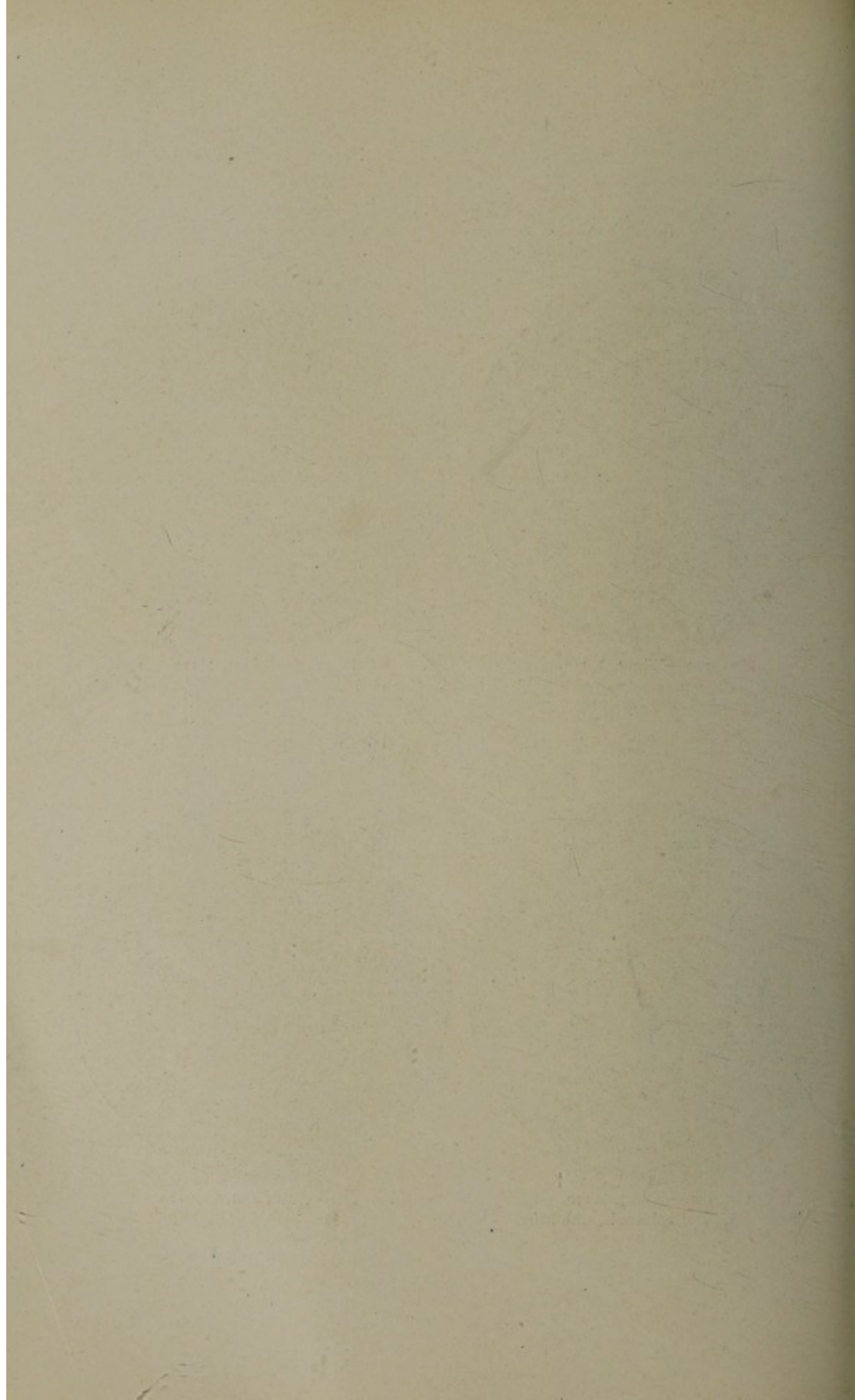


FIG. 151.—Wheat,  $\times 110$ .  
Surface view of epidermis, with hairs.



FIG. 152.—Wheat Starch,  $\times 220$ .







LEGUMES.

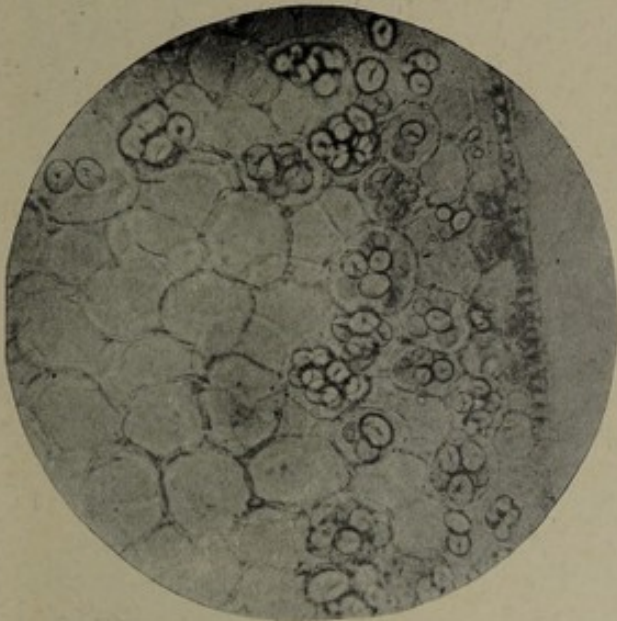


FIG. 153.—Bean,  $\times 110$ .  
Transverse section through starch cells.

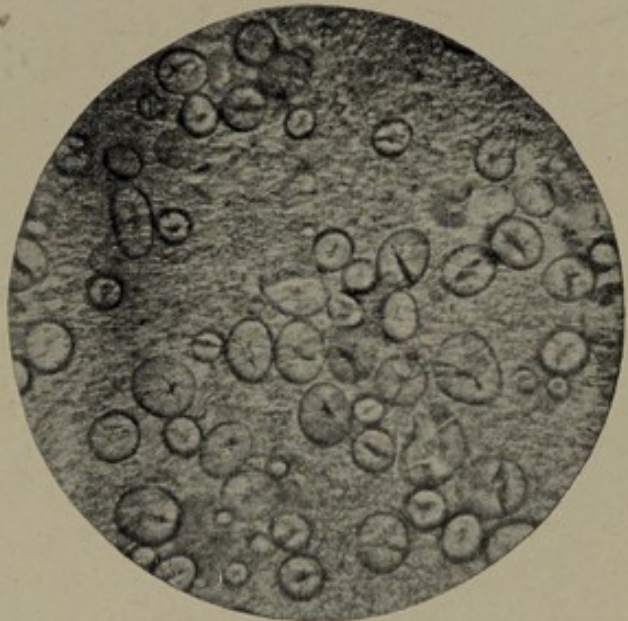


FIG. 154.—Bean Starch,  $\times 220$ .

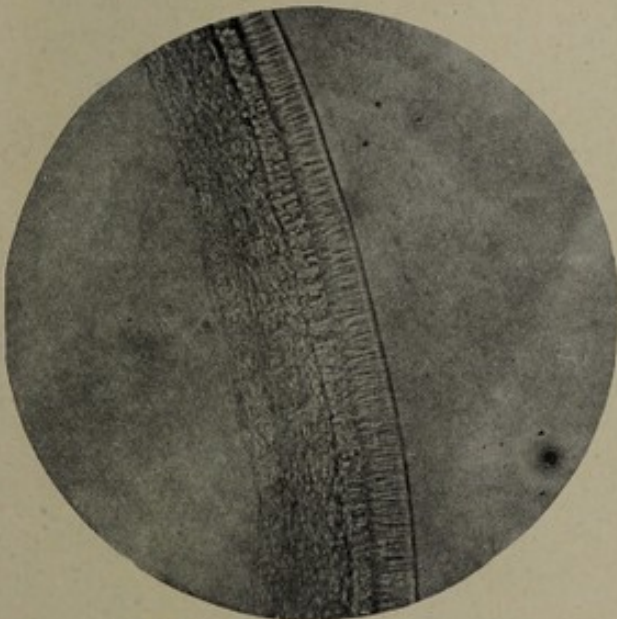
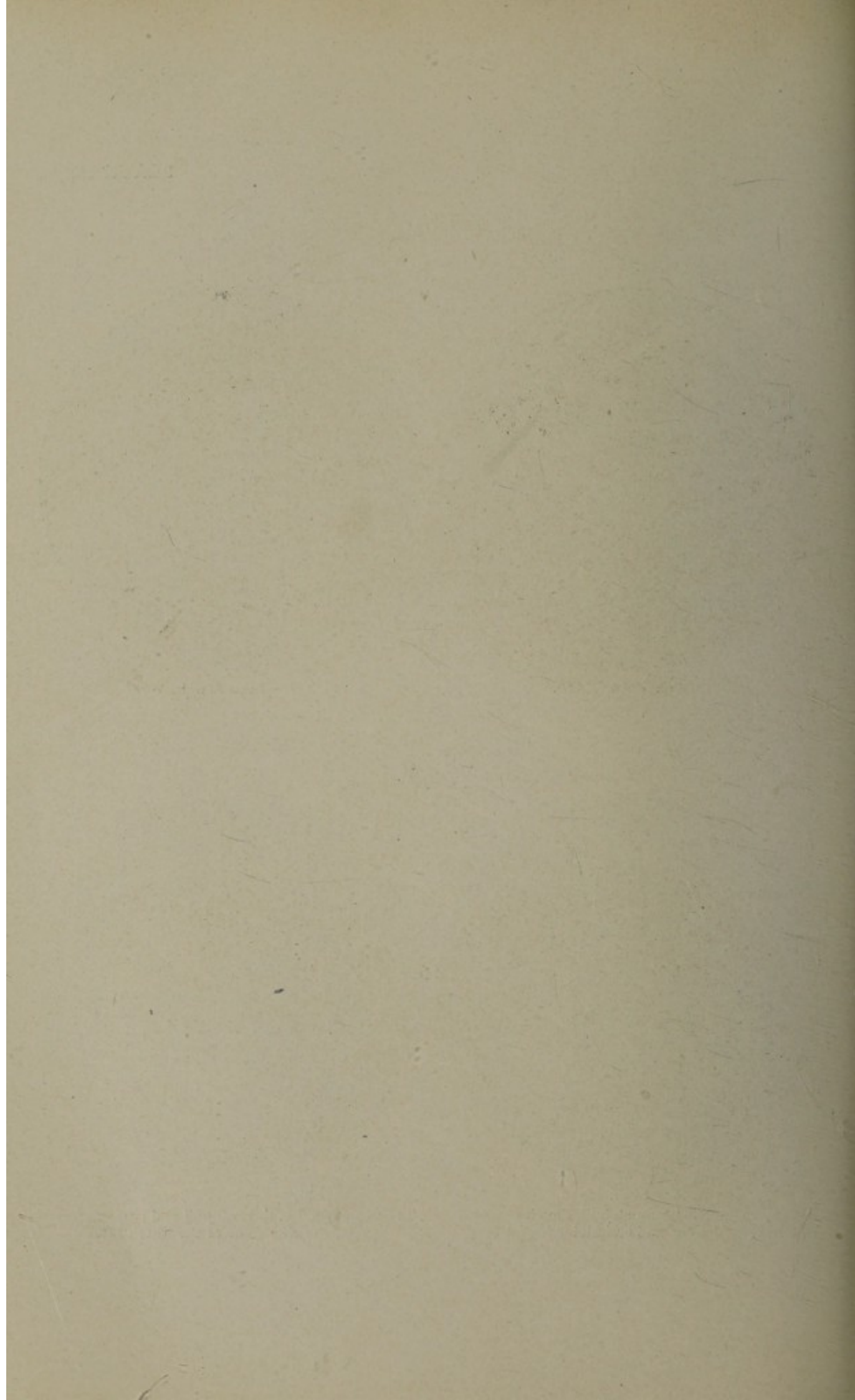


FIG. 155.—Bean,  $\times 110$ .  
Transverse section through hull, showing palisade cells of epidermis, and underlying hypoderma.



FIG. 156.—Lentil,  $\times 110$ .  
Transverse section through hull and part of endosperm, showing some of the starch cells.







LEGUMES.

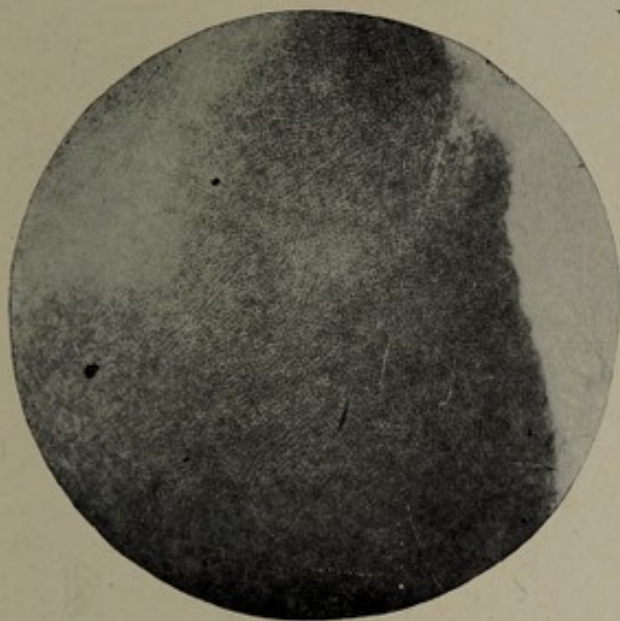


FIG. 157.—Lentil,  $\times 110$ .  
Surface view of epidermis.

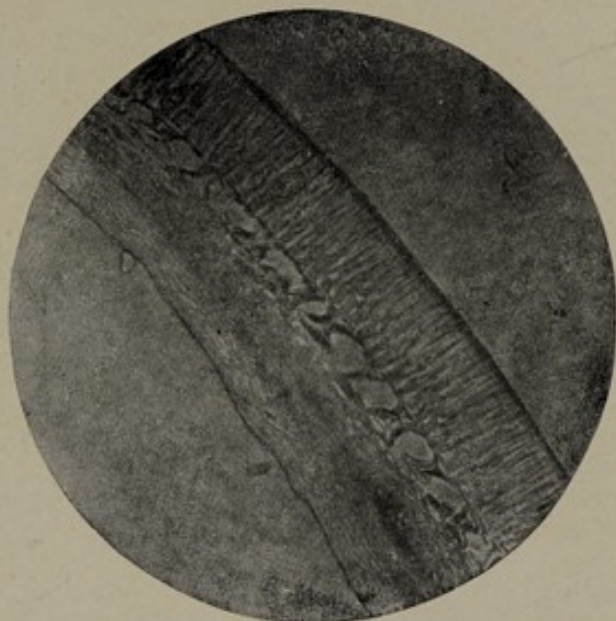


FIG. 158.—Pea,  $\times 110$ .  
Transverse section through hull and seed coat,  
showing outer palisade cells and underlying  
hypodermis.

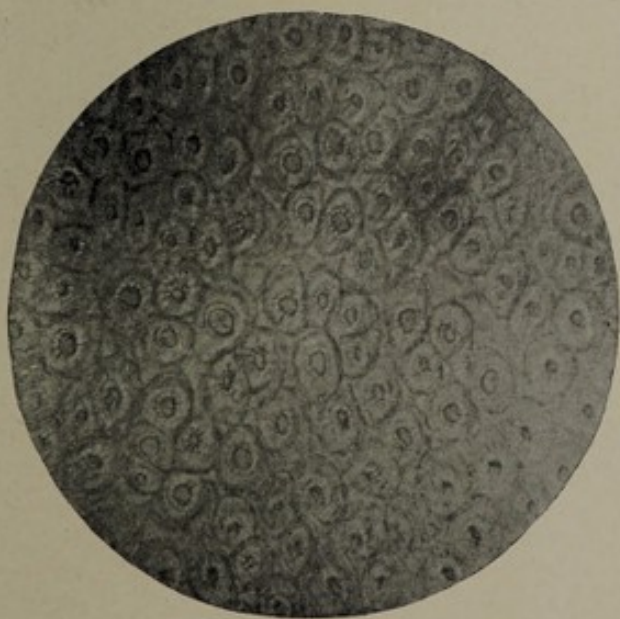
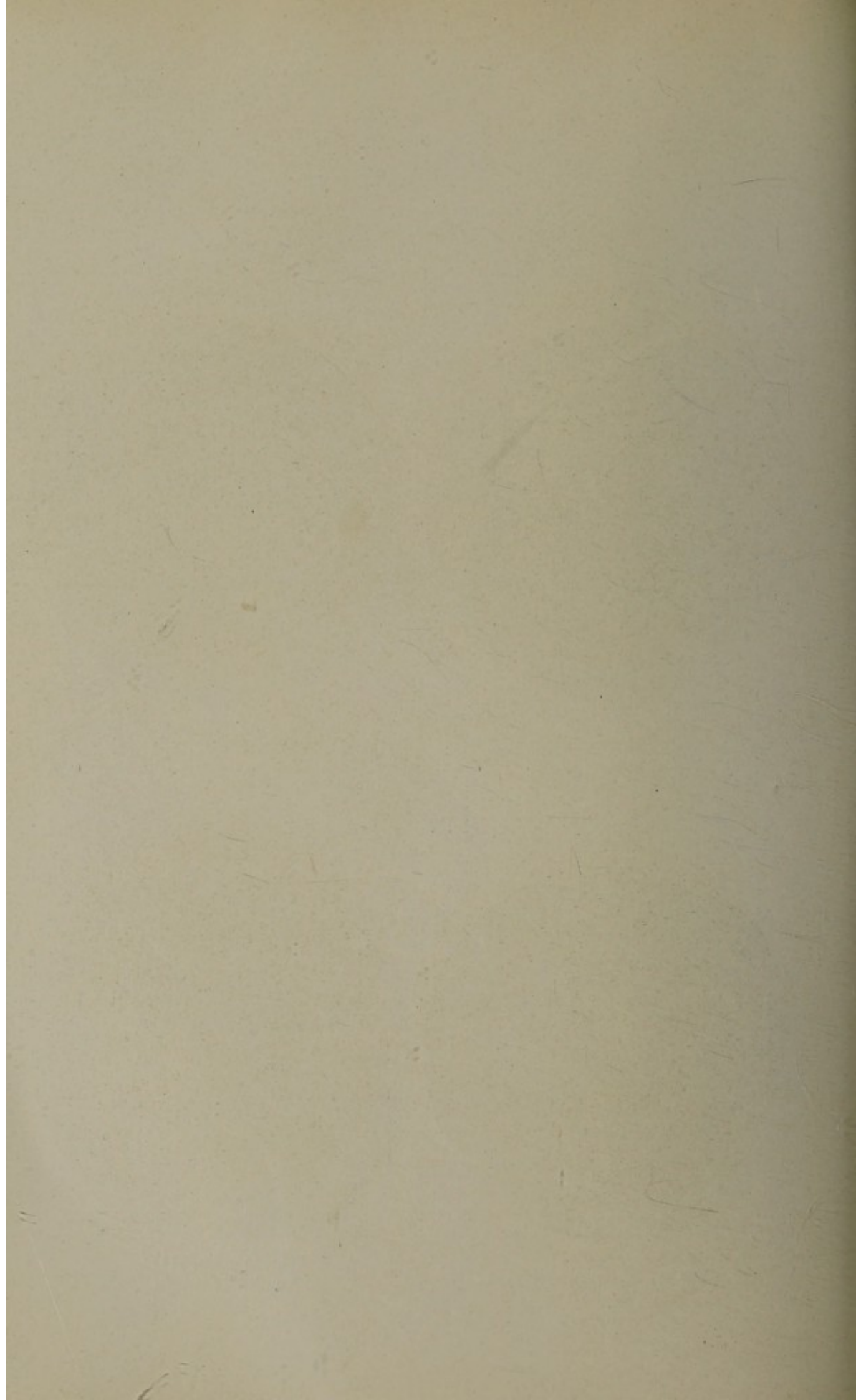


FIG. 159.—Pea,  $\times 110$ .  
Surface section through base of palisade layer.



FIG. 160.—Pea,  $\times 110$ .  
Powdered pea hulls.







LEGUMES.

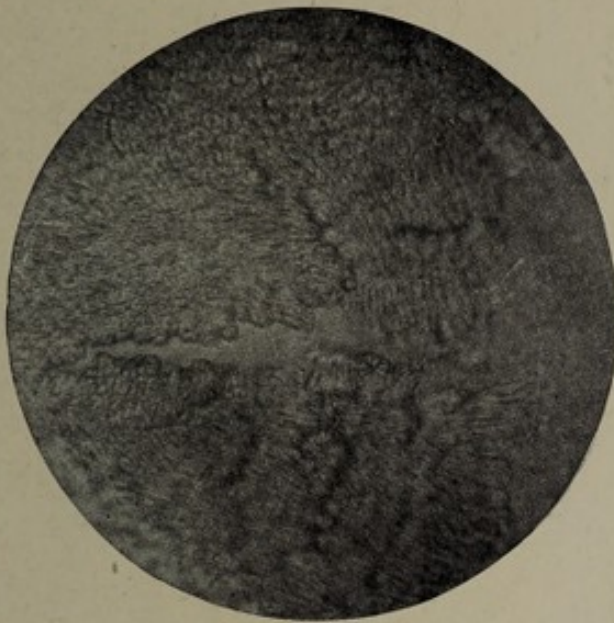


FIG. 161.—Pea,  $\times 110$ .  
Surface view of palisade cells.

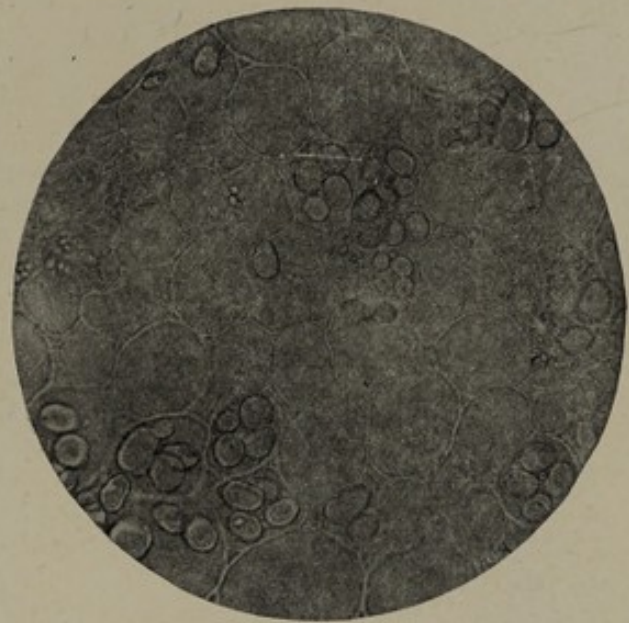


FIG. 162.—Pea,  $\times 110$ .  
Transverse section through starch cells.

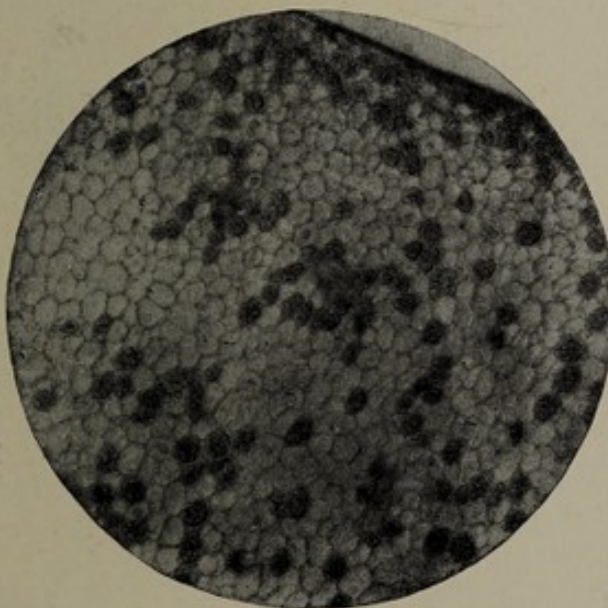


FIG. 163.—Pea,  $\times 30$ .  
Transverse section through starch cells.

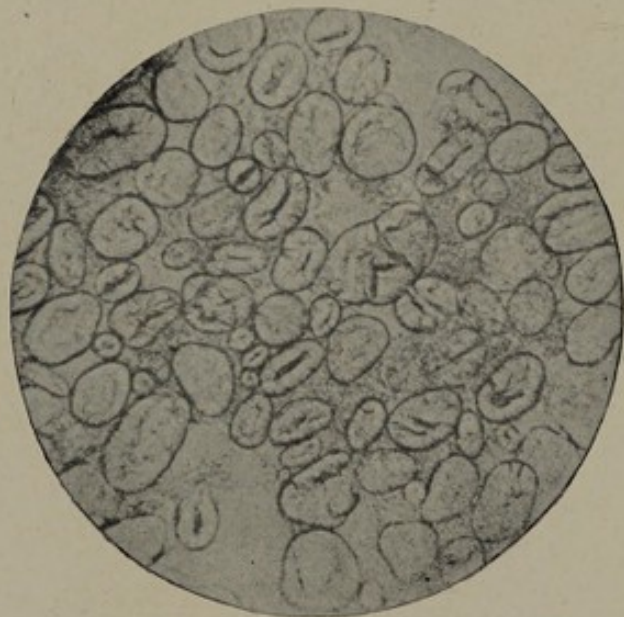
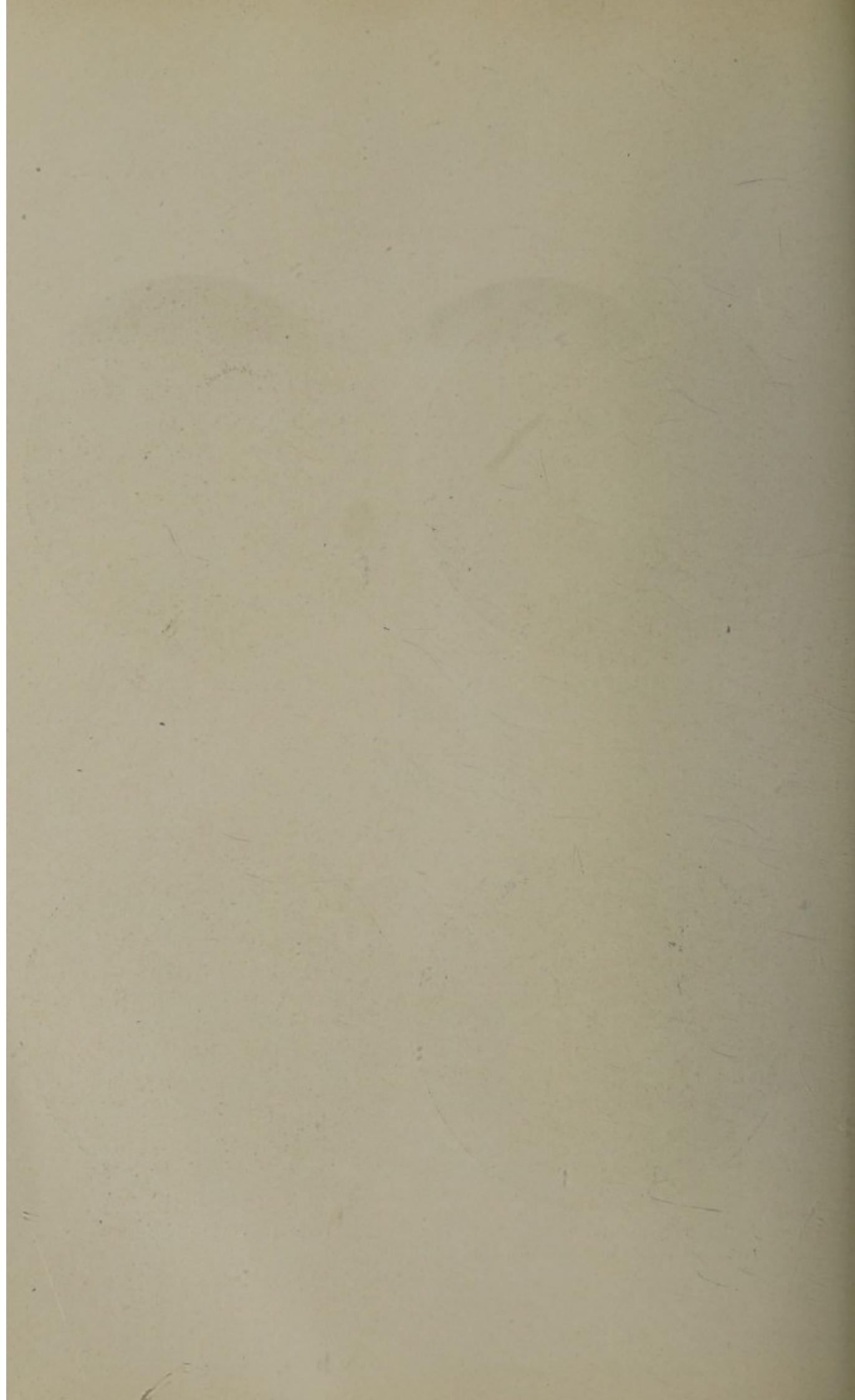


FIG. 164.—Pea Starch,  $\times 220$ .







MISCELLANEOUS STARCHES.

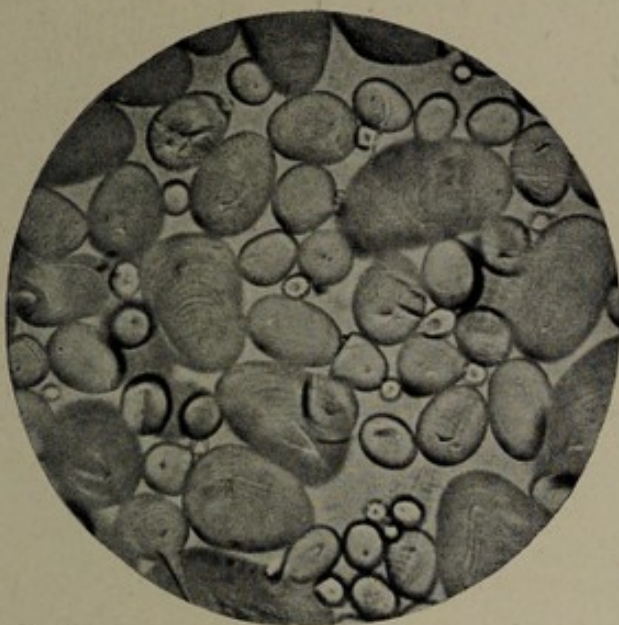


FIG. 165.—Potato Starch,  $\times 220$ .

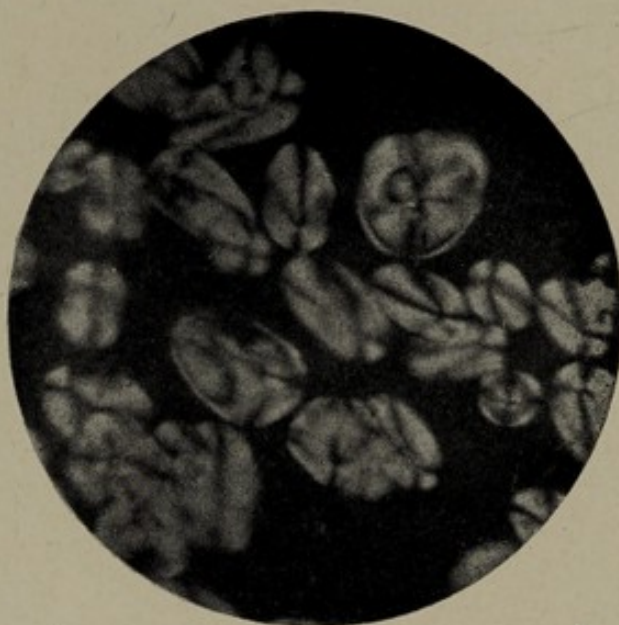


FIG. 166.—Potato Starch,  $\times 220$ .  
With polarized light.

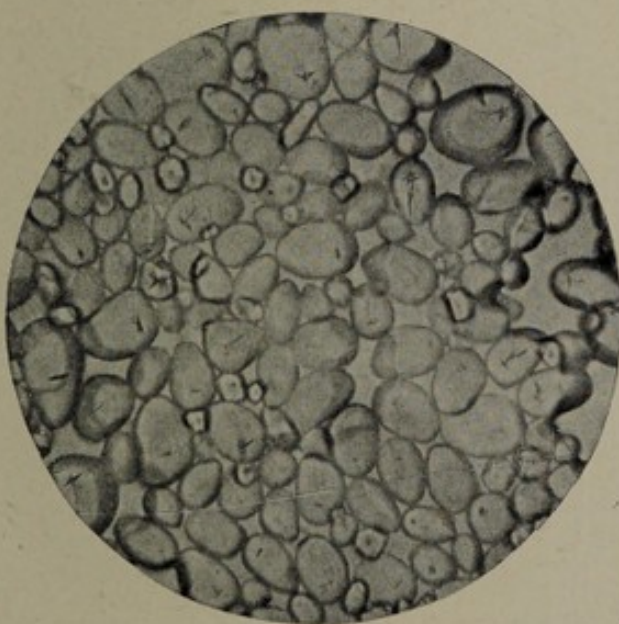


FIG. 167.—Arrowroot Starch,  $\times 220$ .

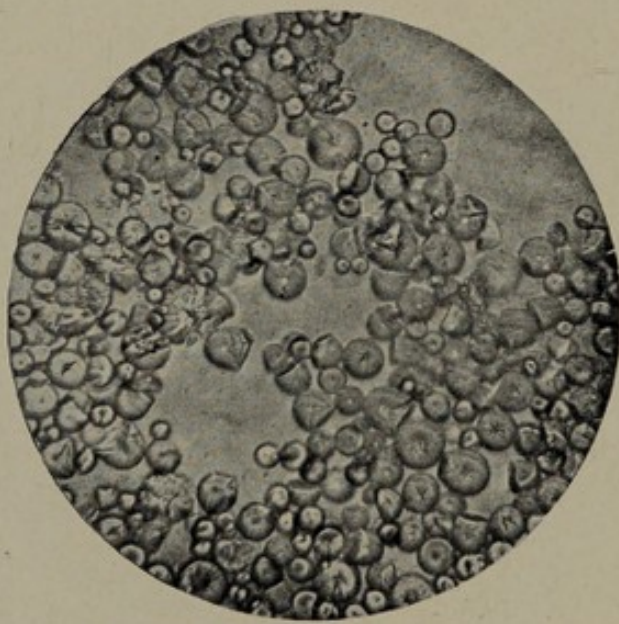
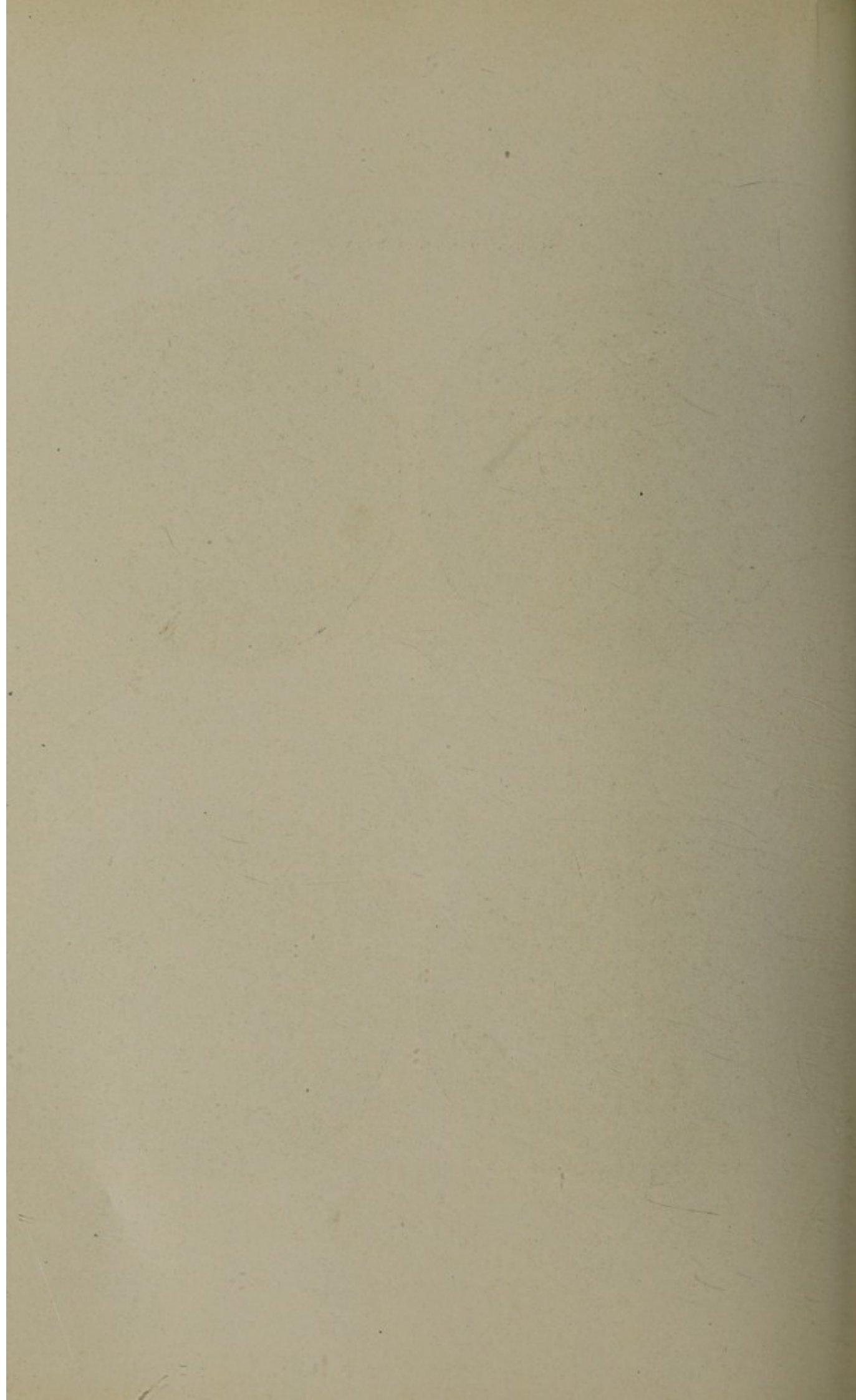


FIG. 168.—Tapioca Starch,  $\times 220$ .  
(Cassava.)







TURMERIC. SAGO.

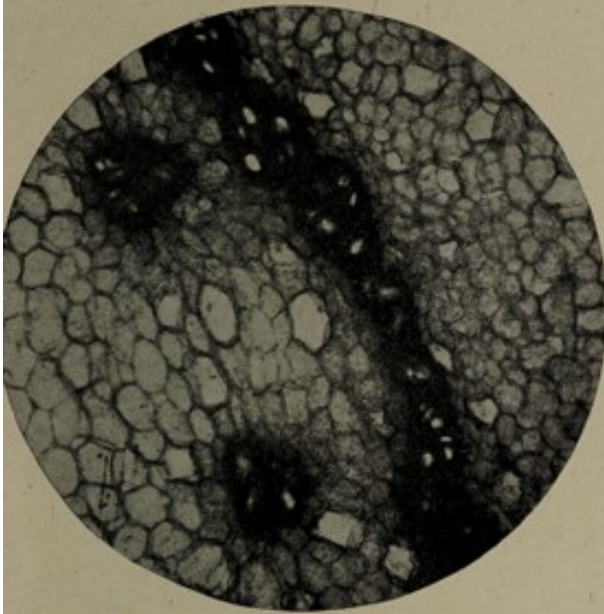


FIG. 169.—Turmeric,  $\times 70$ .  
Transverse section through rhizome.

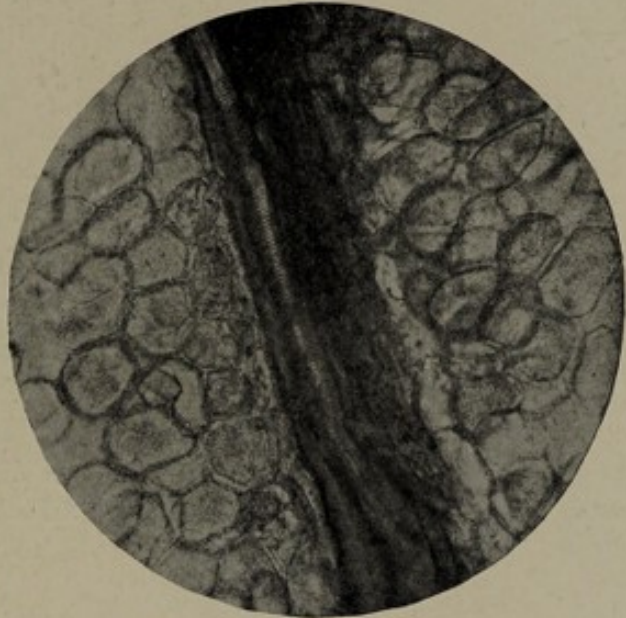


FIG. 170.—Turmeric,  $\times 110$ .  
Longitudinal section. Note spiral ducts through  
the center.

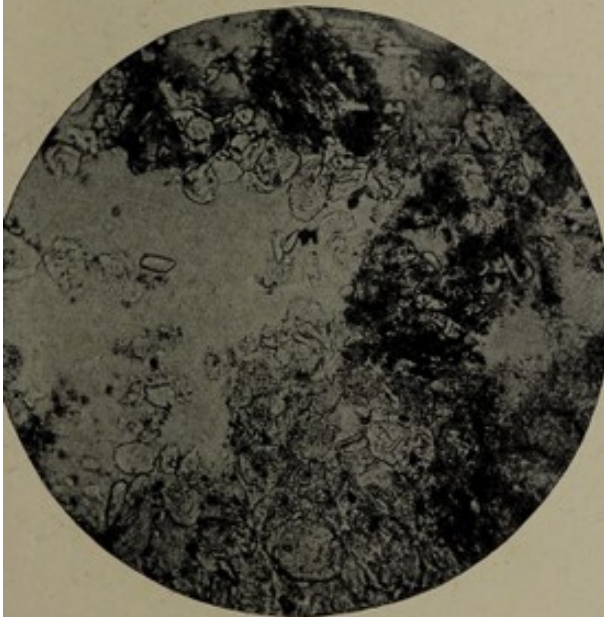


FIG. 171.—Powdered Turmeric,  $\times 110$ .  
Showing starch grains, fragments of cell tissue,  
coloring matter, etc.

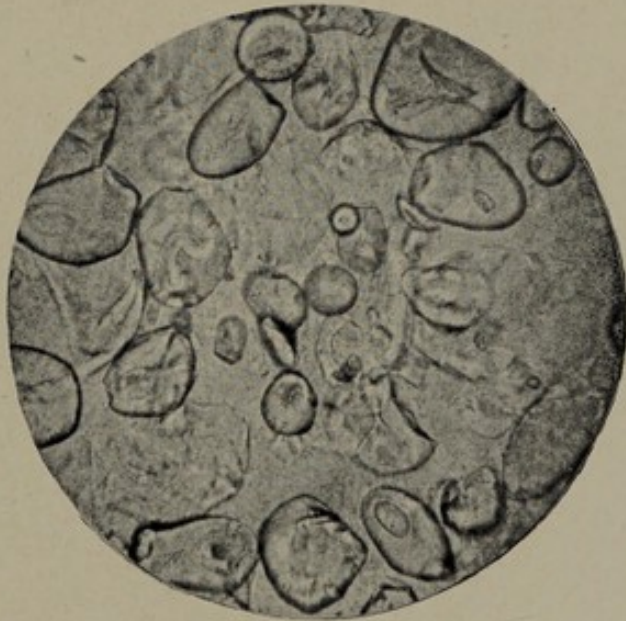
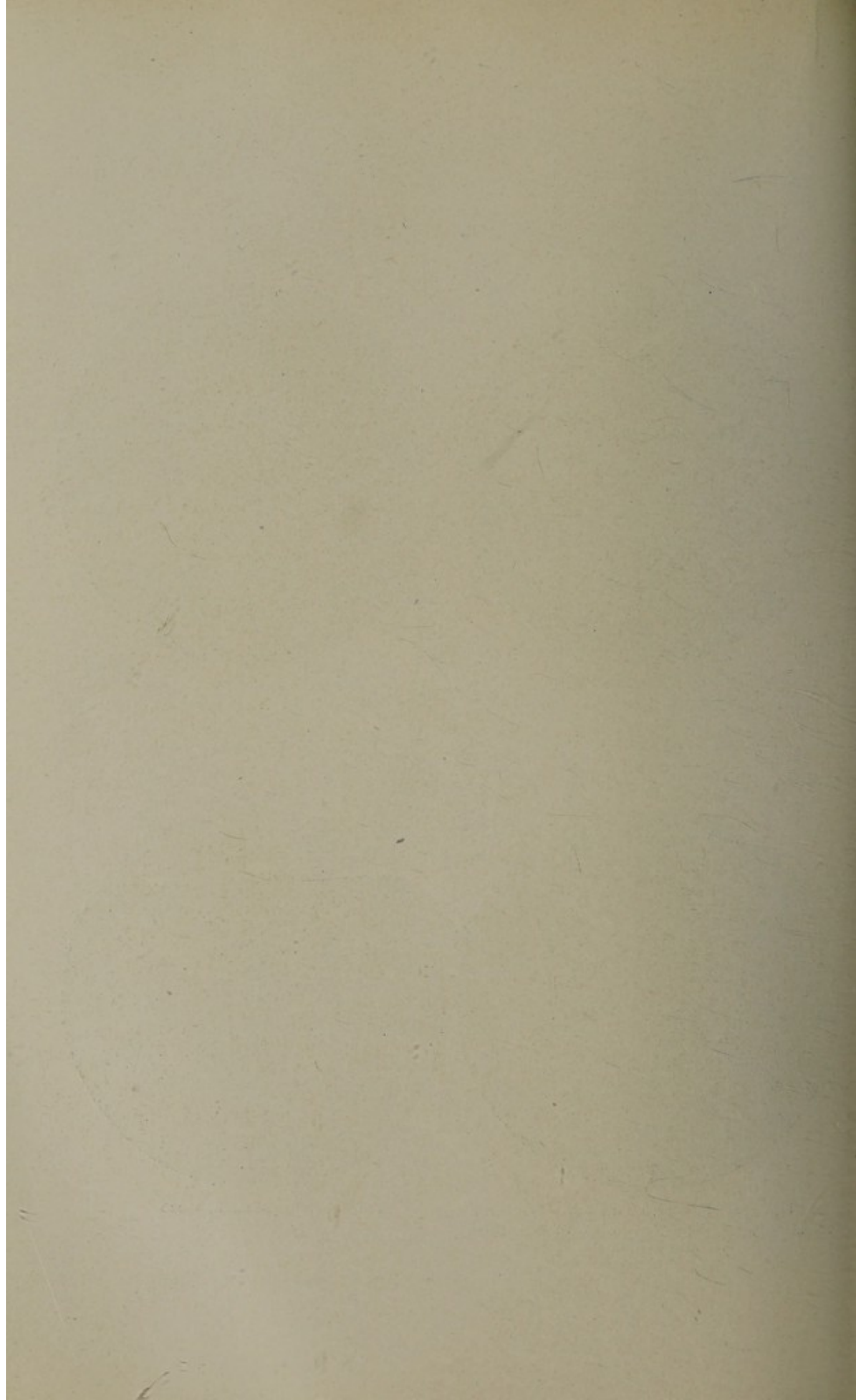


FIG. 172.—Sago Starch,  $\times 220$ .







COFFEE.

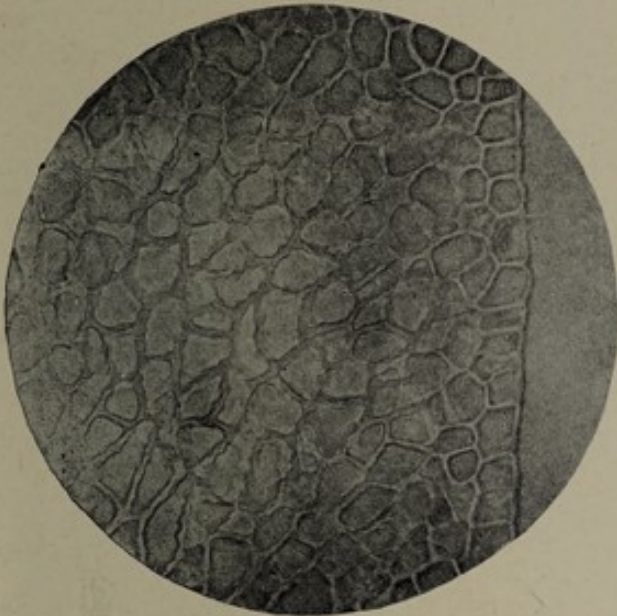


FIG. 173.—Raw Coffee,  $\times 110$ .  
Transverse section of outer portion of endosperm.



FIG. 174.—Roasted Coffee,  $\times 130$ .  
Transverse section through parenchyma of endosperm.

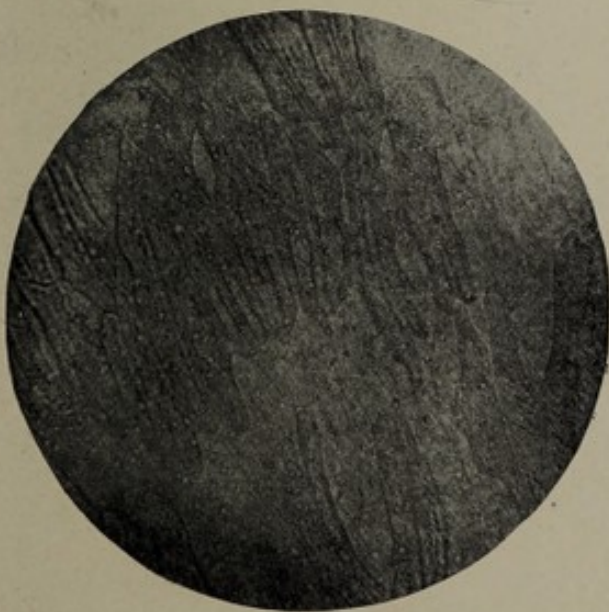
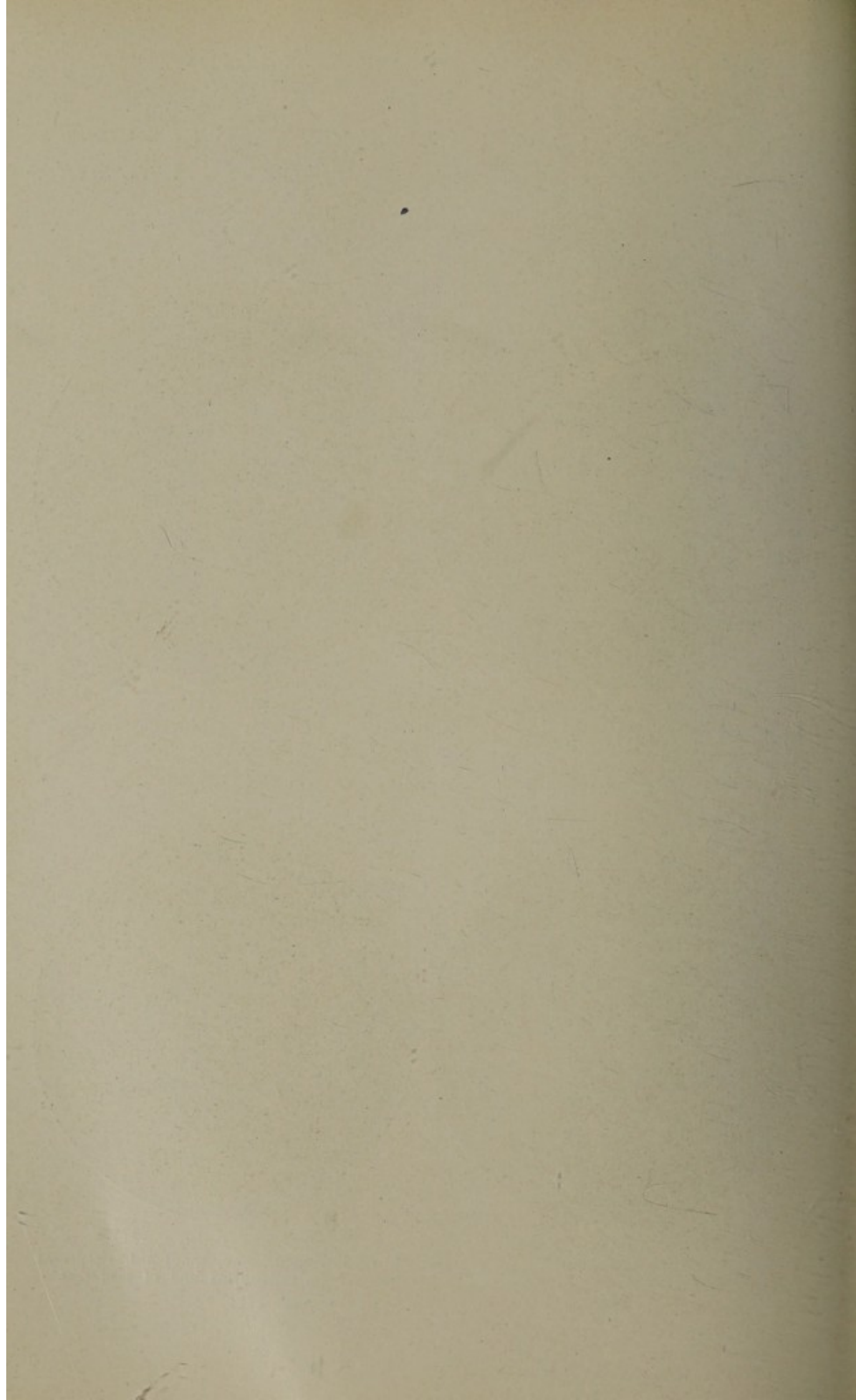


FIG. 175.—Coffee,  $\times 110$ .  
Surface view of seed coat.



FIG. 176.—Coffee,  $\times 110$ .  
Roasted, ground coffee, showing fragments of endosperm parenchyma and of seed coat.







COFFEE. CHICORY.



FIG. 177.—Adulterated Coffee,  $\times 130$ .  
Dark masses of roasted pea starch are shown,  
with transparent fragments of the palisade  
cells of the pea-hull.

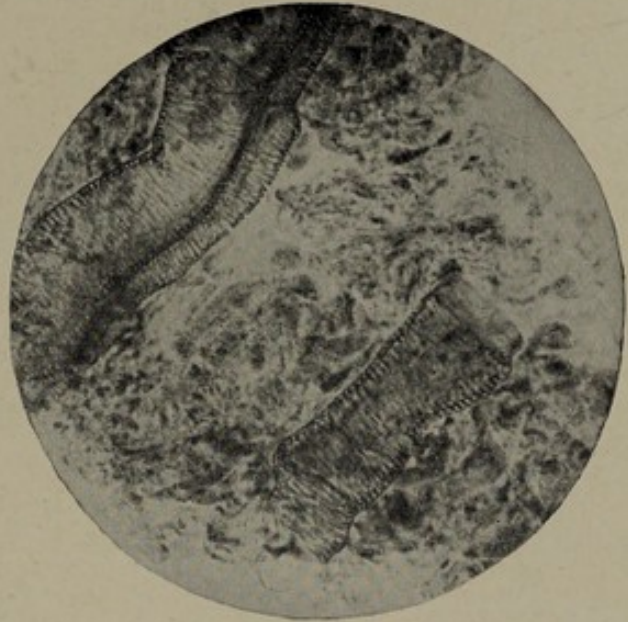


FIG. 178.—Adulterated Coffee,  $\times 130$ .  
The vascular ducts of chicory show most con-  
spicuously in this field.

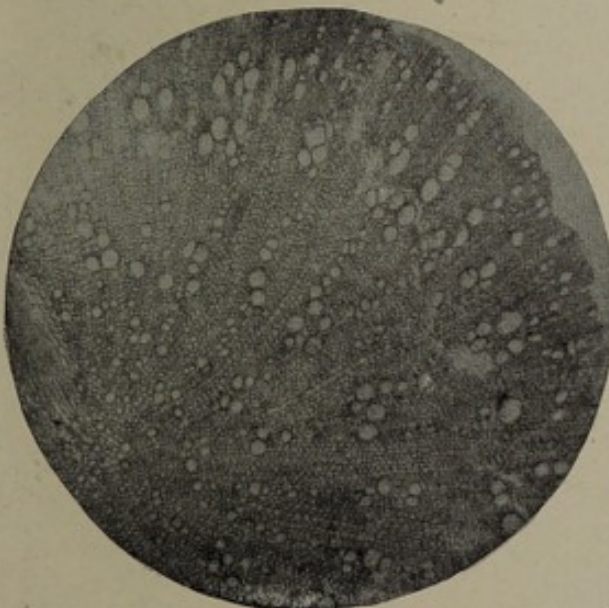


FIG. 179.—Chicory,  $\times 25$ .  
Transverse section through the root.

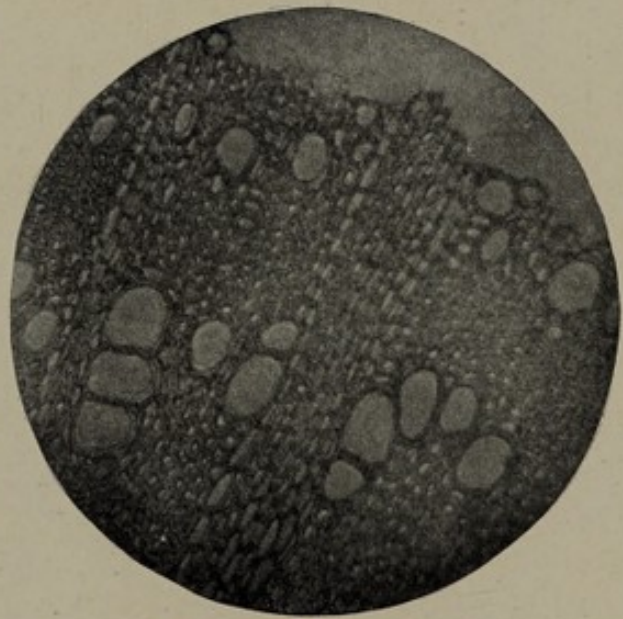
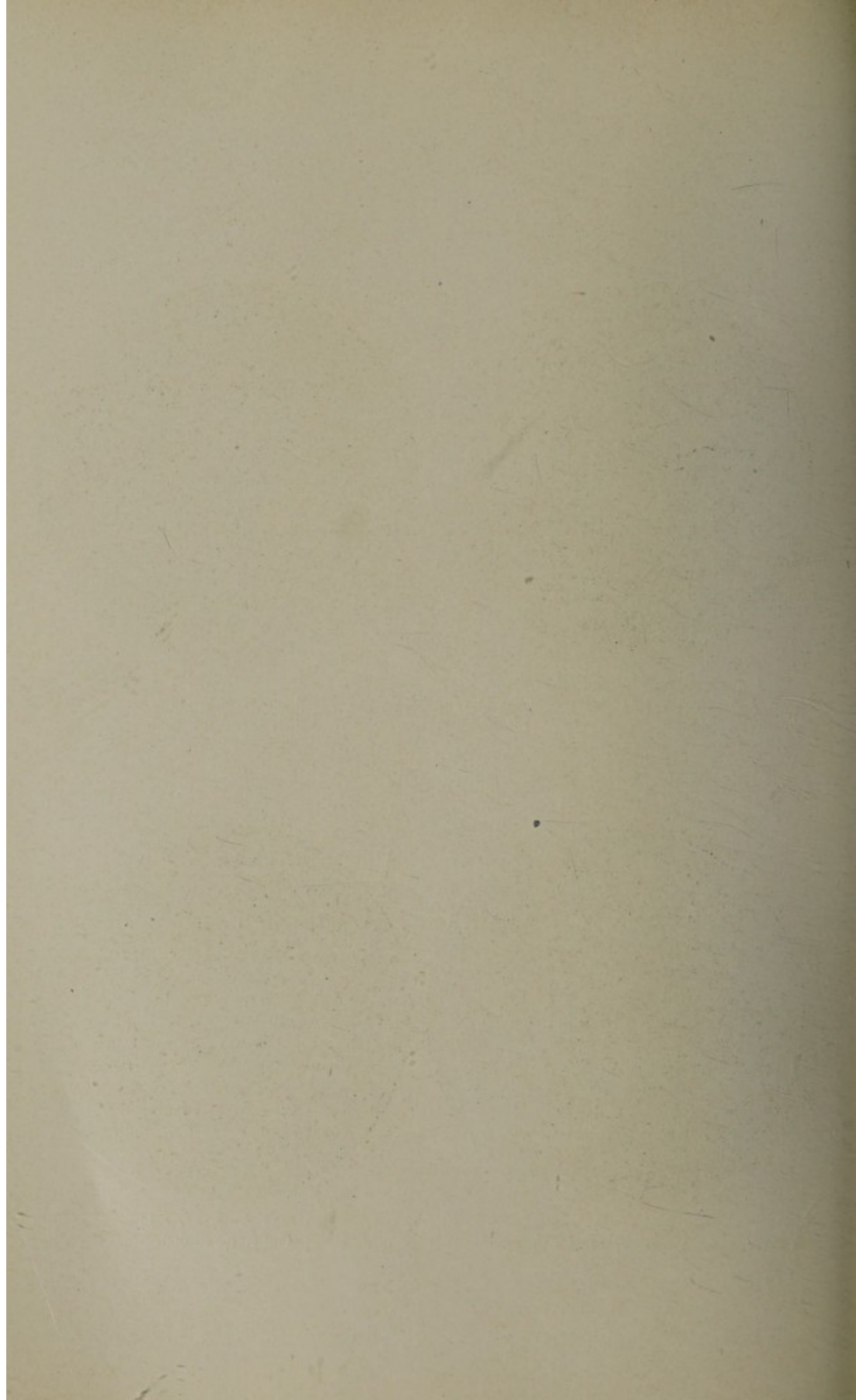


FIG. 180.—Chicory,  $\times 110$ .  
Transverse section.







CHICORY. COCOA.



FIG. 181.—Chicory,  $\times 110$ .  
Tangential section, showing reticulated ducts and  
wood parenchyma.

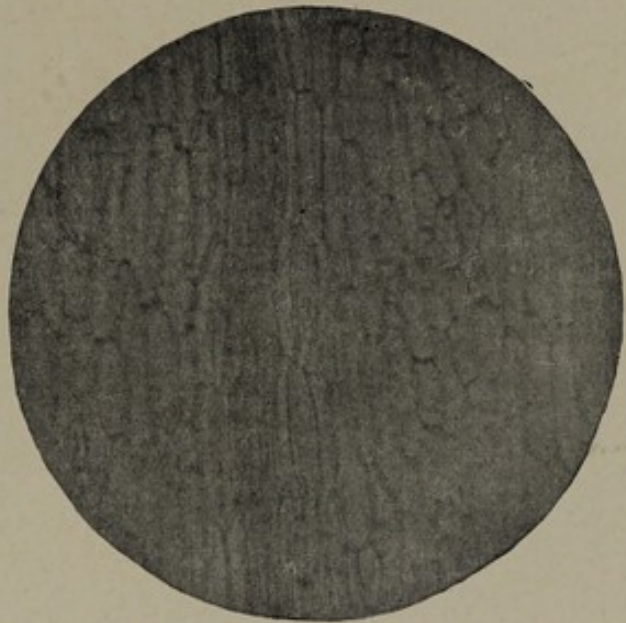


FIG. 182.—Chicory,  $\times 110$   
Radial section, showing bark parenchyma and  
milk ducts.

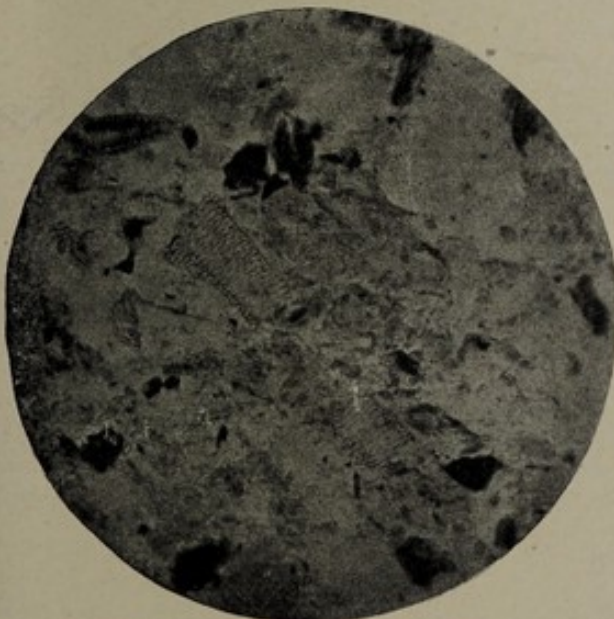


FIG. 183.—Chicory,  $\times 110$ .  
Roasted and ground, showing fragments of  
ducts and other tissues.

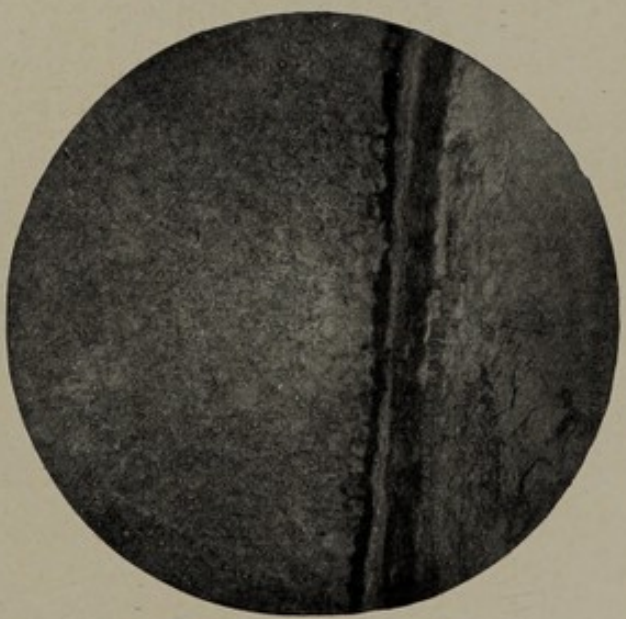
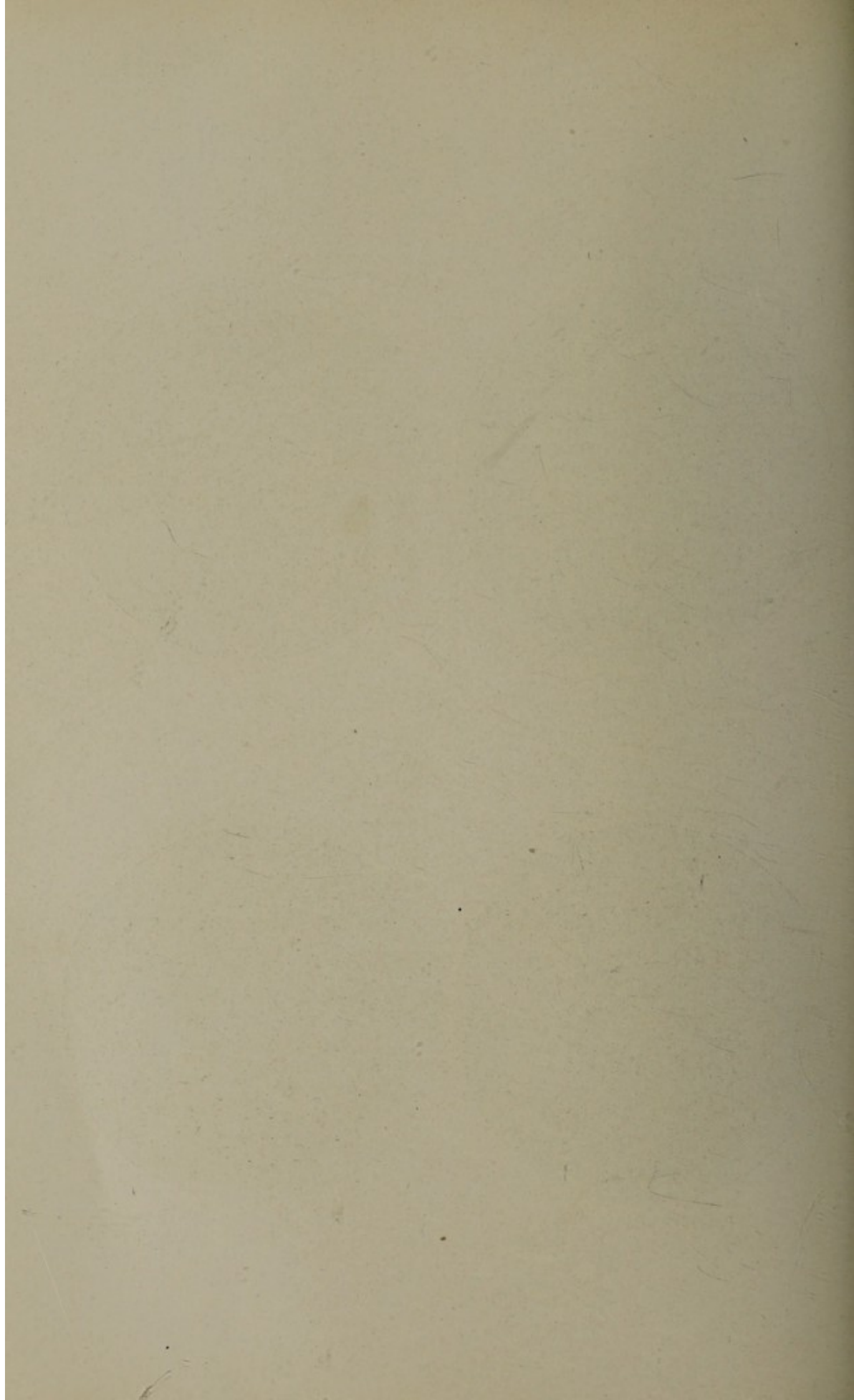


FIG. 184.—Cocoa,  $\times 110$ .  
Transverse section through periphery of seed,  
seed coats, and cotyledon.







COCOA.

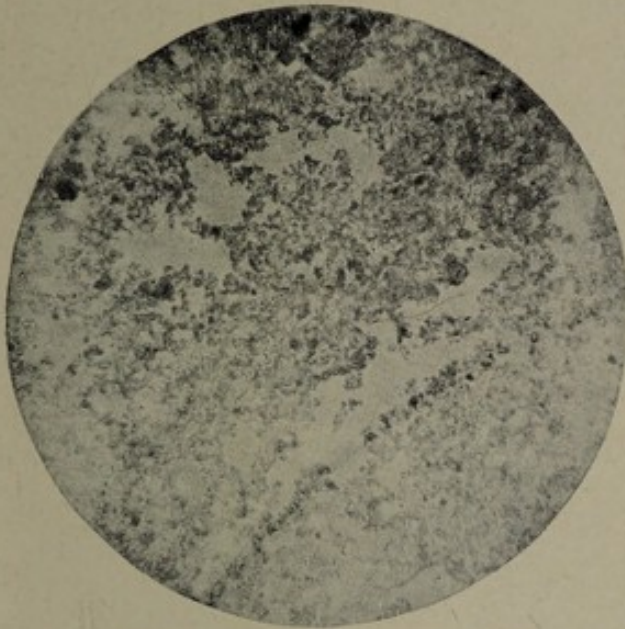


FIG. 185.—Powdered Cocoa,  $\times 110$ .

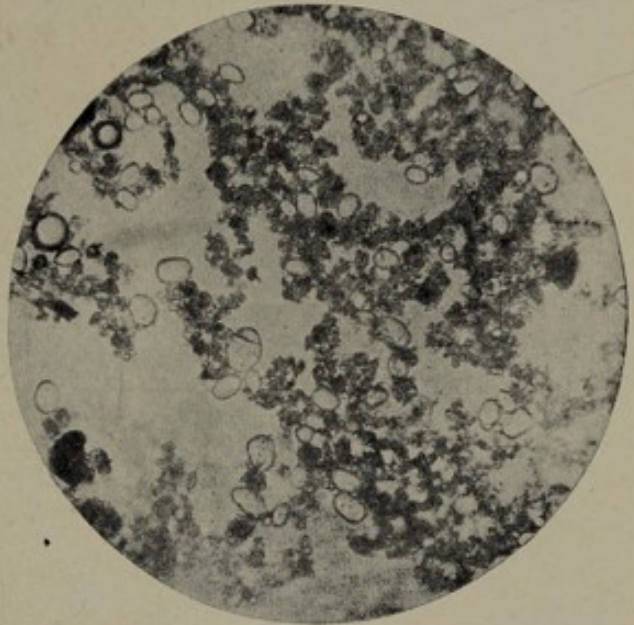


FIG. 186.—Adulterated Cocoa,  $\times 110$ .  
Showing admixture of arrowroot with the cocoa powder.



FIG. 187.—Cocoa Shell,  $\times 110$ .  
Transverse section through epidermis, pulp, and  
mucilaginous layers of the pericarp and seed  
coat.

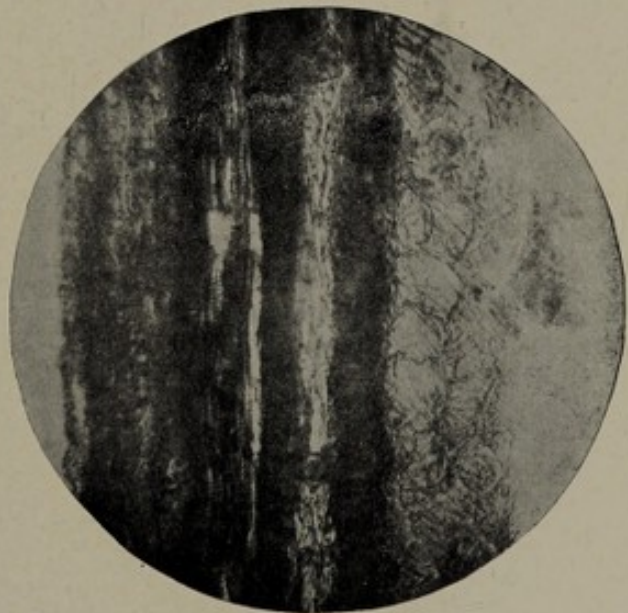


FIG. 188.—Cocoa Shell,  $\times 110$ .  
Longitudinal section through shell.



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TEA. SPICES.

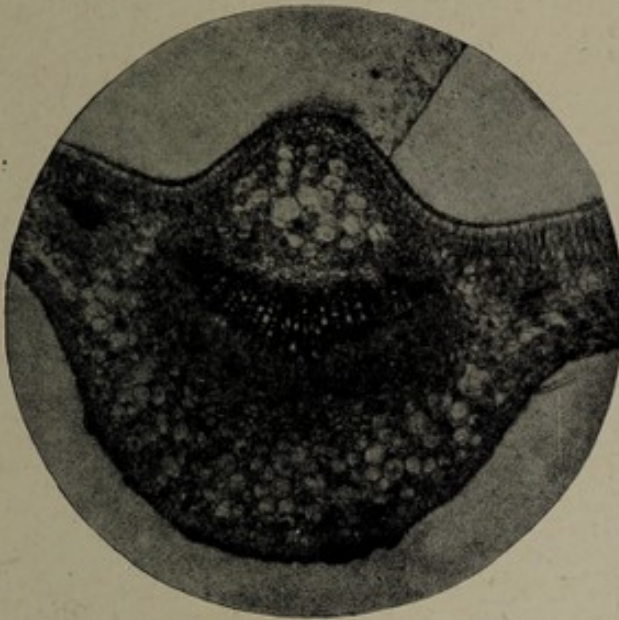


FIG. 189.—Tea,  $\times 55$ .

Transverse section through midrib of leaf. Note the palisade layer below the upper epidermis, the inner wood vessels above the center, and the parenchyma of the pulp.

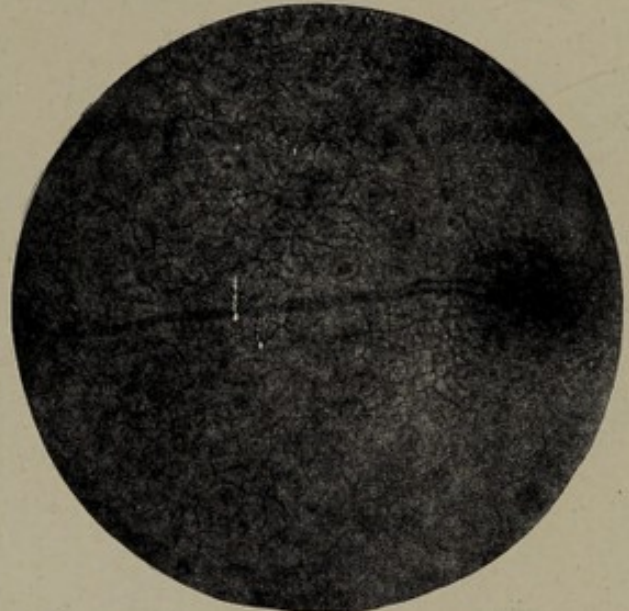


FIG. 190.—Tea,  $\times 110$ .

Surface view of lower epidermis, with stomata and one of the hairs.

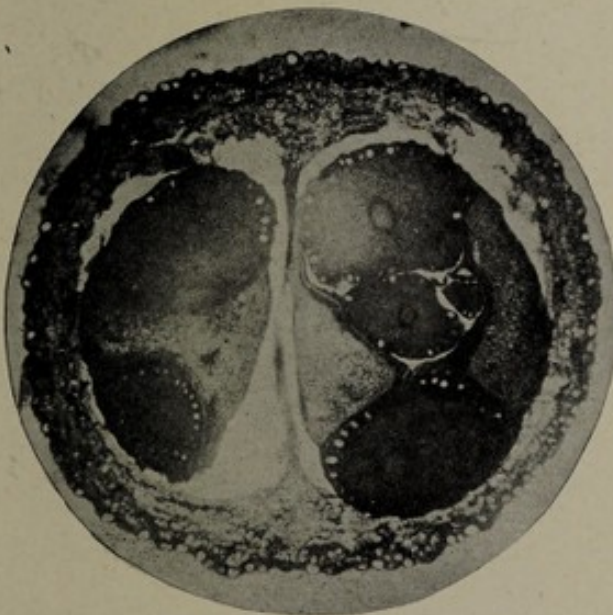


FIG. 191.—Allspice,  $\times 9$ .

Transverse section through the entire berry, showing the two cells, with kidney shaped seed in each.

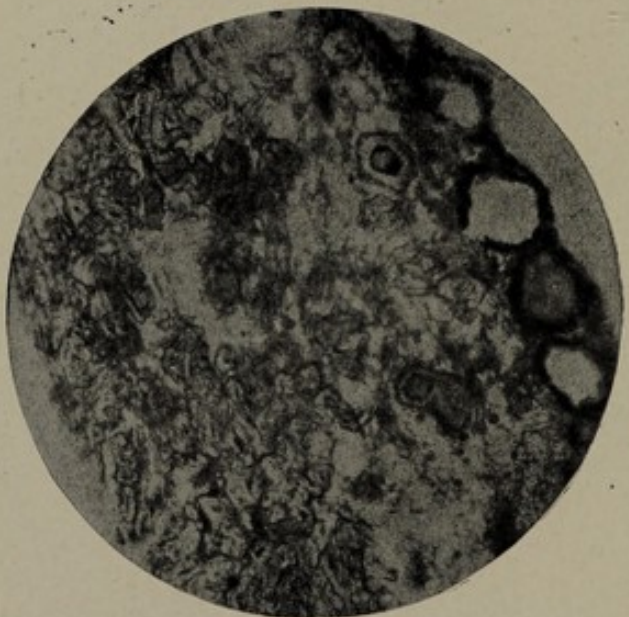
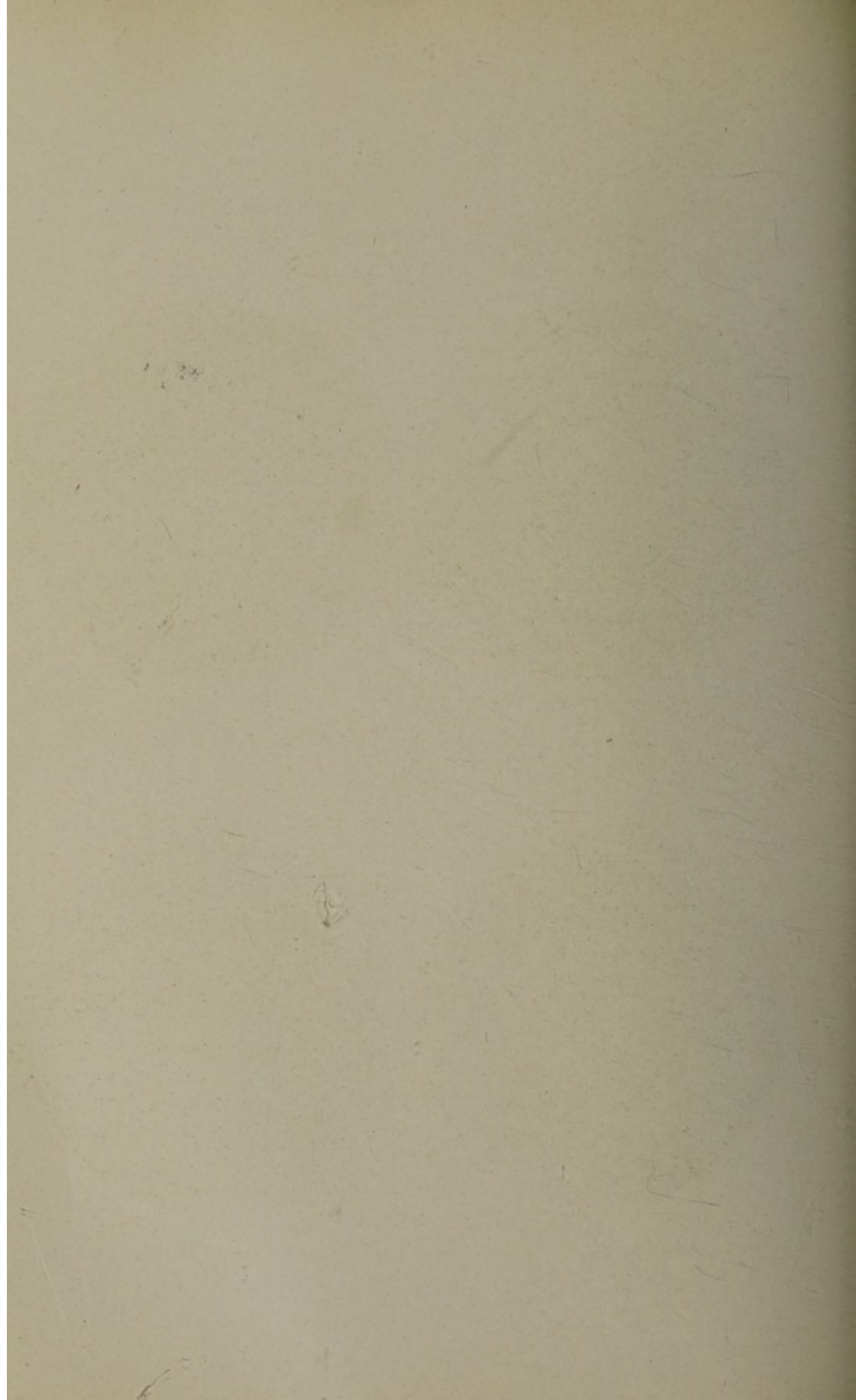


FIG. 192.—Allspice.  $\times 70$ .

Transverse section through pericarp, showing oil spaces and stone cells.







SPICES.

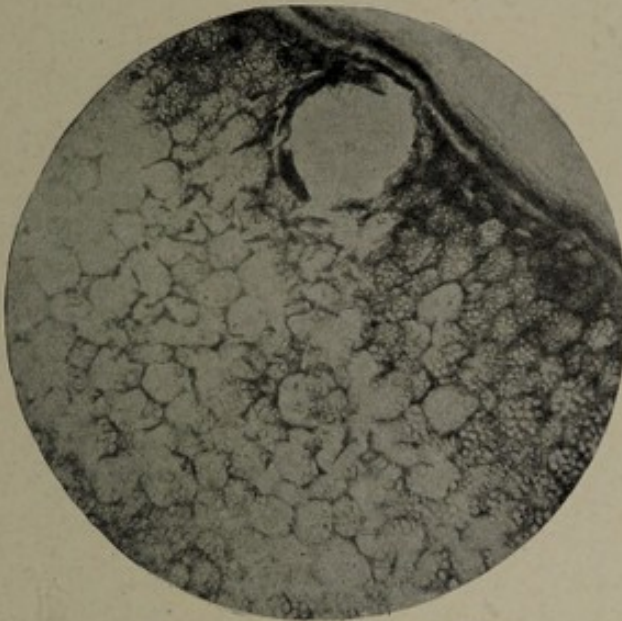


FIG. 193.—Allspice Seed,  $\times 110$ .  
Transverse section through seed shell and part of  
embryo, showing starch cells.

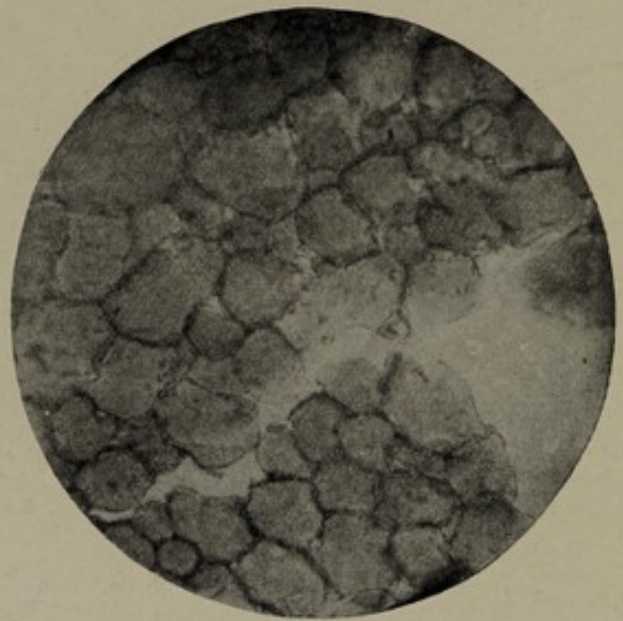


FIG. 194.—Allspice Seed,  $\times 110$ .  
Transverse section through the resinous portion of  
the seed coat, showing port wine colored lumps  
of gum or resin.

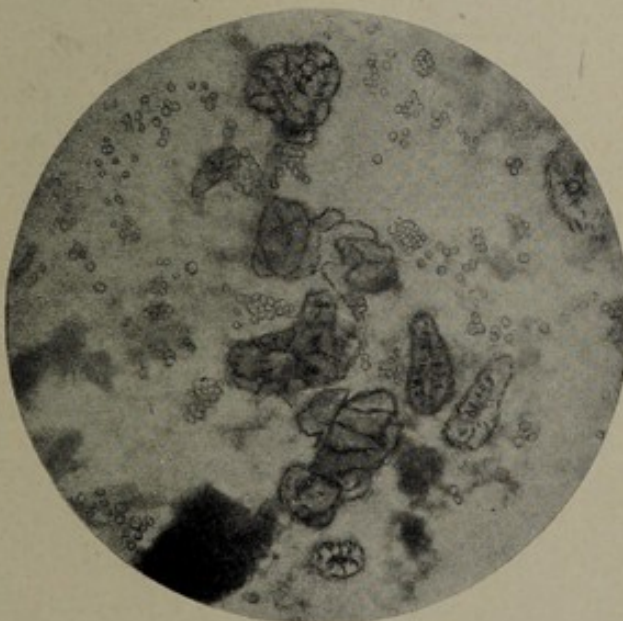


FIG. 195.—Powdered Allspice,  $\times 110$ .  
Showing stone cells, resinous lumps, and starch.

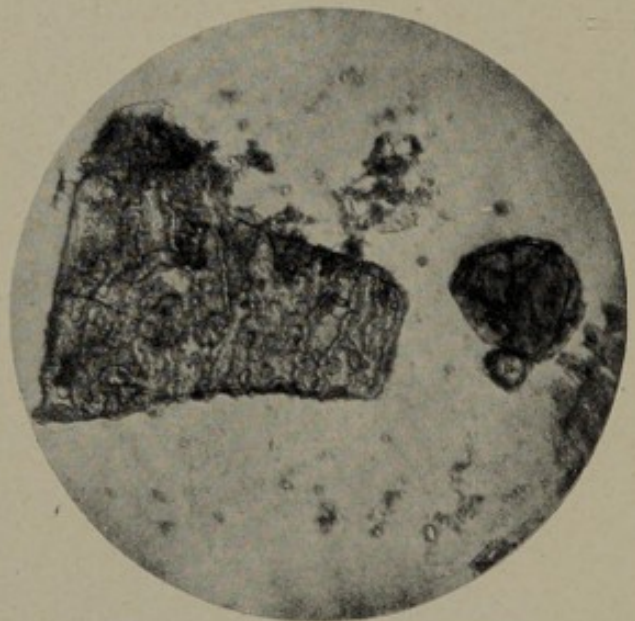
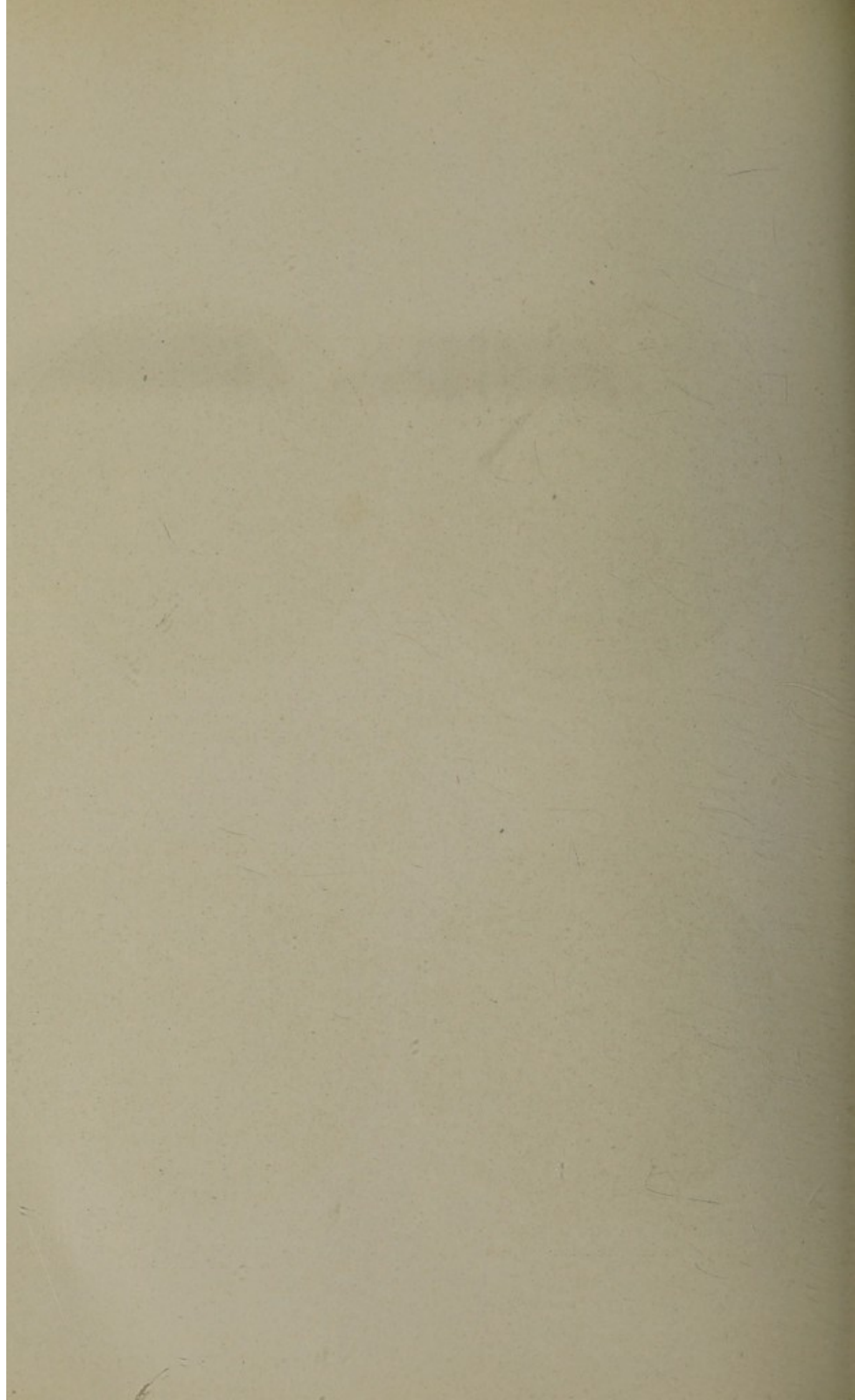


FIG. 196.—Adulterated Allspice,  $\times 110$ .  
Showing a large fragment of the seed skin of  
cayenne at the left.







SPICES.

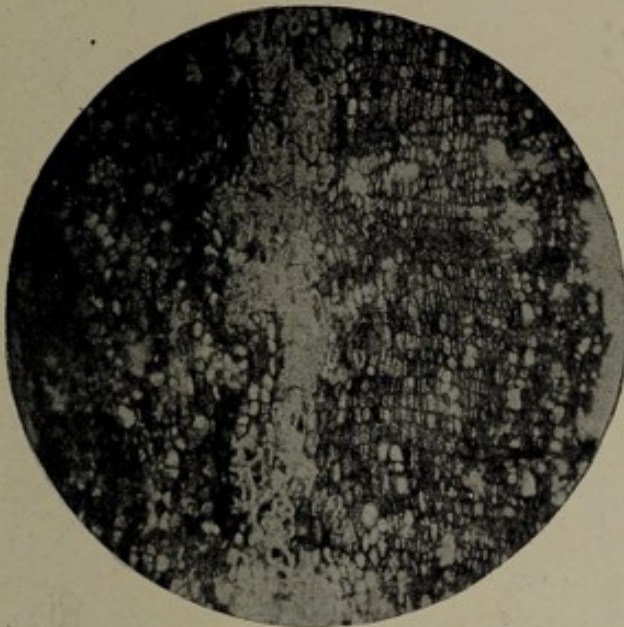


FIG. 197.—Cassia Bark,  $\times 45$ .  
Transverse section through the bark.



FIG. 198.—Cassia Bark,  $\times 45$ .  
Longitudinal section.

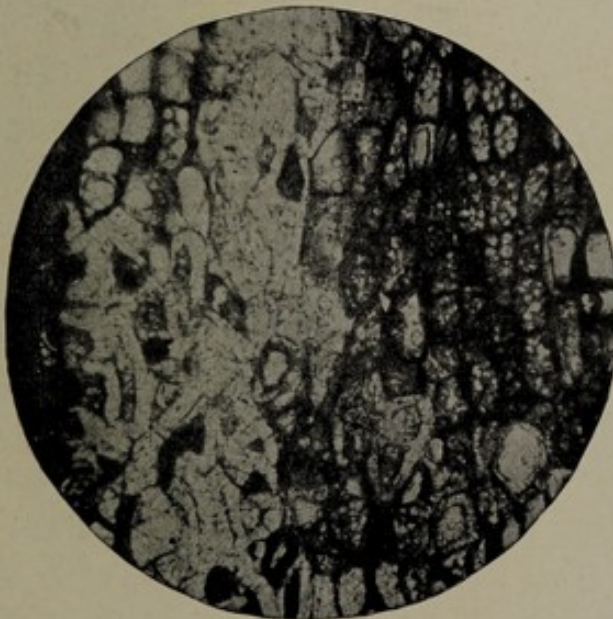


FIG. 199.—Cassia Bark,  $\times 110$ .  
Transverse section, showing cork cells, parenchyma, and stone cells.

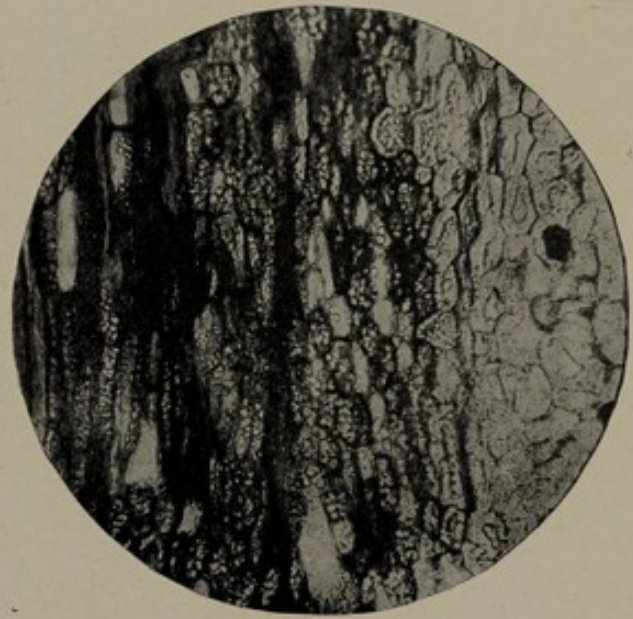
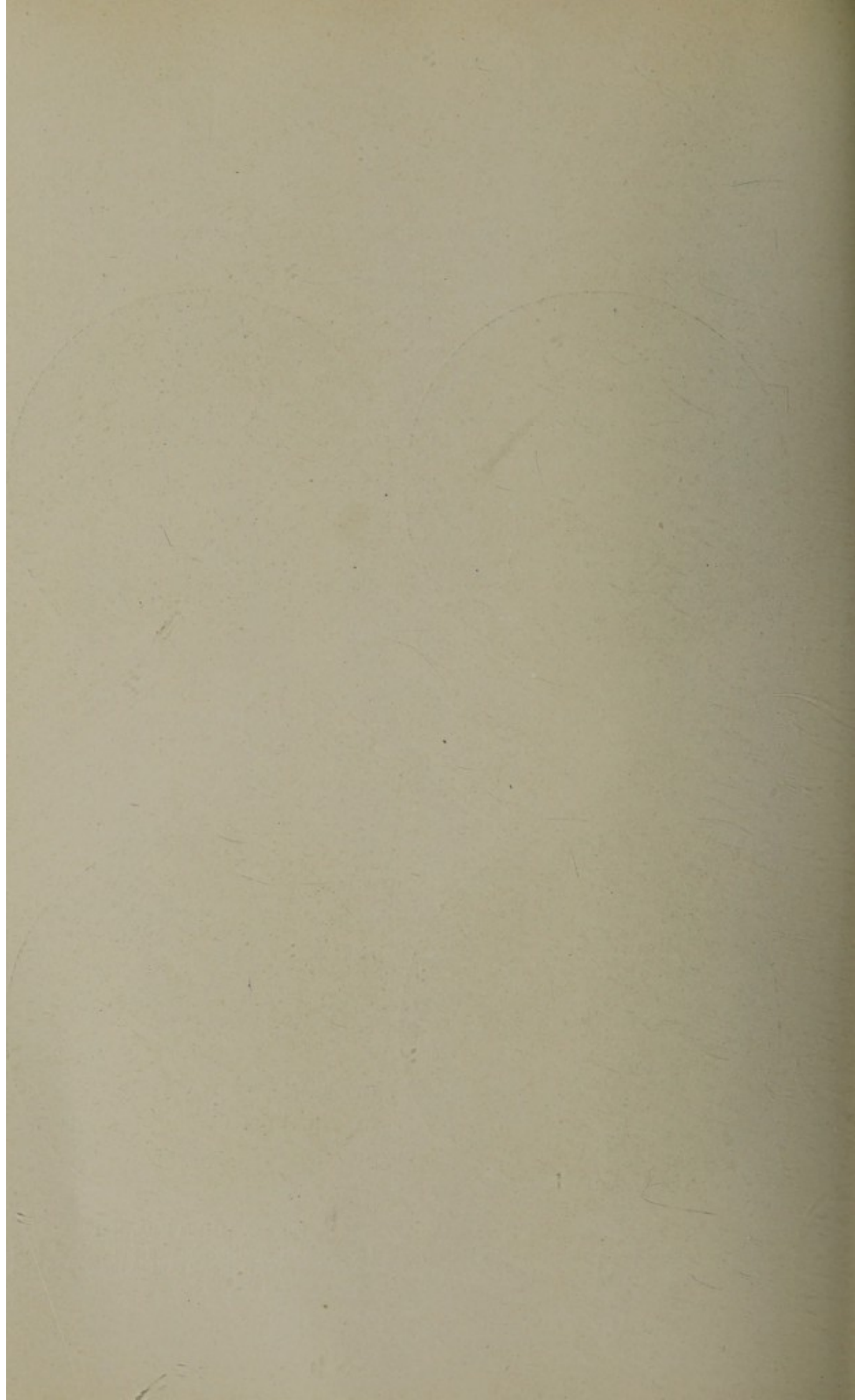


FIG. 200.—Cassia Bark,  $\times 110$ .  
Longitudinal section, showing bunches of bast fibers at the left, starch cells in the center, and stone cells at the right.







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FIG. 201.—Ceylon Cinnamon Bark,  $\times 110$ .  
Transverse section, showing many bast fibers and  
starch cells.



FIG. 202.—Ceylon Cinnamon Bark,  $\times 110$ .  
Longitudinal section, showing bast fibers, stone  
cells, and parenchyma.

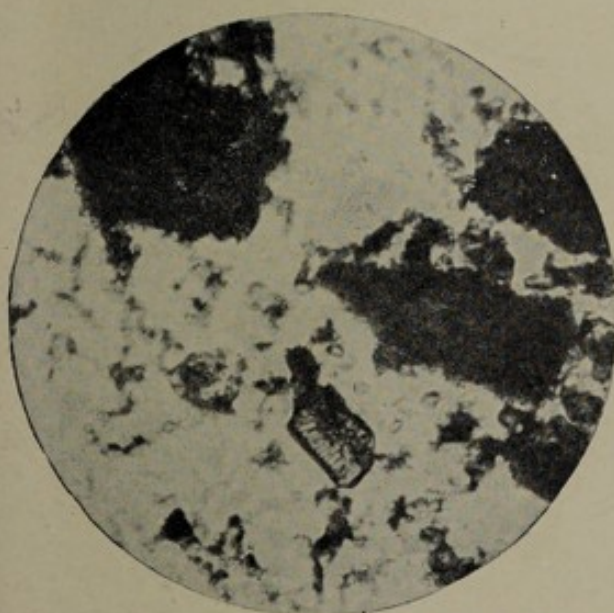
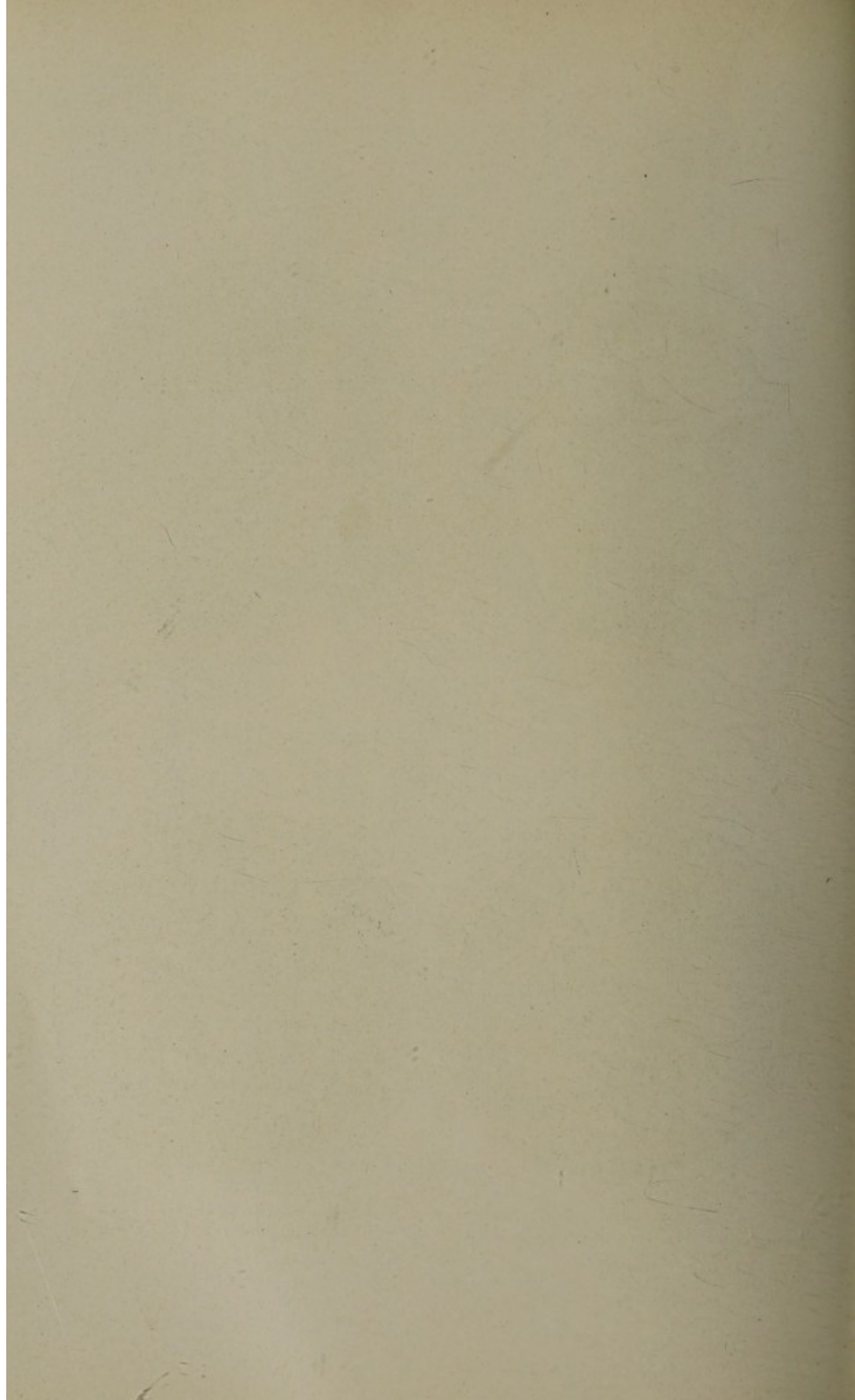


FIG. 203.—Powdered Cassia,  $\times 110$ .  
Showing stone cells, starch, and corky tissue.



FIG. 204.—Powdered Cassia,  $\times 110$ .  
Showing bast fibers and starch.







SPICES.

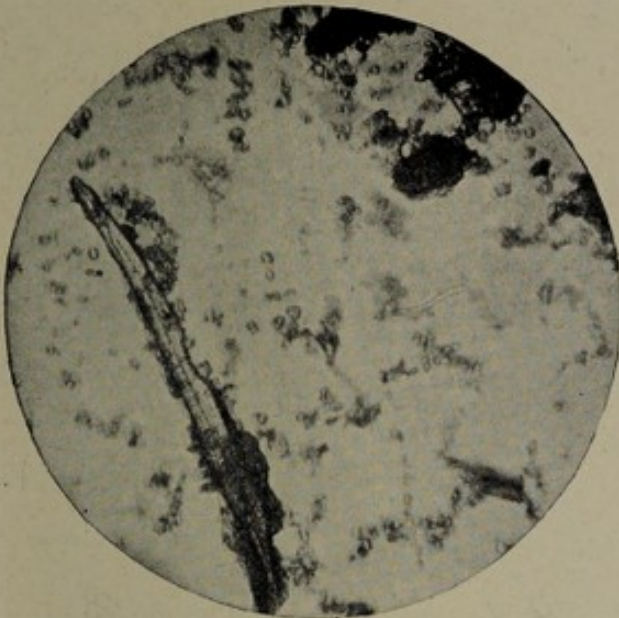


FIG. 205.—Powdered Cassia,  $\times 110$ .  
Showing large bast fiber and starch grains.

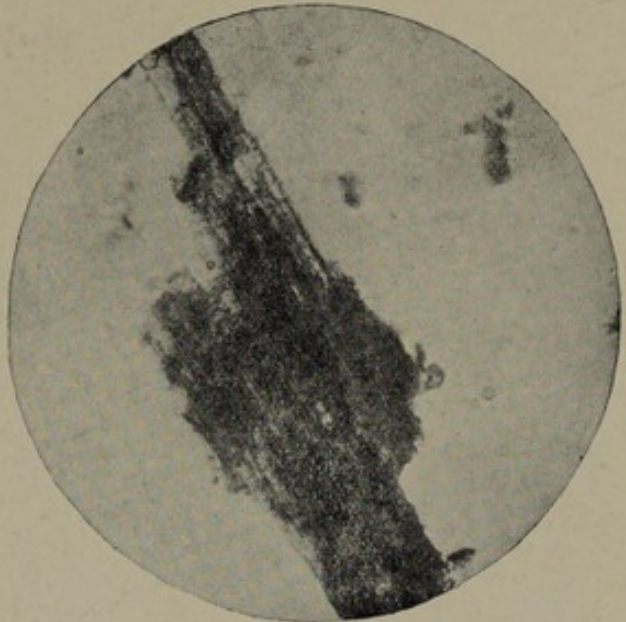


FIG. 206.—Adulterated Cassia,  $\times 110$ .  
A mass of foreign bark.

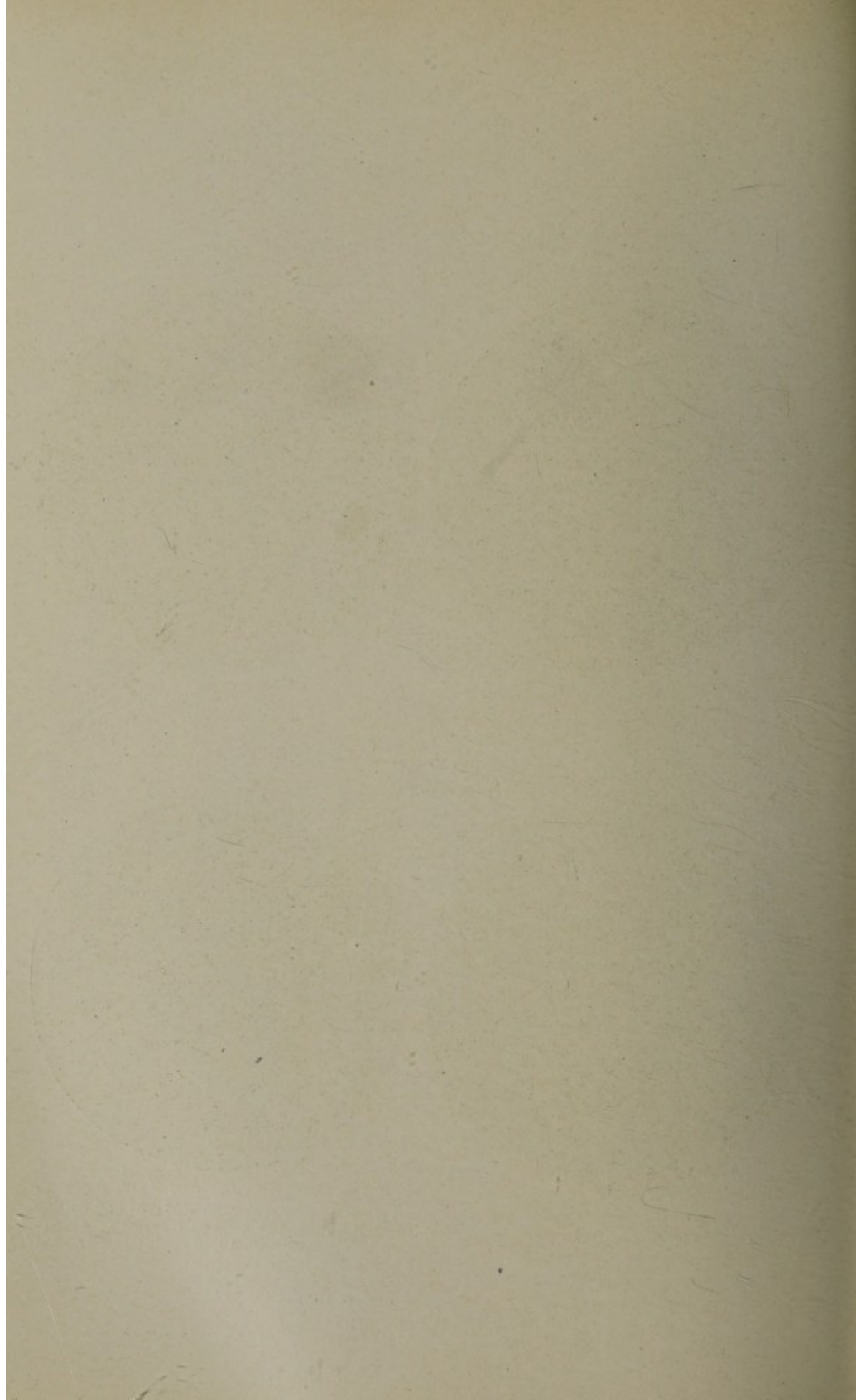


FIG. 207.—Cayenne,  $\times 110$ .  
Transverse section through pericarp.



FIG. 208.—Cayenne,  $\times 110$ .  
Transverse section through seed coat and part of  
endosperm. Collapsed parenchyma cells separate  
endosperm from long epidermal cells.







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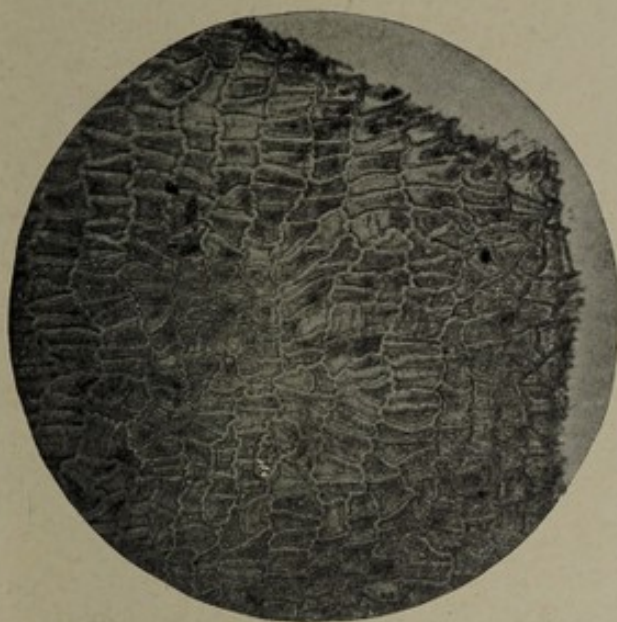


FIG. 209.—Cayenne,  $\times 110$ .  
Surface view of fruit epidermis.

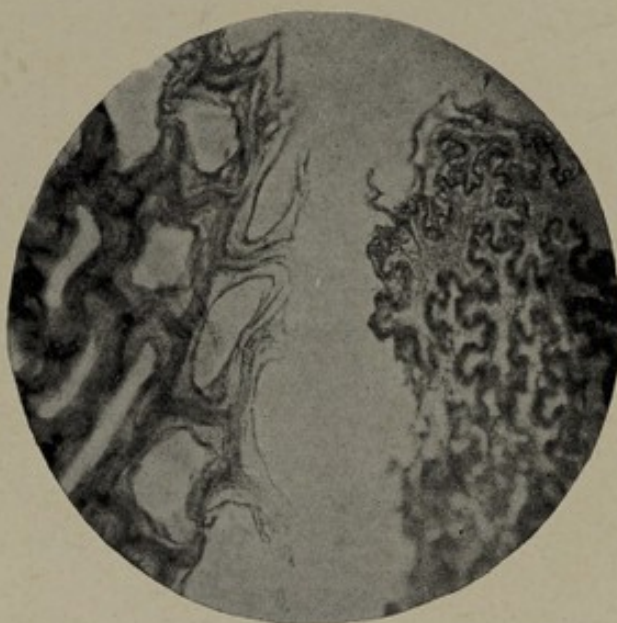


FIG. 210.—Cayenne,  $\times 110$ .  
Surface view of two layers of seed coat.

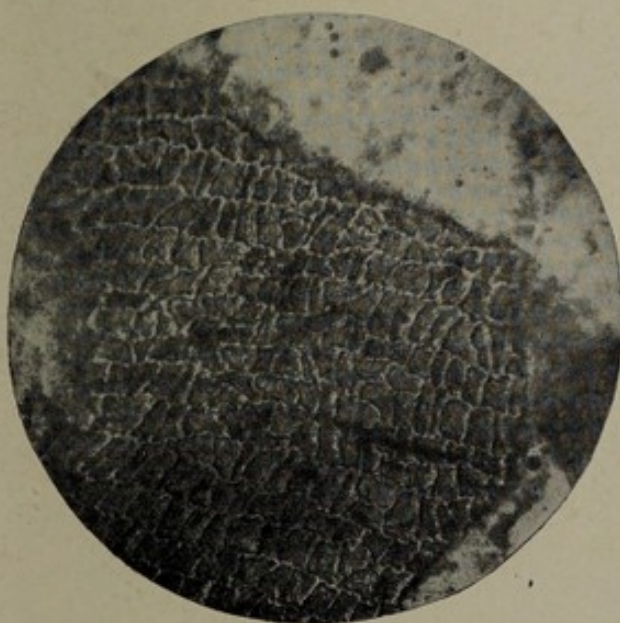


FIG. 211.—Powdered Cayenne,  $\times 110$ .  
A large mass of fruit epidermis.

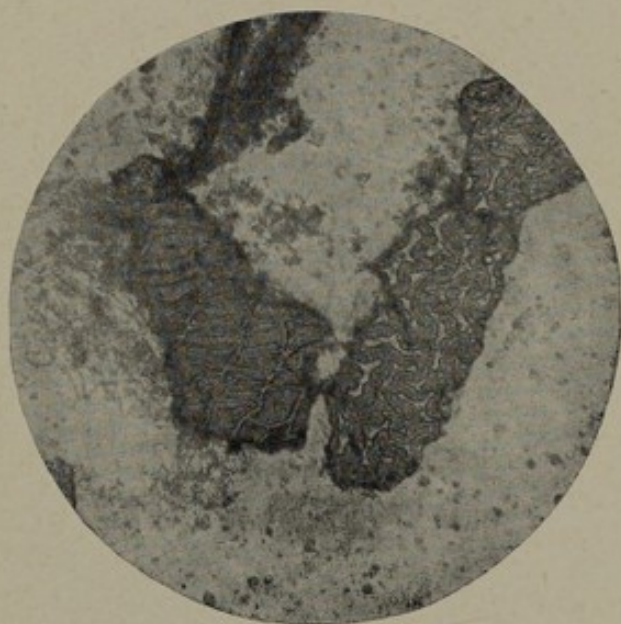
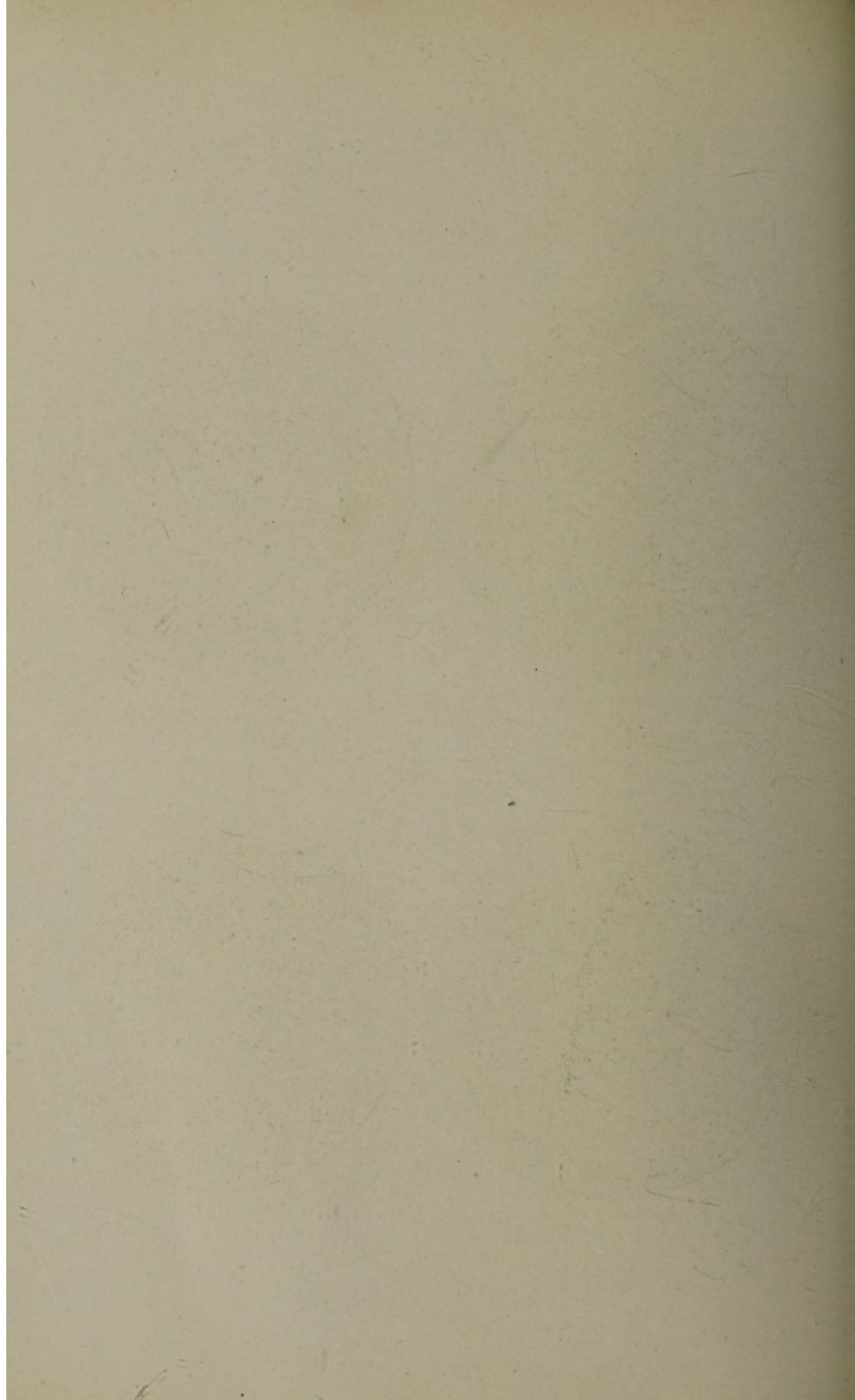


FIG. 212.—Powdered Cayenne,  $\times 110$ .  
Showing chiefly two of the seed coat layers.







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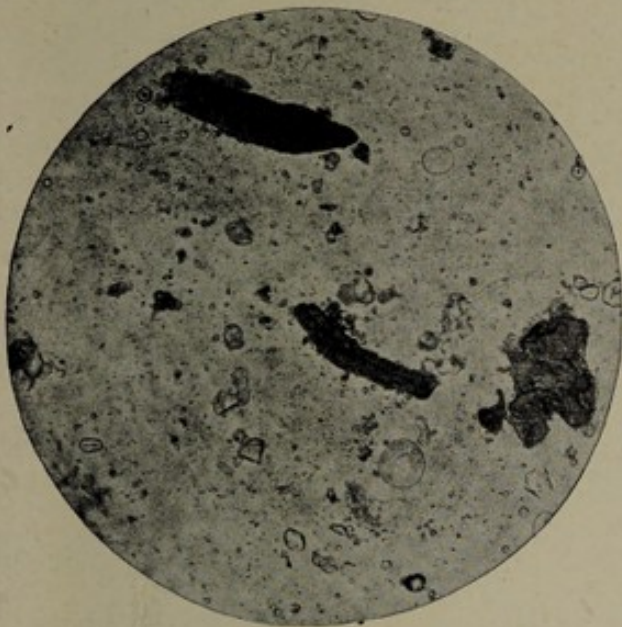


FIG. 213.—Adulterated Cayenne,  $\times 130$ .  
Corn and wheat starch and coconut shells appear chiefly. A bit of cayenne is shown at the right.



FIG. 214.—Adulterated Cayenne,  $\times 214$ .  
The central mass is ground red wood, surrounded by corn starch grains.

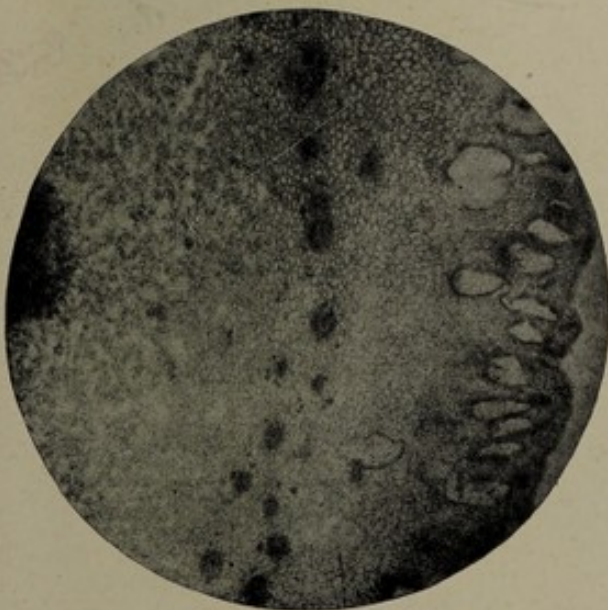
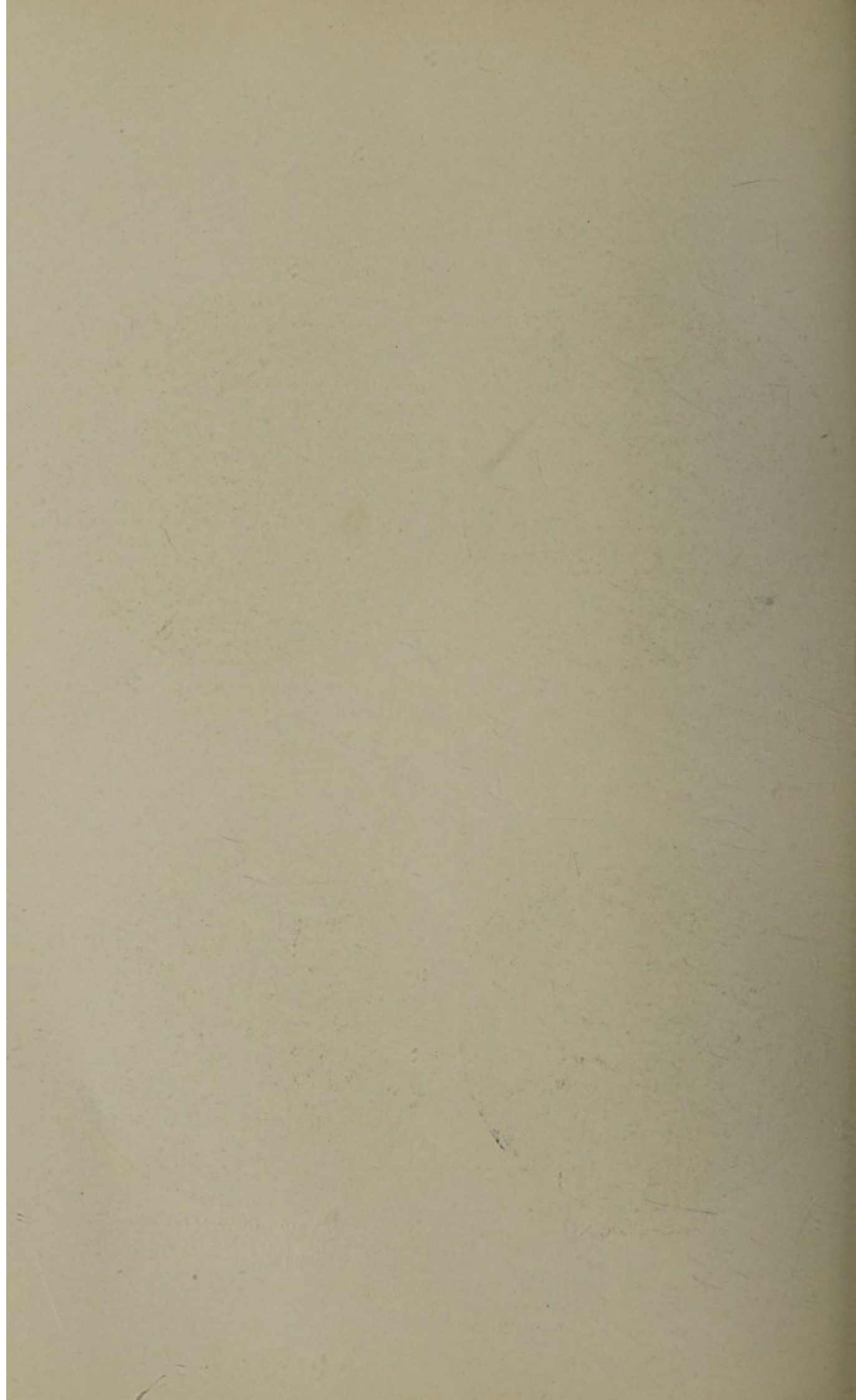


FIG. 215.—Clove,  $\times 65$ .  
Transverse section from the center outward to epidermis, showing parenchyma.



FIG. 216.—Clove,  $\times 110$ .  
Transverse section near epidermis, showing large oil cavities.







SPICES.

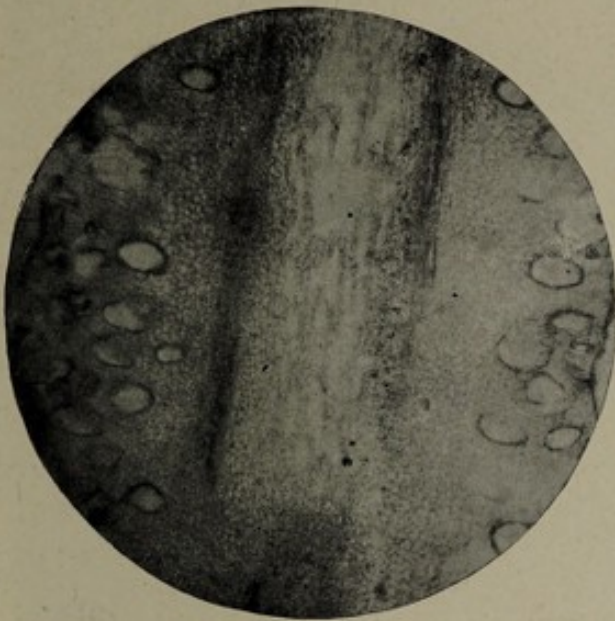


FIG. 217.—Clove,  $\times 28$ .  
Longitudinal section through entire clove.

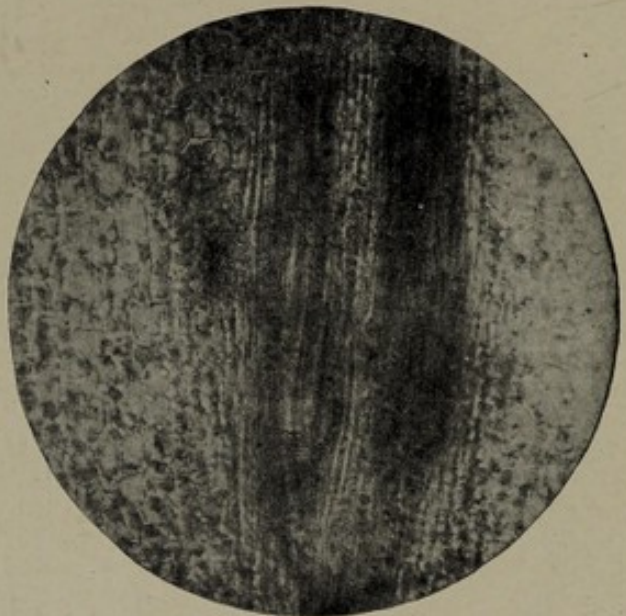


FIG. 218.—Clove,  $\times 70$ .  
Central longitudinal section, showing duct bundles.

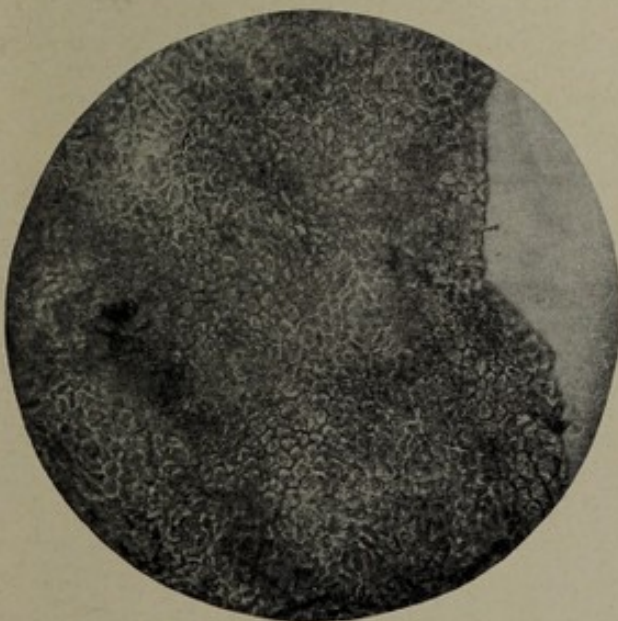


FIG. 219.—Clove,  $\times 110$ .  
Surface view of epidermis.



FIG. 220.—Powdered Cloves,  $\times 130$ .  
Dense, spongy tissue, with small oil drops.







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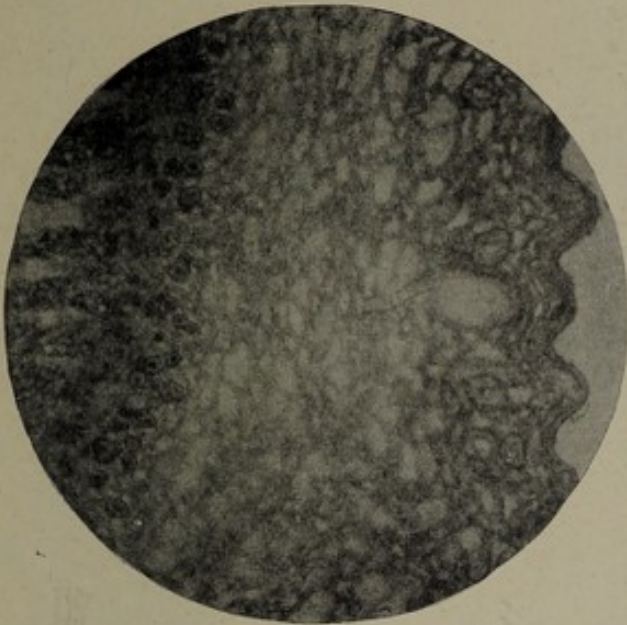


FIG. 221.—Clove Stem,  $\times 70$ .

Transverse section through outer part of stem, showing bast fibers at the left, parenchyma in the center, and stone cells near the epidermis.



FIG. 222.—Clove Stem,  $\times 25$ .

Central longitudinal section through entire stem, showing bast fibers in the center, and stone cells at the right.



FIG. 223.—Clove Stem,  $\times 70$ .

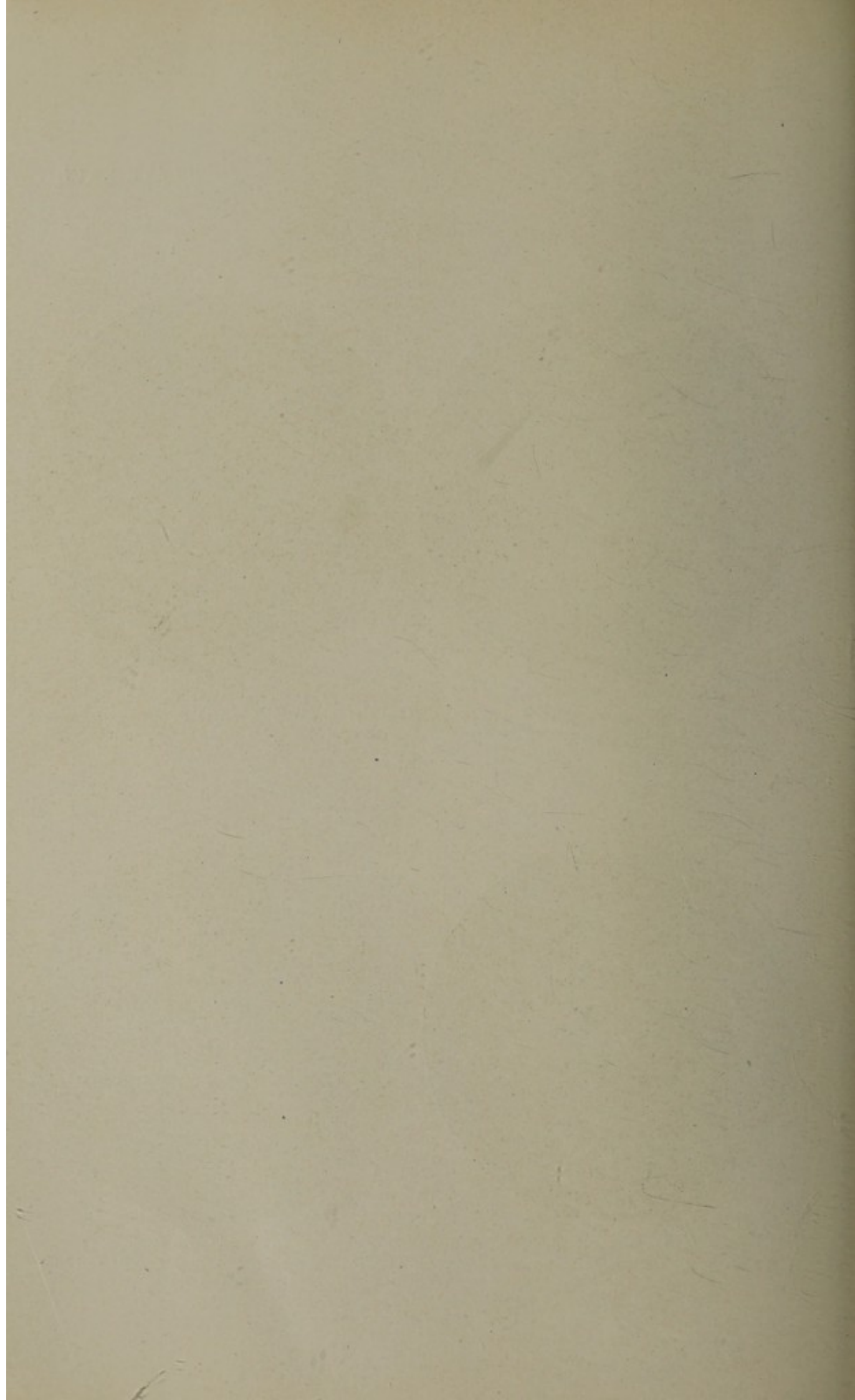
Longitudinal section, showing the stone cells.



FIG. 224.—Powdered Clove Stems,  $\times 110$ .

Showing fragments of tissues, stone cells, and bast fibers.







SPICES.

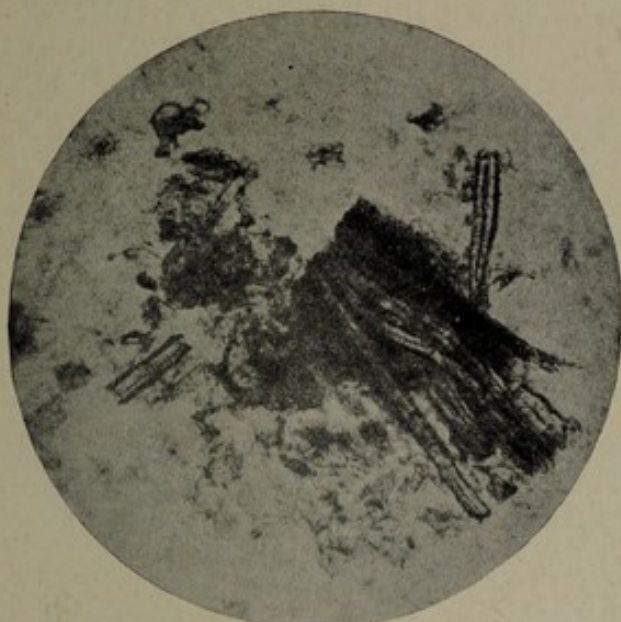


FIG. 225.—Powdered Clove Stems,  $\times 110$ .  
Showing bundle of bast fibers.

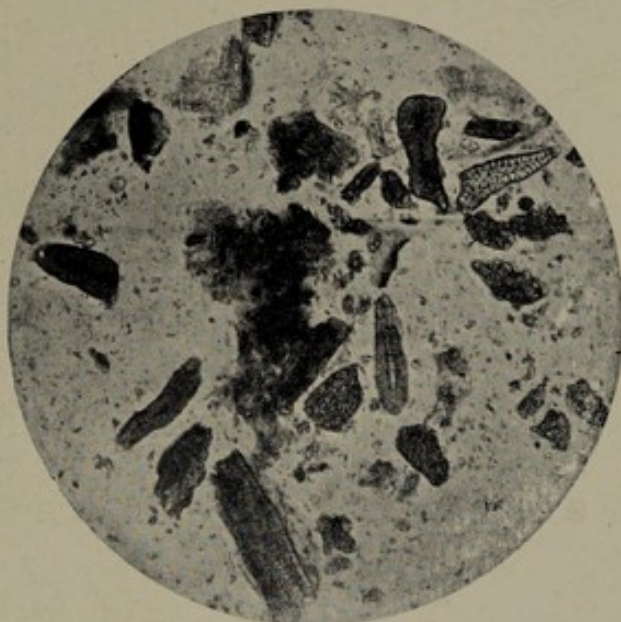


FIG. 226.—Adulterated Cloves,  $\times 130$ .  
Showing chiefly stone cells of cocoanut shells.

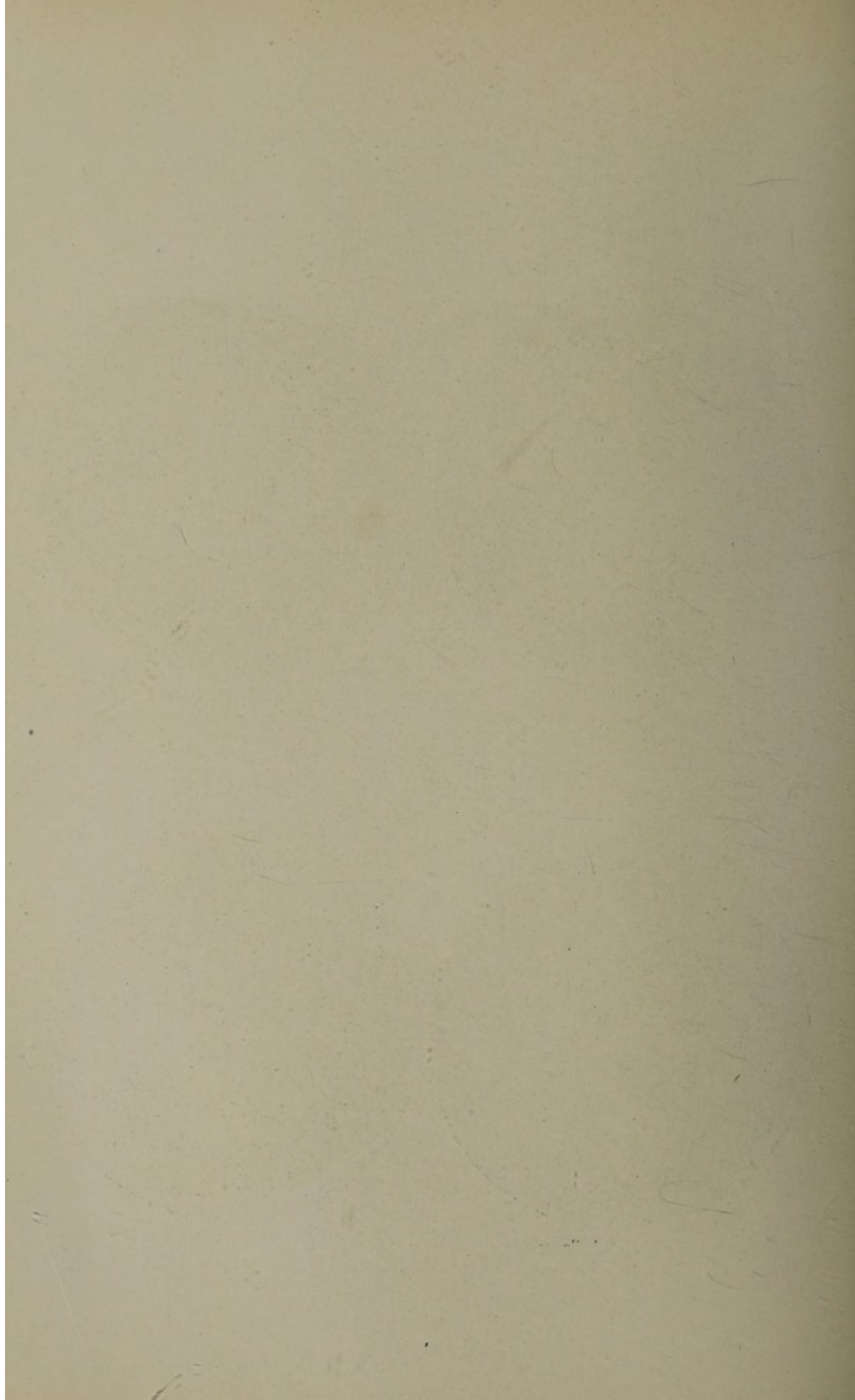


FIG. 227.—Adulterated Cloves,  $\times 130$ .  
With large admixture of cocoanut shells.



FIG. 228.—Ginger,  $\times 110$ .  
Transverse section, showing starch cells with  
contents.







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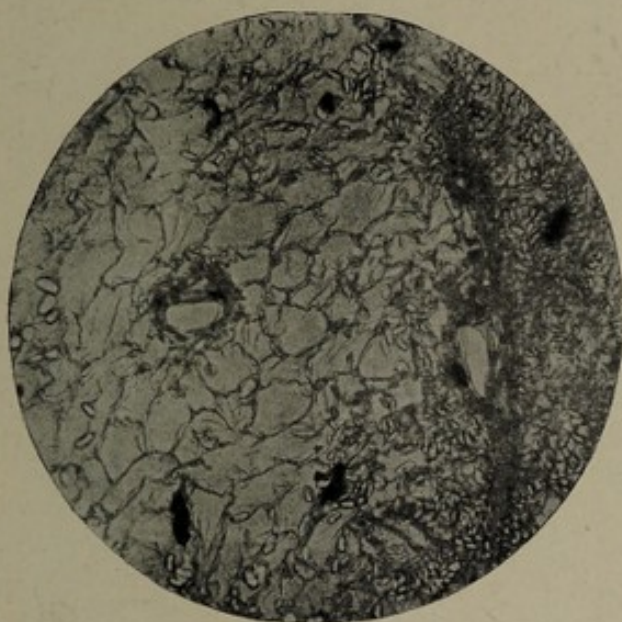


FIG. 229.—Ginger,  $\times 110$ .  
Transverse section, showing parenchyma, starch grains, and duct vessels.



FIG. 230.—Ginger,  $\times 110$ .  
Longitudinal section, showing spiral ducts and pigment cells.

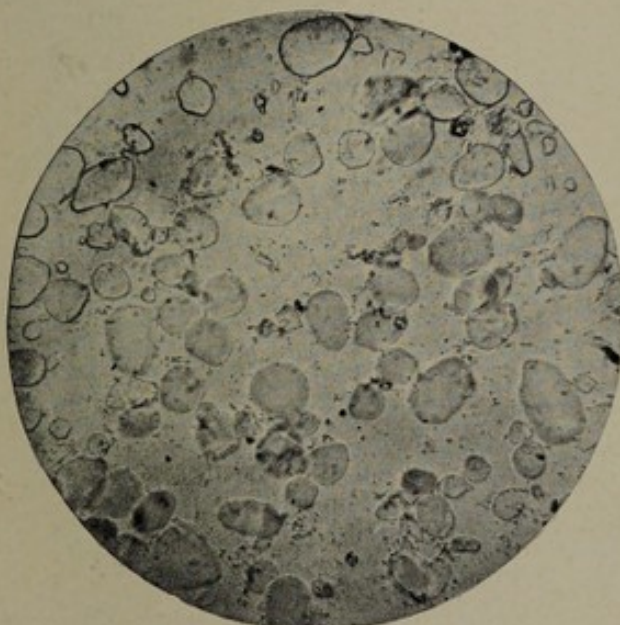


FIG. 231.—Ginger Starch,  $\times 220$ .

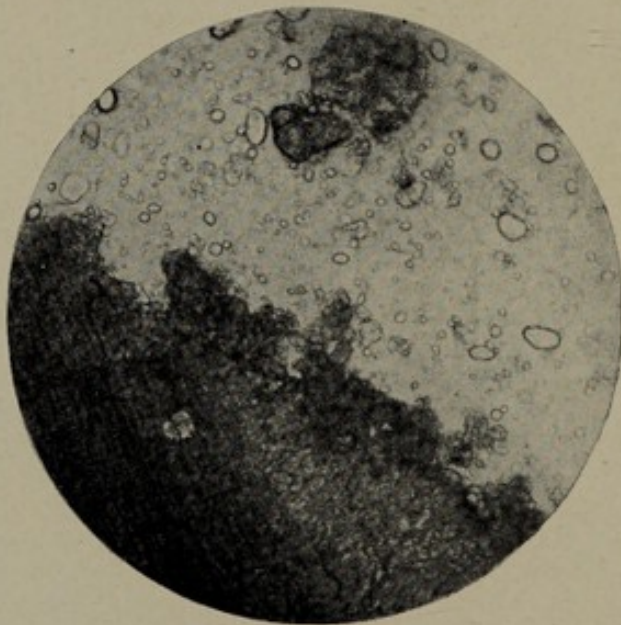
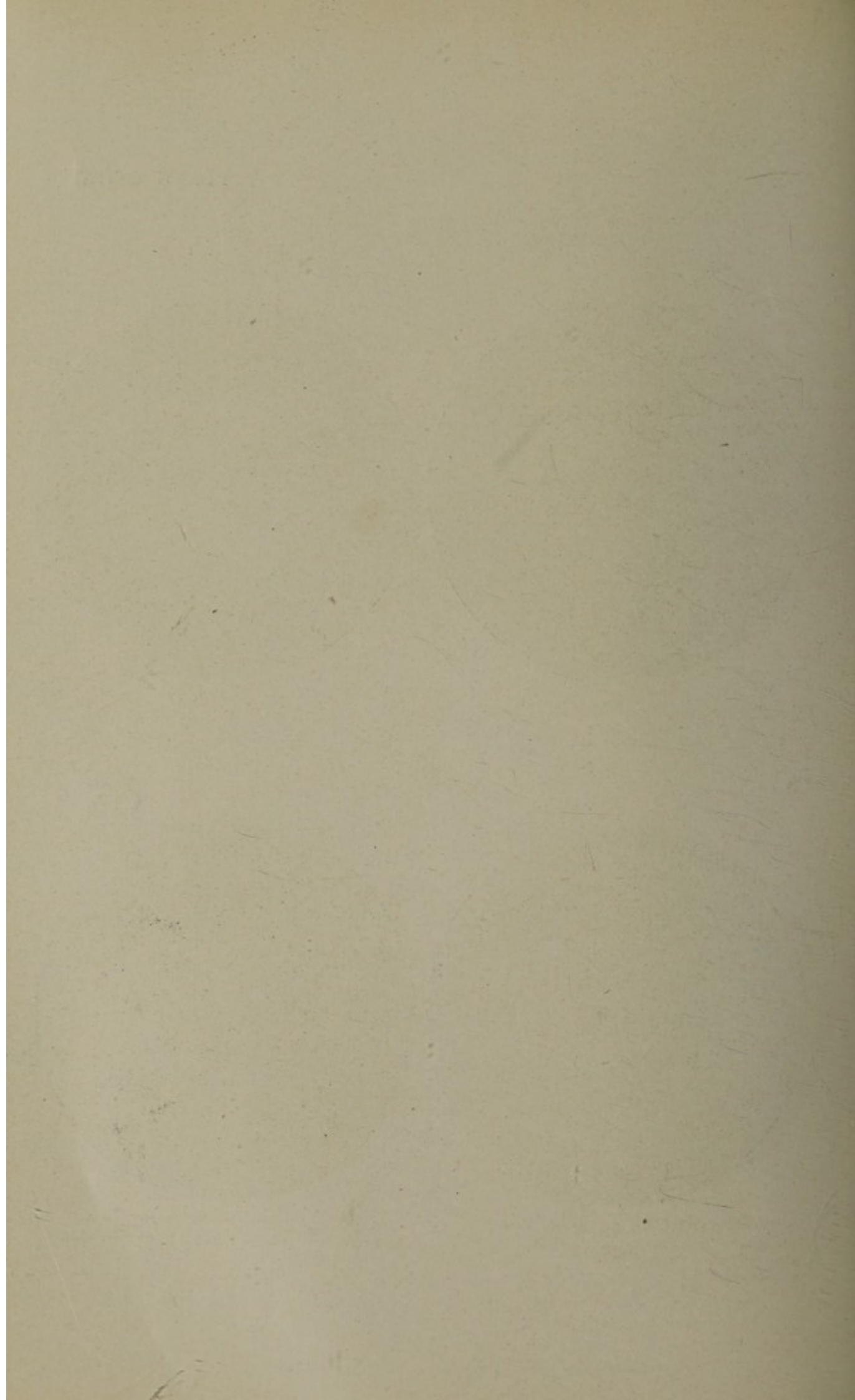


FIG. 232.—Adulterated Ginger,  $\times 130$ .  
A mass of wheat bran tissue is most conspicuous.







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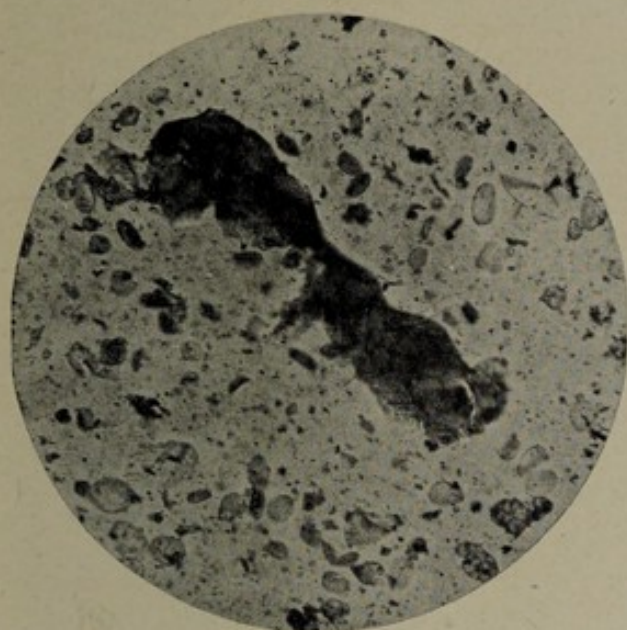


FIG. 233.—Adulterated Ginger,  $\times 130$ .  
The central dark mass is a yellow fragment of turmeric.

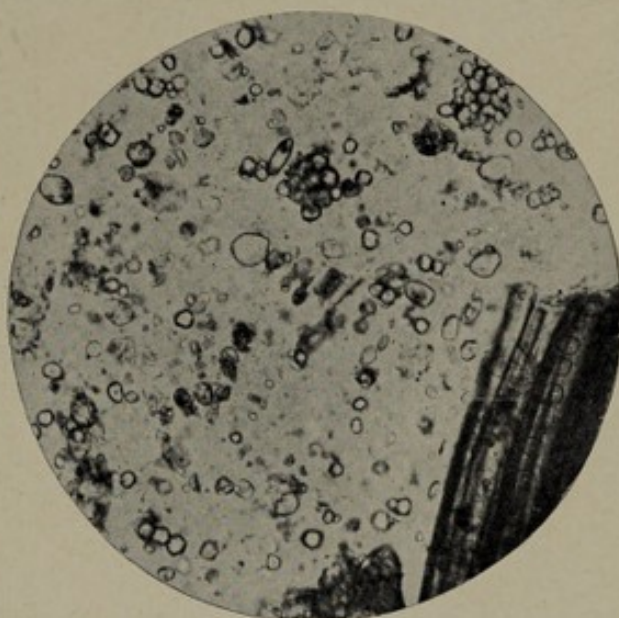


FIG. 234.—Adulterated Ginger,  $\times 130$ .  
Containing a large admixture of corn and wheat starches.

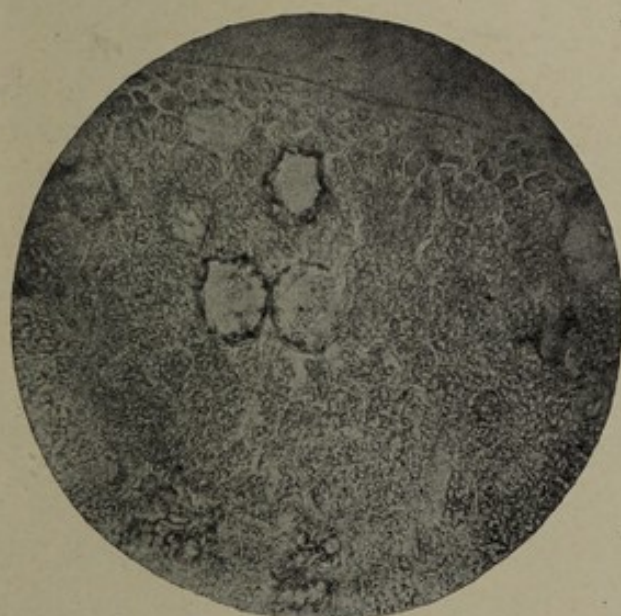
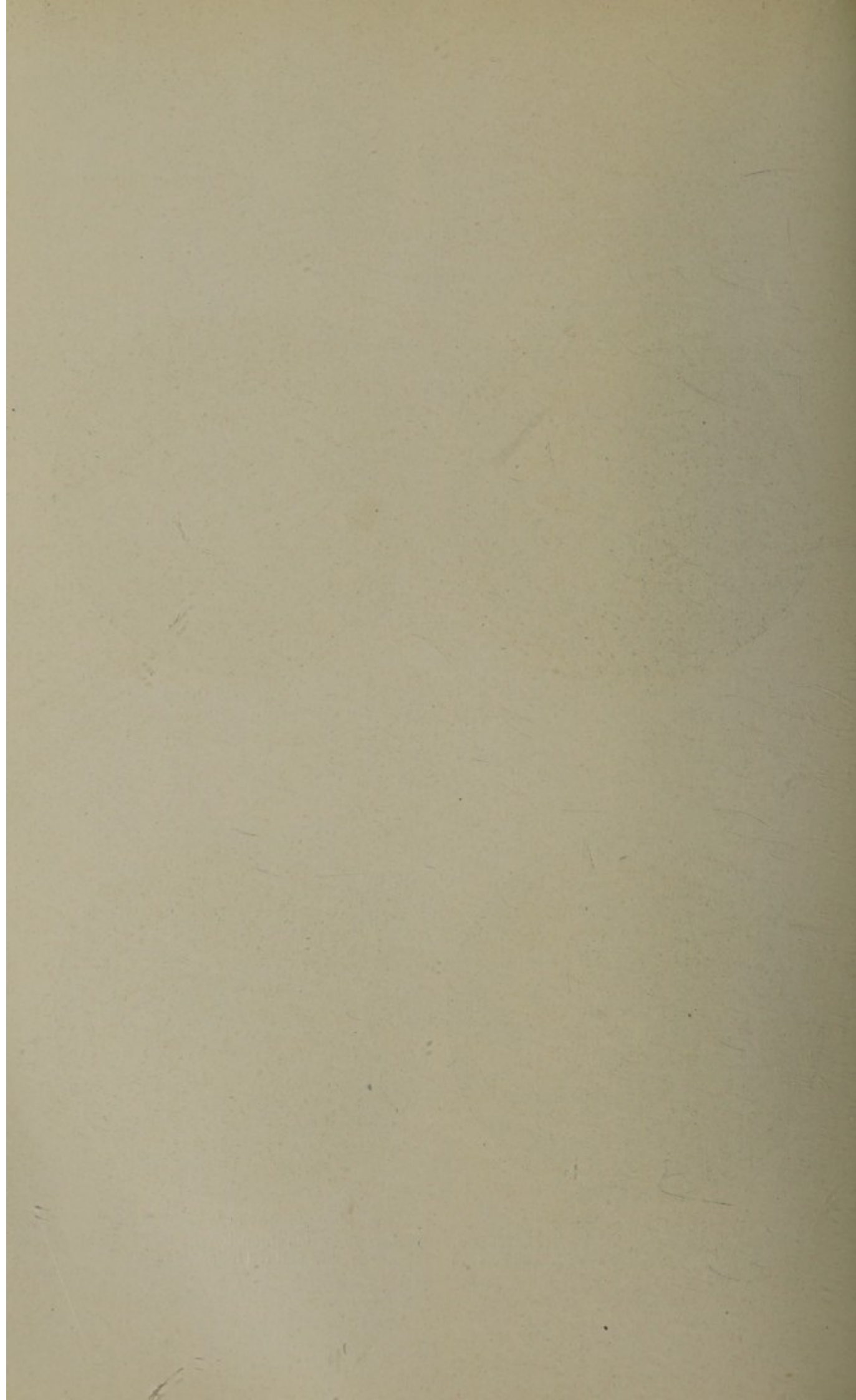


FIG. 235.—Penang Mace,  $\times 110$ .  
Transverse section through epidermis and oil cells, showing also parenchyma with contents of amylopectin.



FIG. 236.—Bombay or Wild Mace,  $\times 110$ .  
Transverse section through outer layers, showing yellow and red resinous lumps.







SPICES.

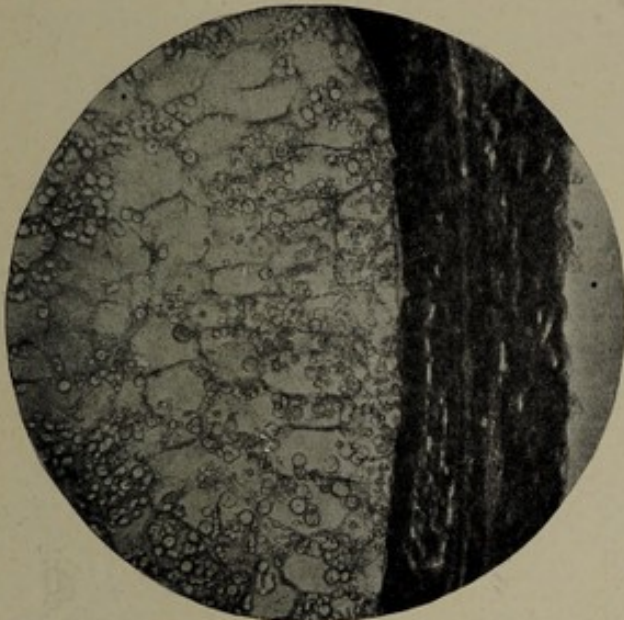


FIG. 237.—Nutmeg,  $\times 110$ .  
Transverse section through the exterior and interior teguments of the seed and part of the endosperm, showing starch cells.

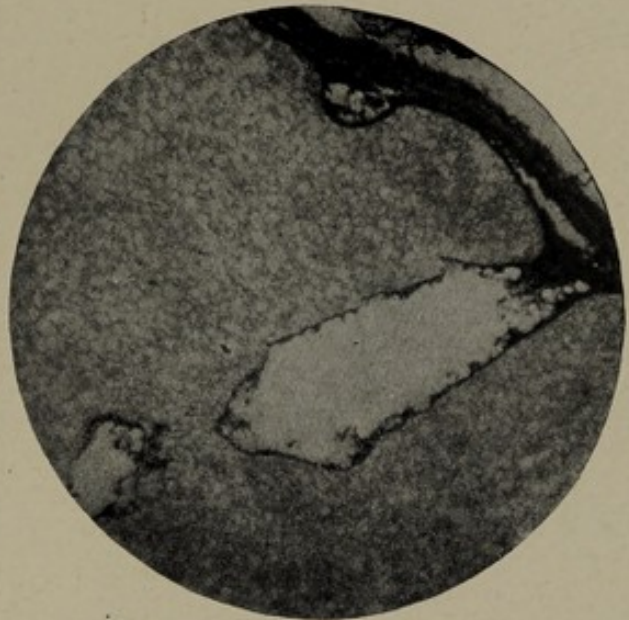


FIG. 238.—Nutmeg,  $\times 25$ .  
Transverse section near exterior of seed.



FIG. 239.—Nutmeg,  $\times 110$ .  
Surface view of seed coat, showing also portions of underlying tissues.

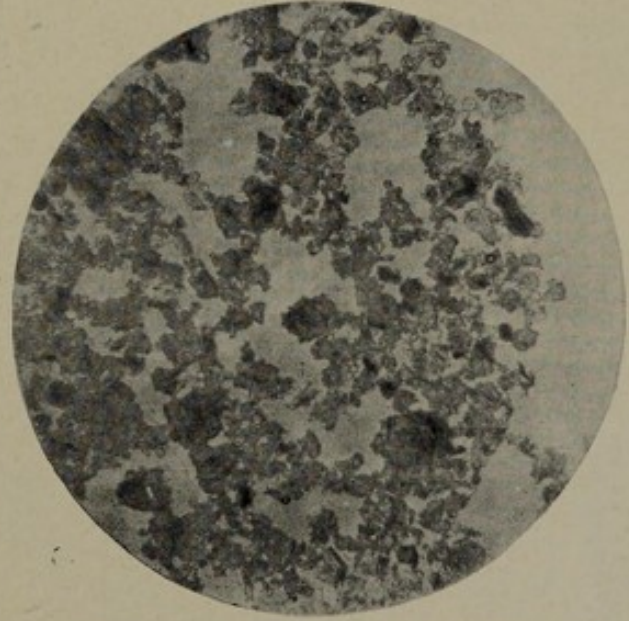
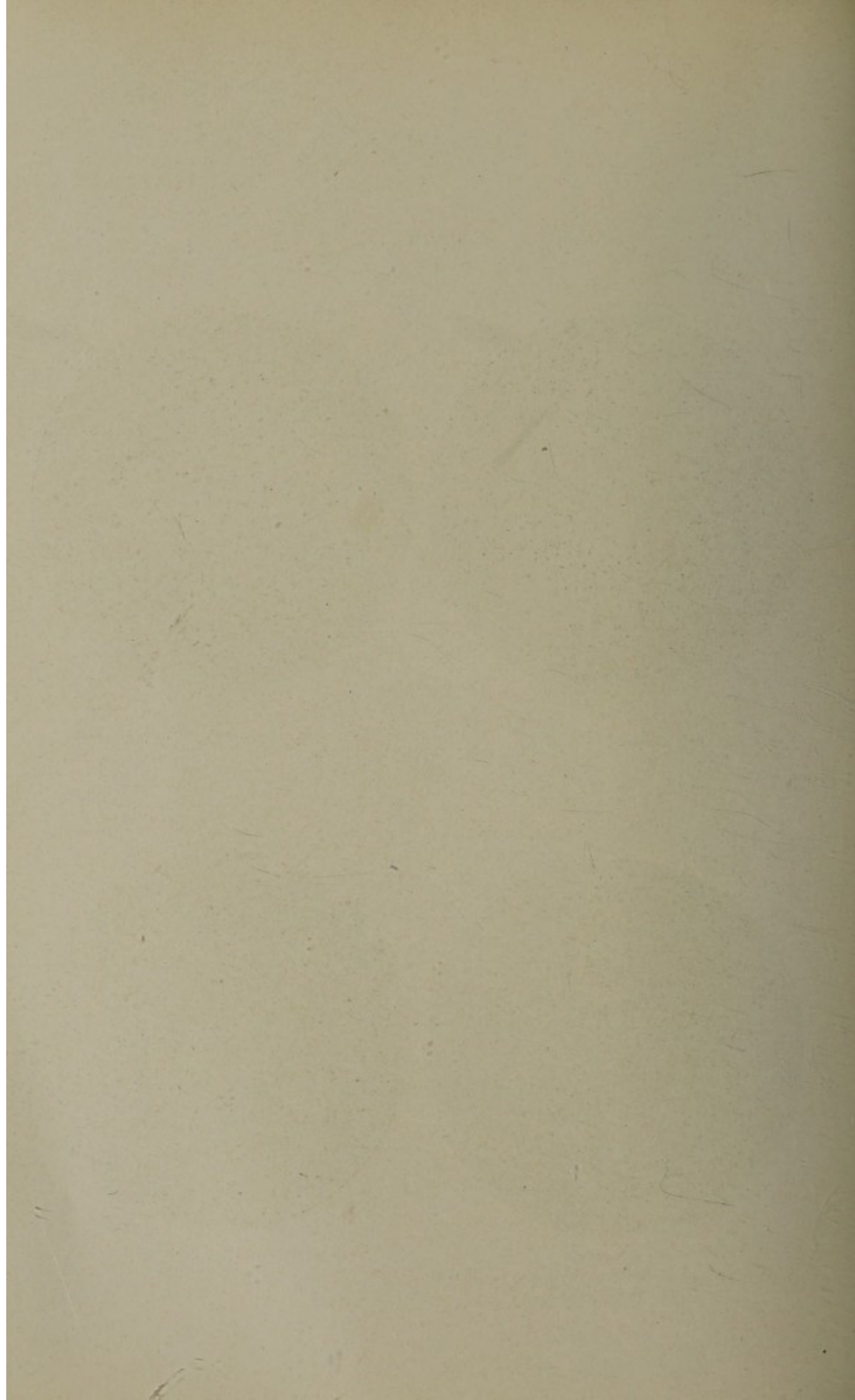


FIG. 240.—Powdered Nutmeg,  $\times 110$ .







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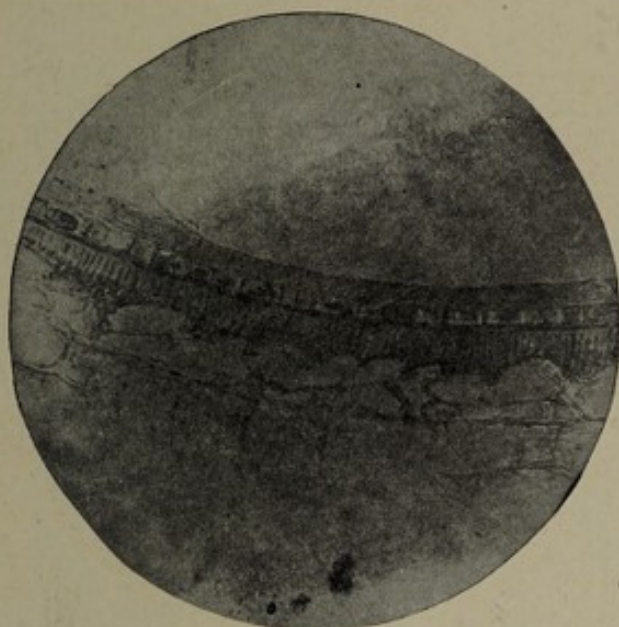


FIG. 241.—White Mustard,  $\times 110$ .  
Transverse section through mucilaginous epidermis, sub-epidermal parenchyma layer (square cells), palisade cells, and broken parenchyma layer of the hull.

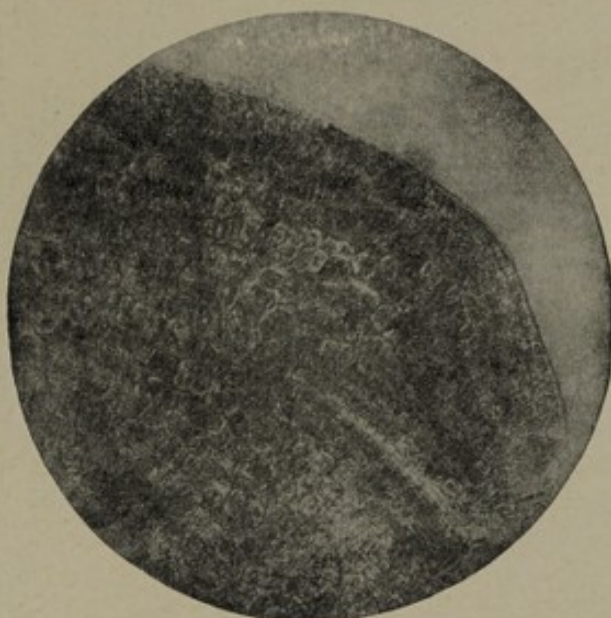


FIG. 242.—White Mustard,  $\times 110$ .  
Transverse section through the tissue of the radicle.

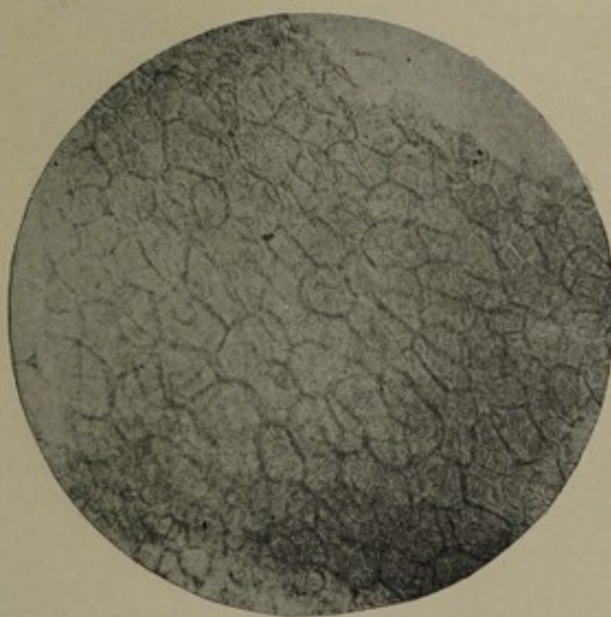


FIG. 243.—White Mustard  $\times 110$ .  
Surface view of two layers of the hull or seed coat.

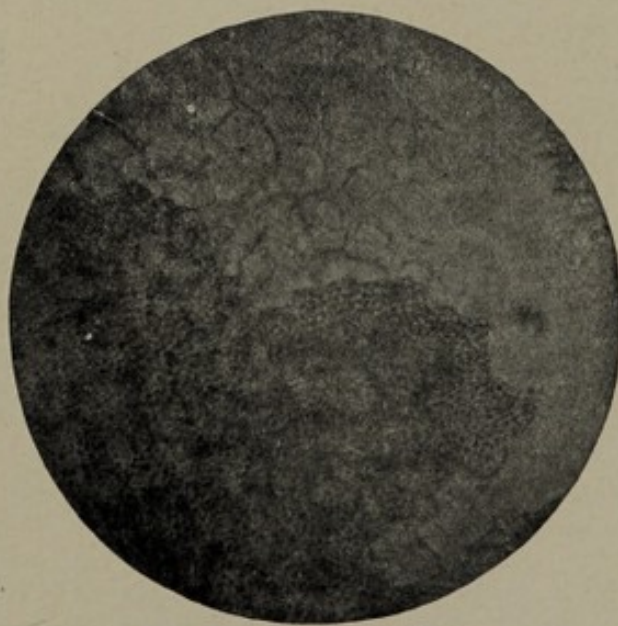
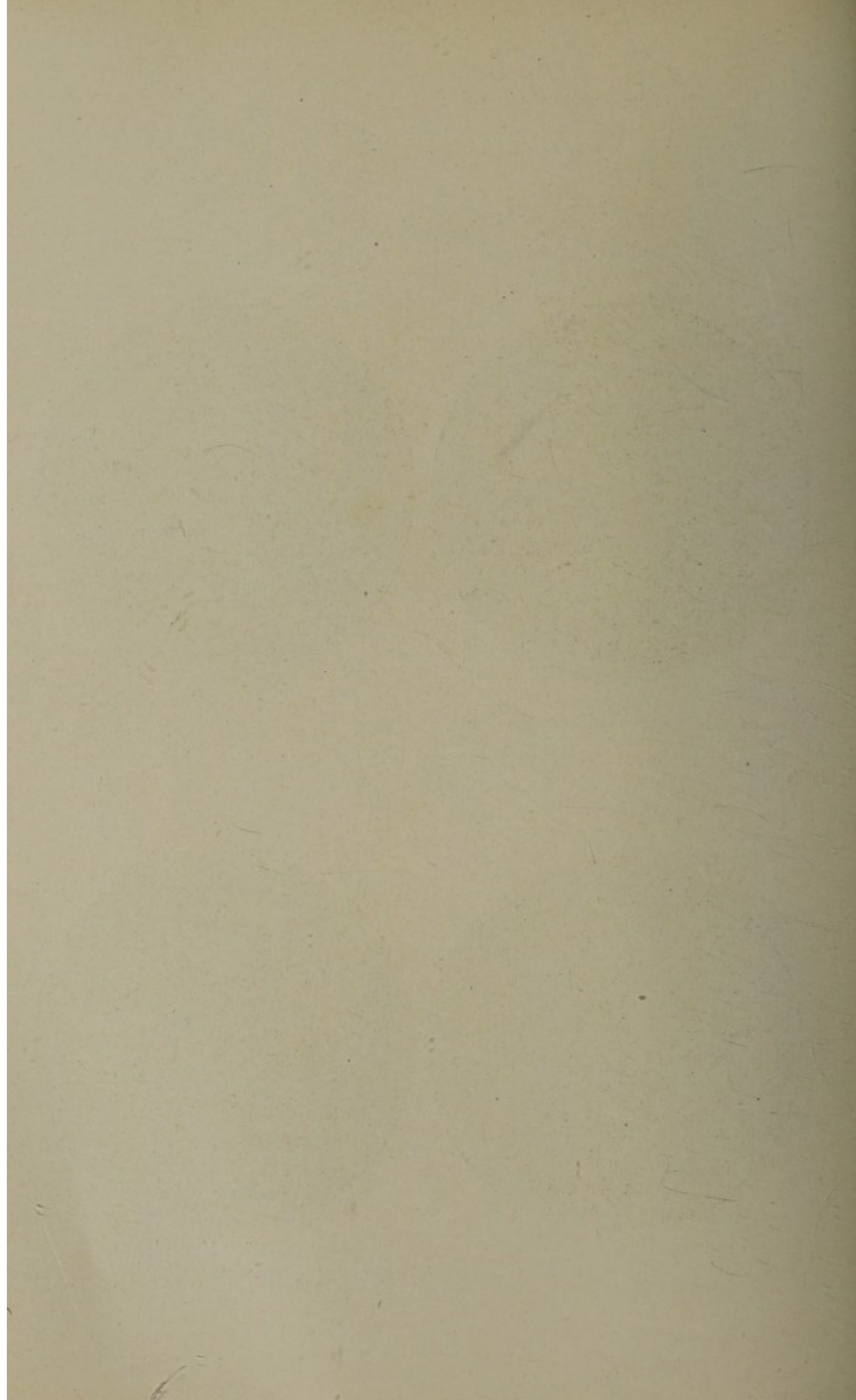


FIG. 244.—White Mustard,  $\times 110$ .  
Surface section through palisade cells and underlying layer of the seed coat.







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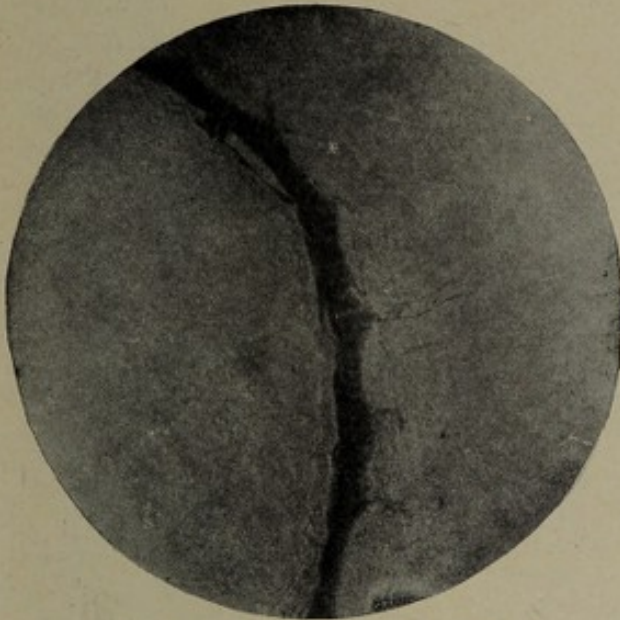


FIG. 245.—Black Mustard,  $\times 110$ .

Transverse section, showing fragments of the epidermis and dark colored palisade cells of the seed coat.

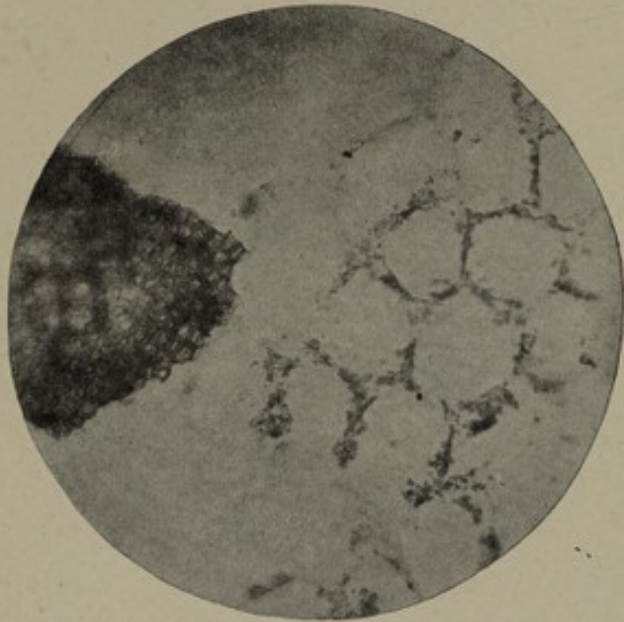


FIG. 246.—Black Mustard,  $\times 110$ .

Surface view of two of the seed coat layers.

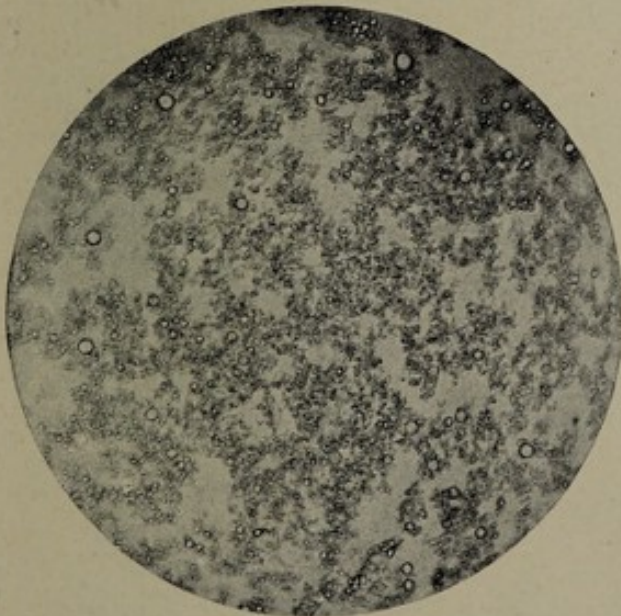


FIG. 247.—Ground Mustard,  $\times 130$ .  
Ground without removal of the oil.

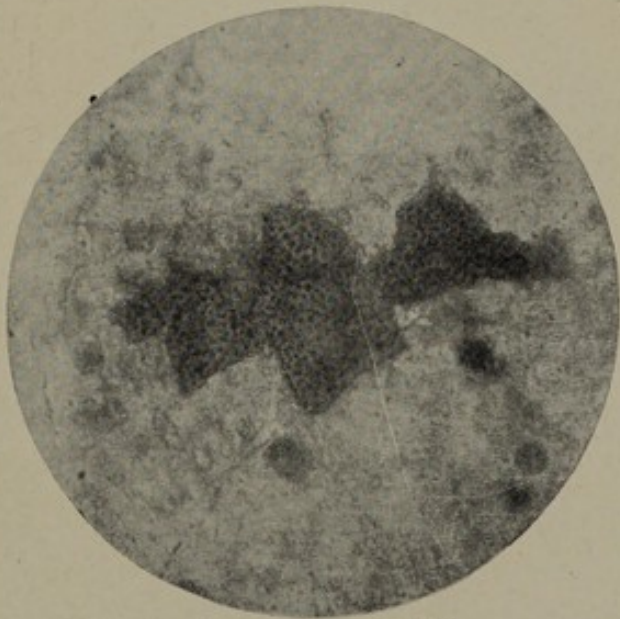
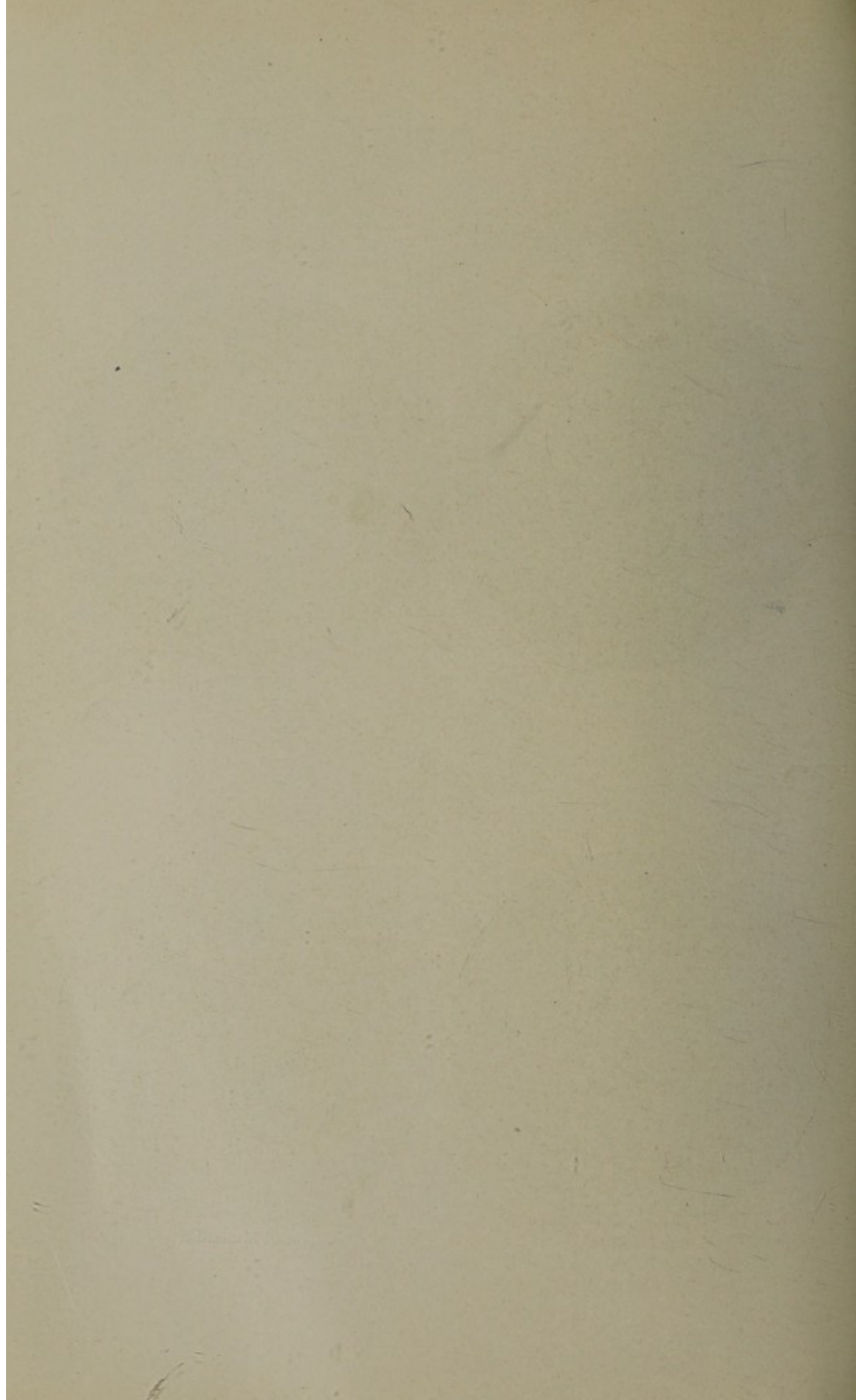


FIG. 248.—Ground Mustard Hulls,  $\times 110$ .







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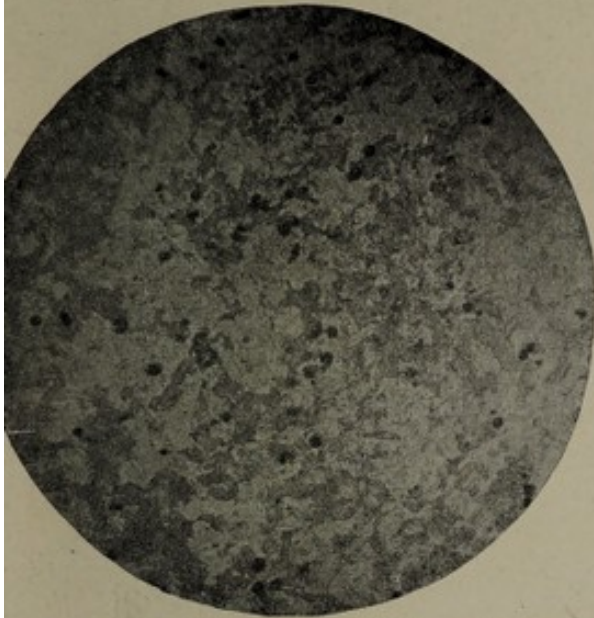


FIG. 249.—Dakota Mustard Flour,  $\times 110$ .  
Dark spots show starch grains of foreign weed seed, stained with iodine.

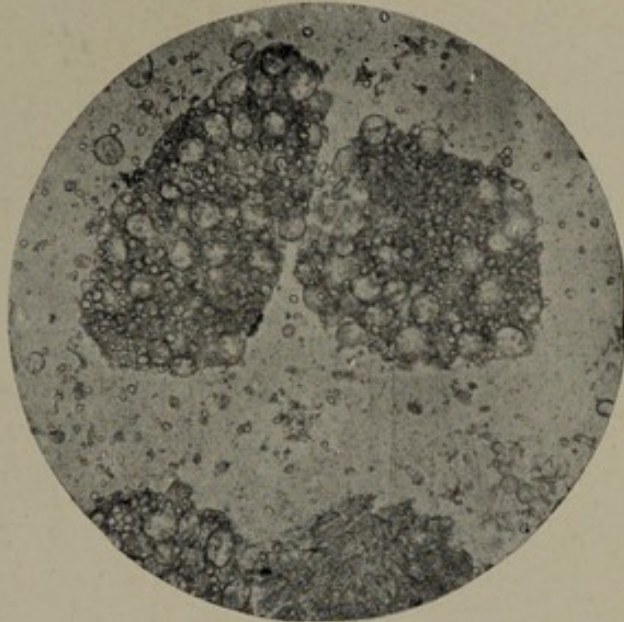


FIG. 250.—Adulterated Mustard Flour,  $\times 130$ .  
Showing masses of wheat starch.

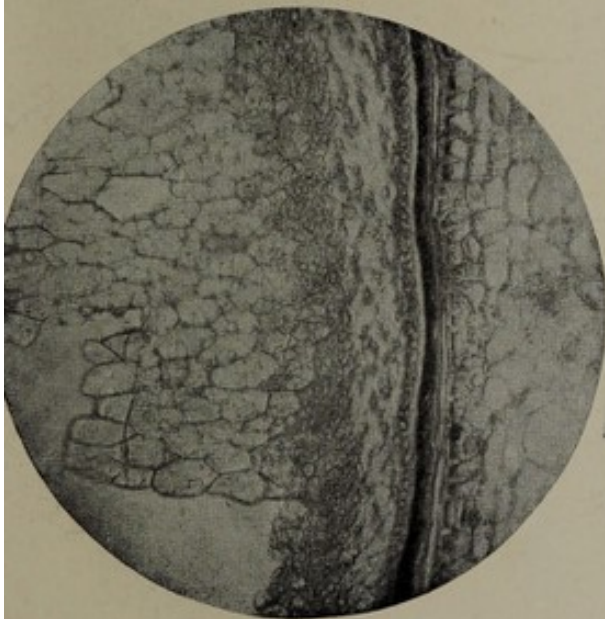


FIG. 251.—Pepper,  $\times 110$ .  
Transverse section through inner part of pericarp (including parenchyma and seed coat layers) and portion of perisperm, showing starch and oil cells.

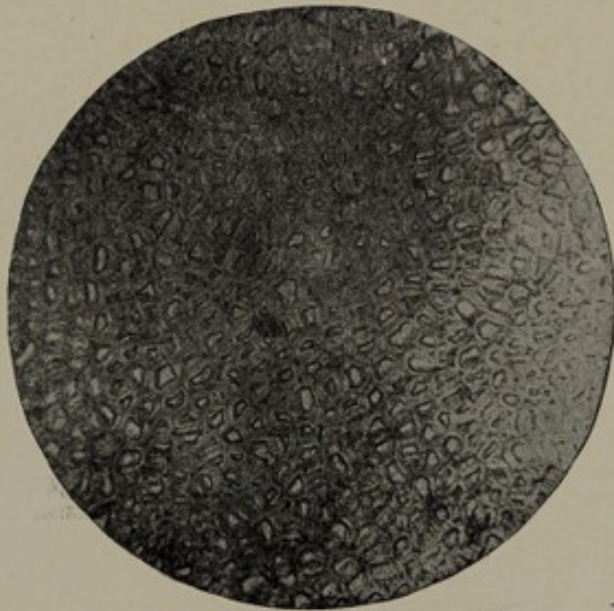
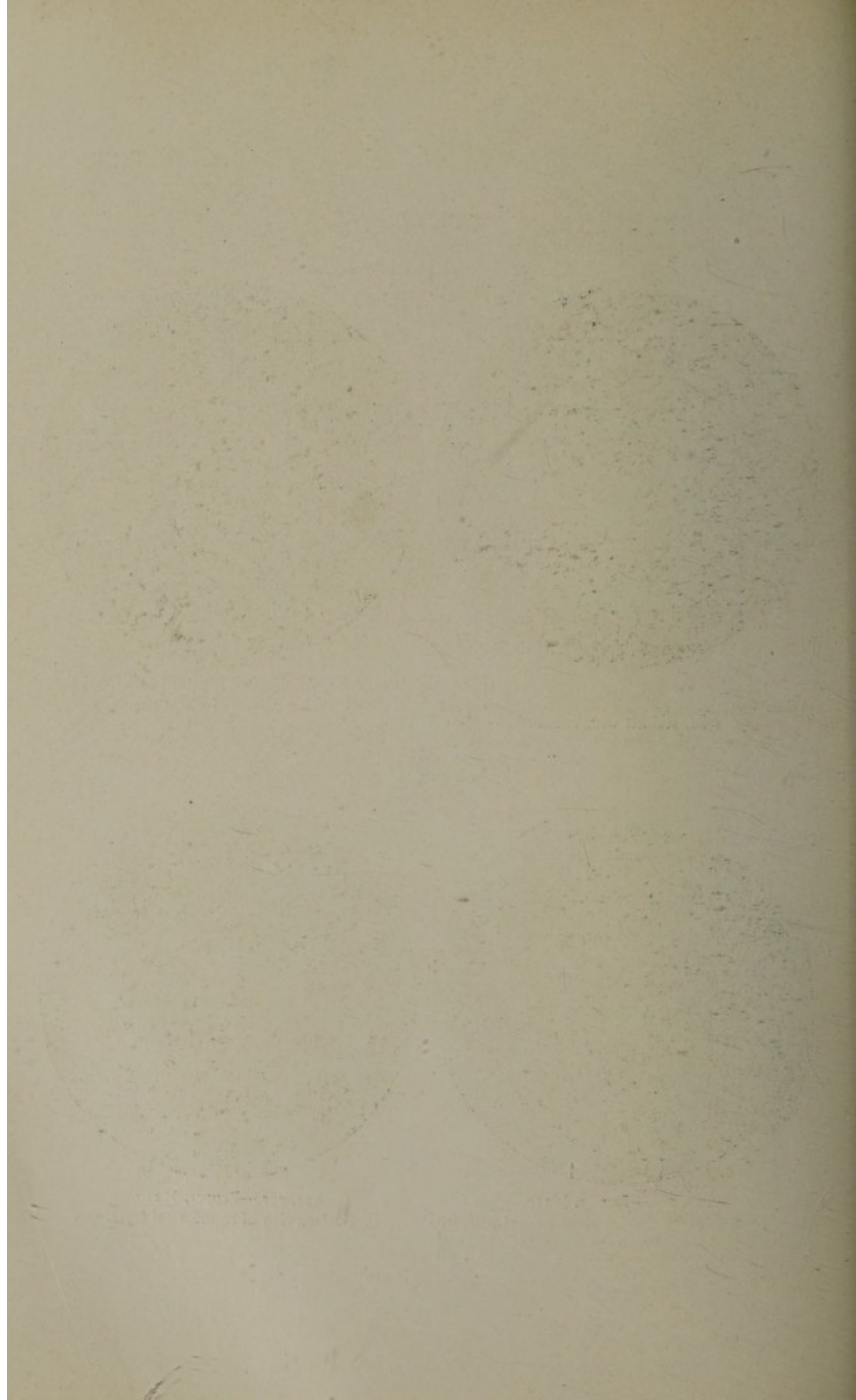


FIG. 252.—Pepper,  $\times 110$ .  
Surface view of hypodermal layer.







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FIG. 253.—Pepper,  $\times 110$ .

Transverse section through outer part of pericarp, showing epidermis, underlying stone cell layers, parenchyma, and seed coat.



FIG. 254.—Pepper,  $\times 110$ .

Surface section through stone cell layer

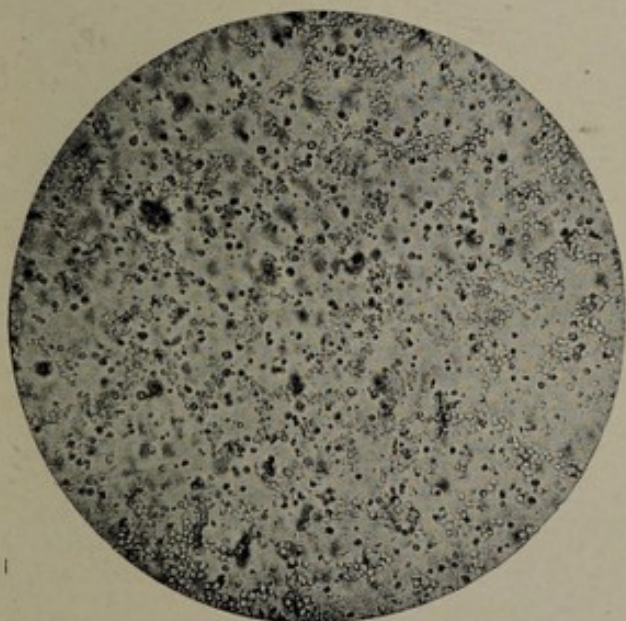


FIG. 255.—Pepper Starch,  $\times 220$ .  
Starch granules separated.

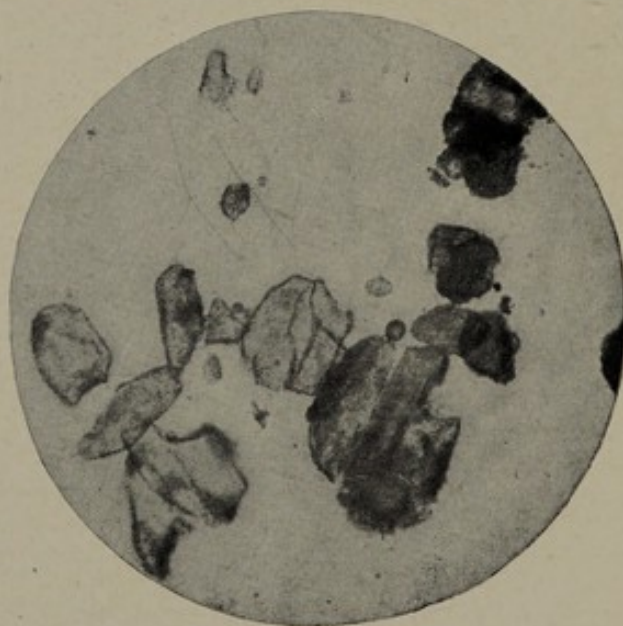
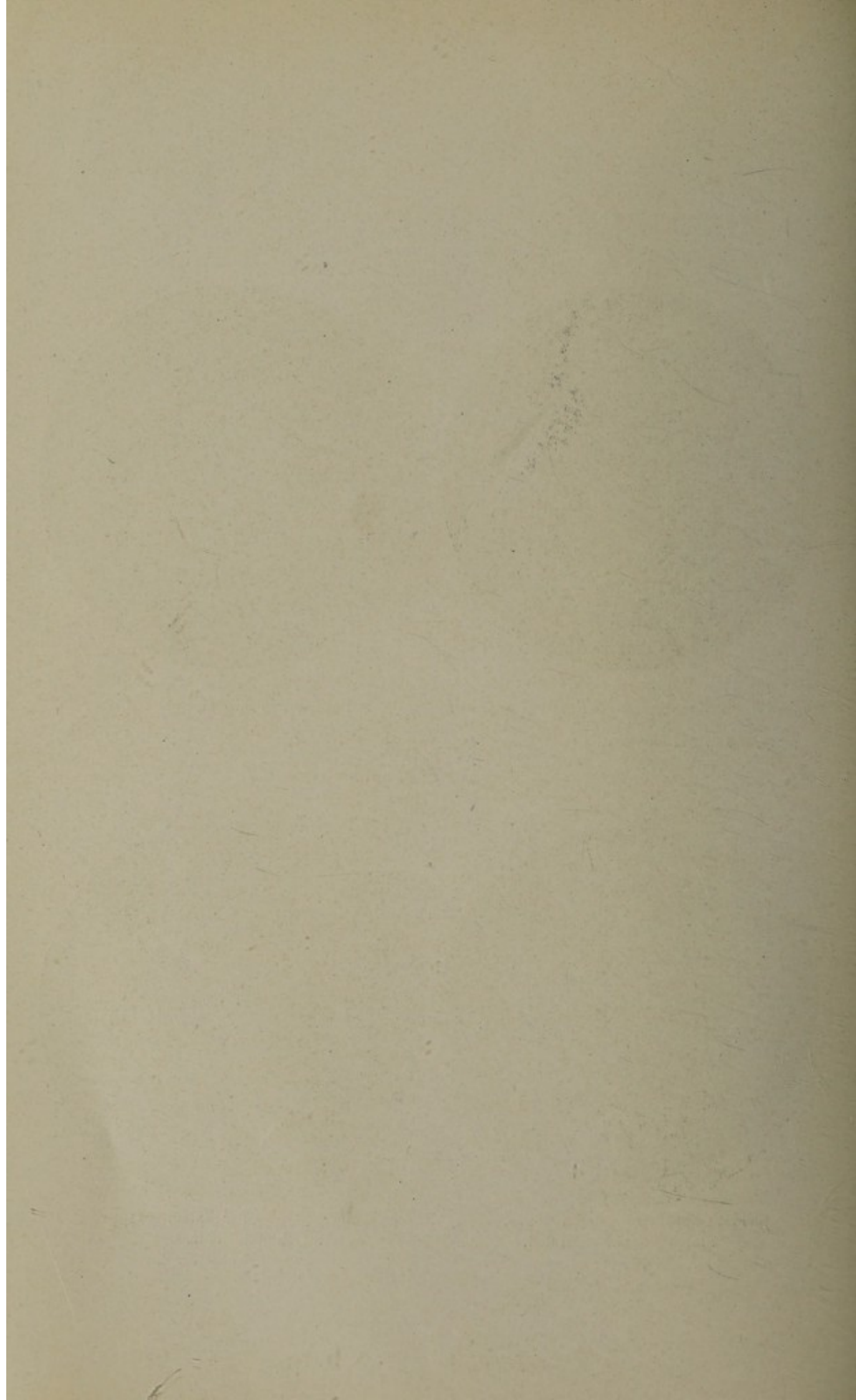


FIG. 256.—Pepper Starch,  $\times 110$ .  
Starch grains in masses.







SPICES.

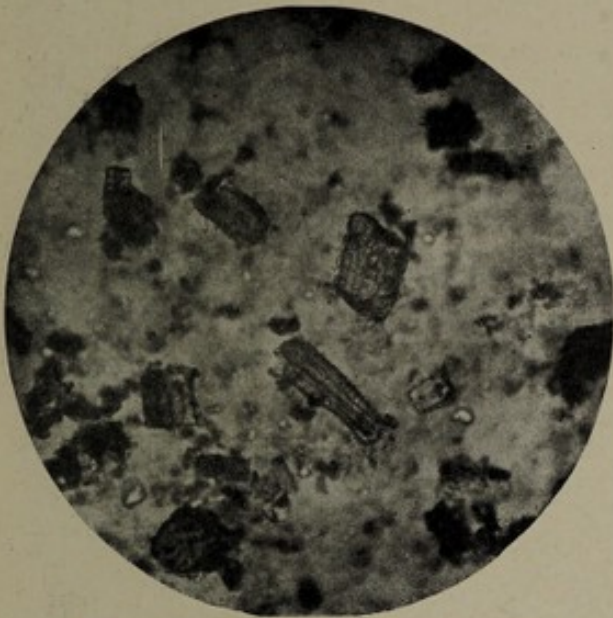


FIG. 257.—Ground Pepper Shells,  $\times 110$ .  
Mainly showing stone cells.

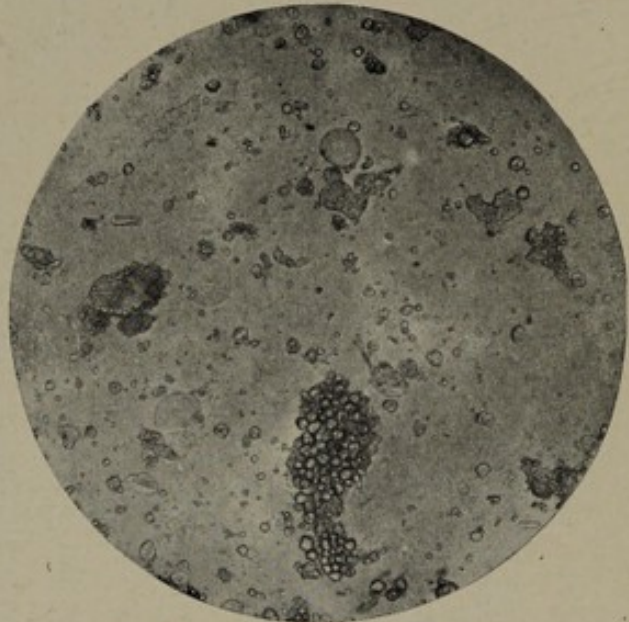


FIG. 258.—Adulterated Pepper,  $\times 130$ .  
Showing wheat and buckwheat starches.

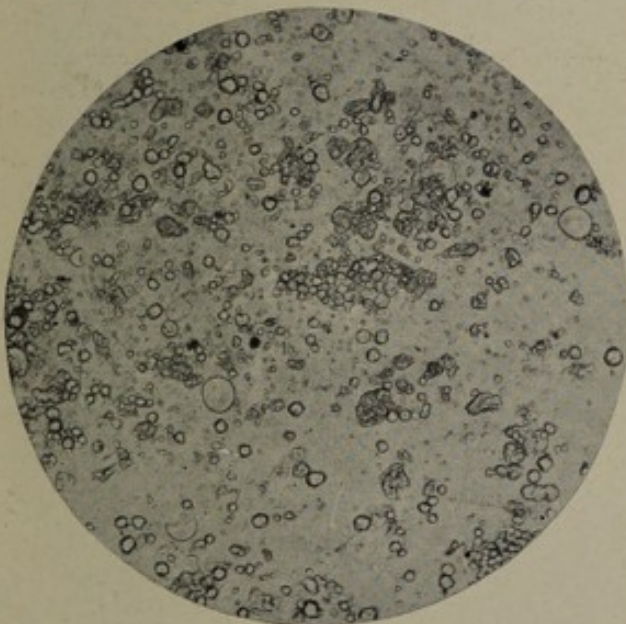


FIG. 259.—Adulterated Pepper,  $\times 130$ .  
Showing wheat, corn, and rice starches.

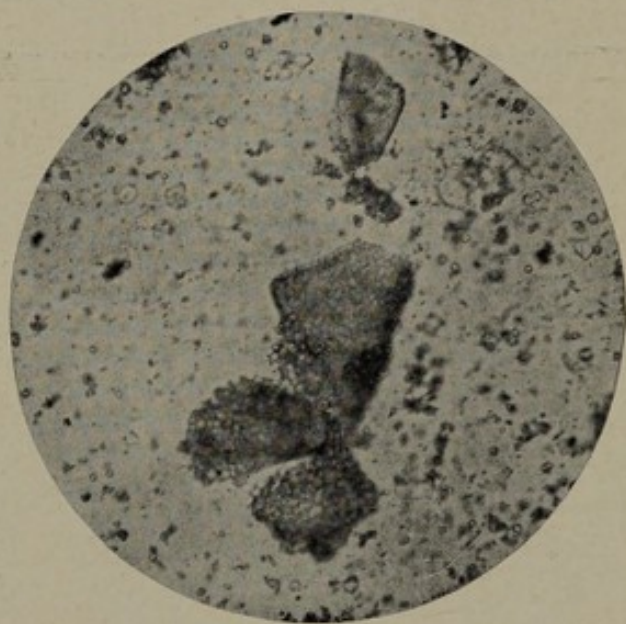
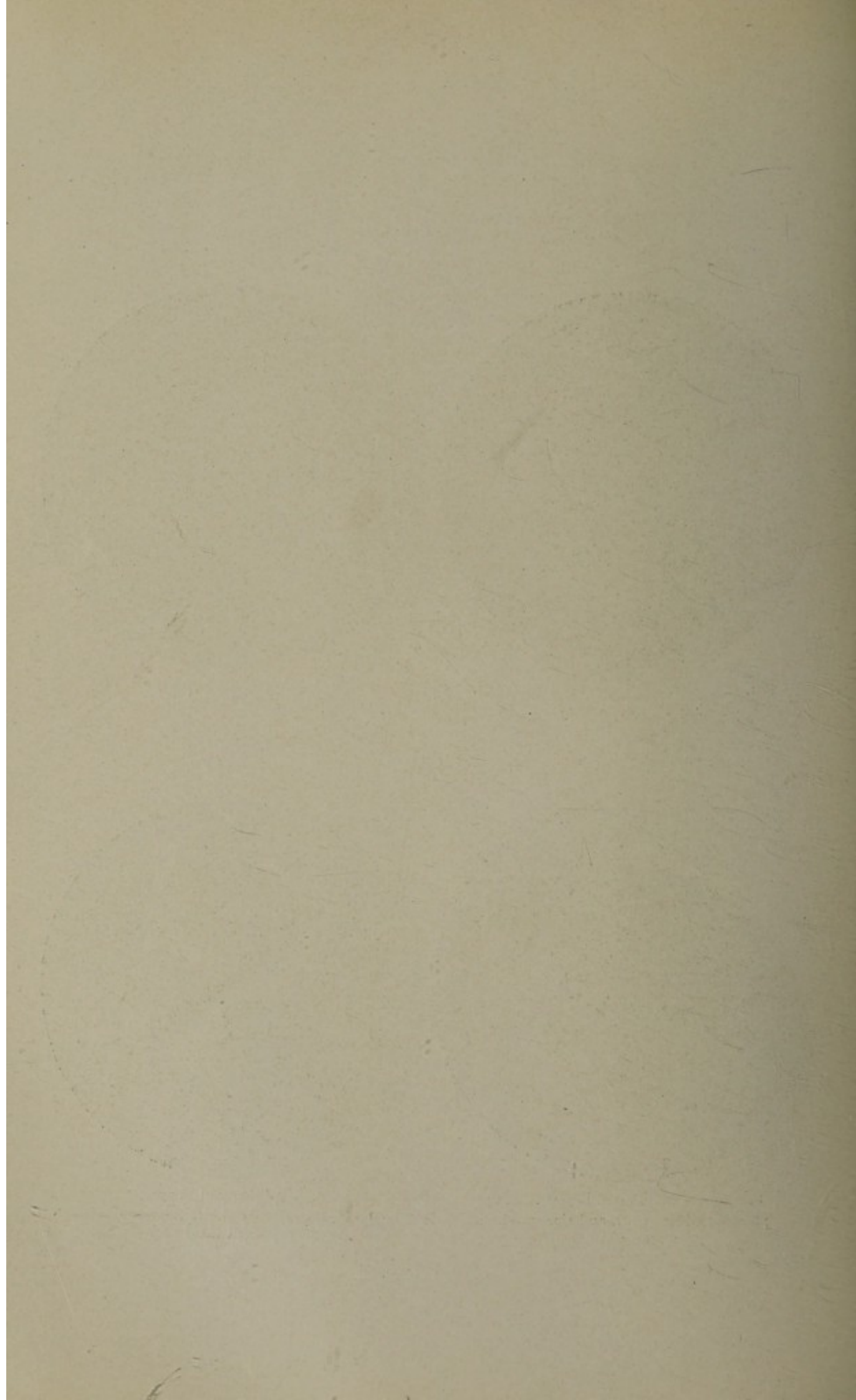


FIG. 260.—Adulterated Pepper,  $\times 130$ .  
The large, lower mass shows buckwheat starch,  
while the finer-grained mass near the top is of  
pepper.







SPICES. SPICE ADULTERANTS.



FIG. 261.—Adulterated Pepper,  $\times 110$ .  
The central mass shows the sclerenchyma cells of olive stones.

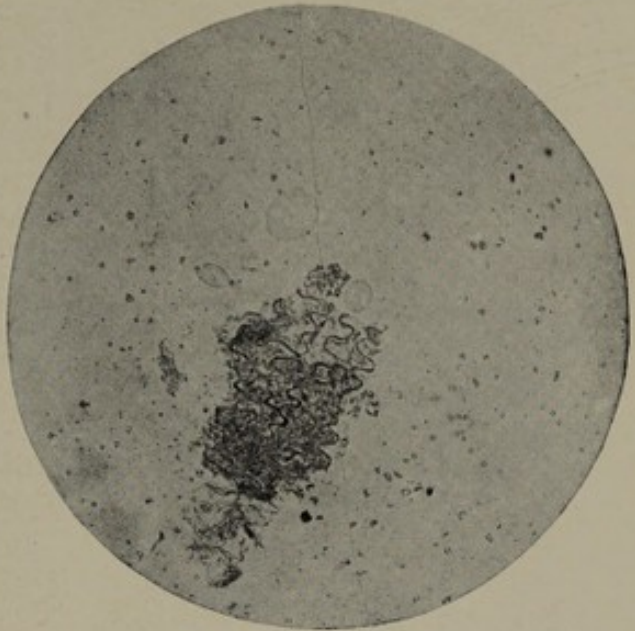


FIG. 262.—Adulterated Pepper,  $\times 130$ .  
Cayenne and wheat starch are the adulterants.



FIG. 263.—Powdered Olive Stones,  $\times 110$ .

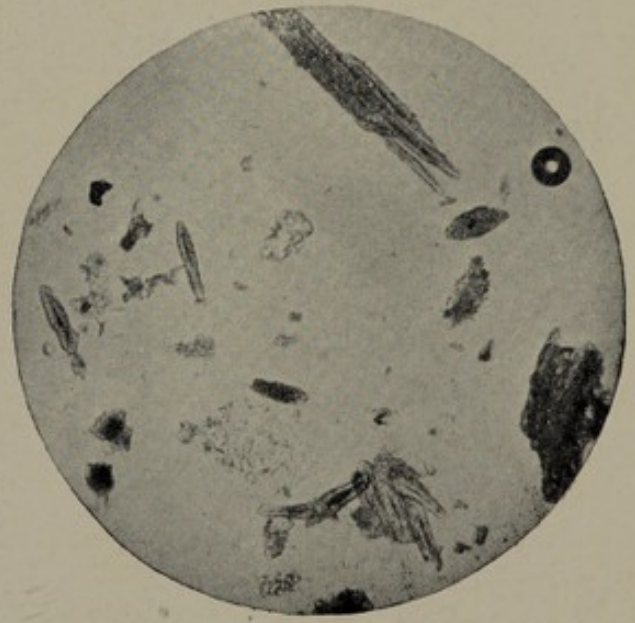
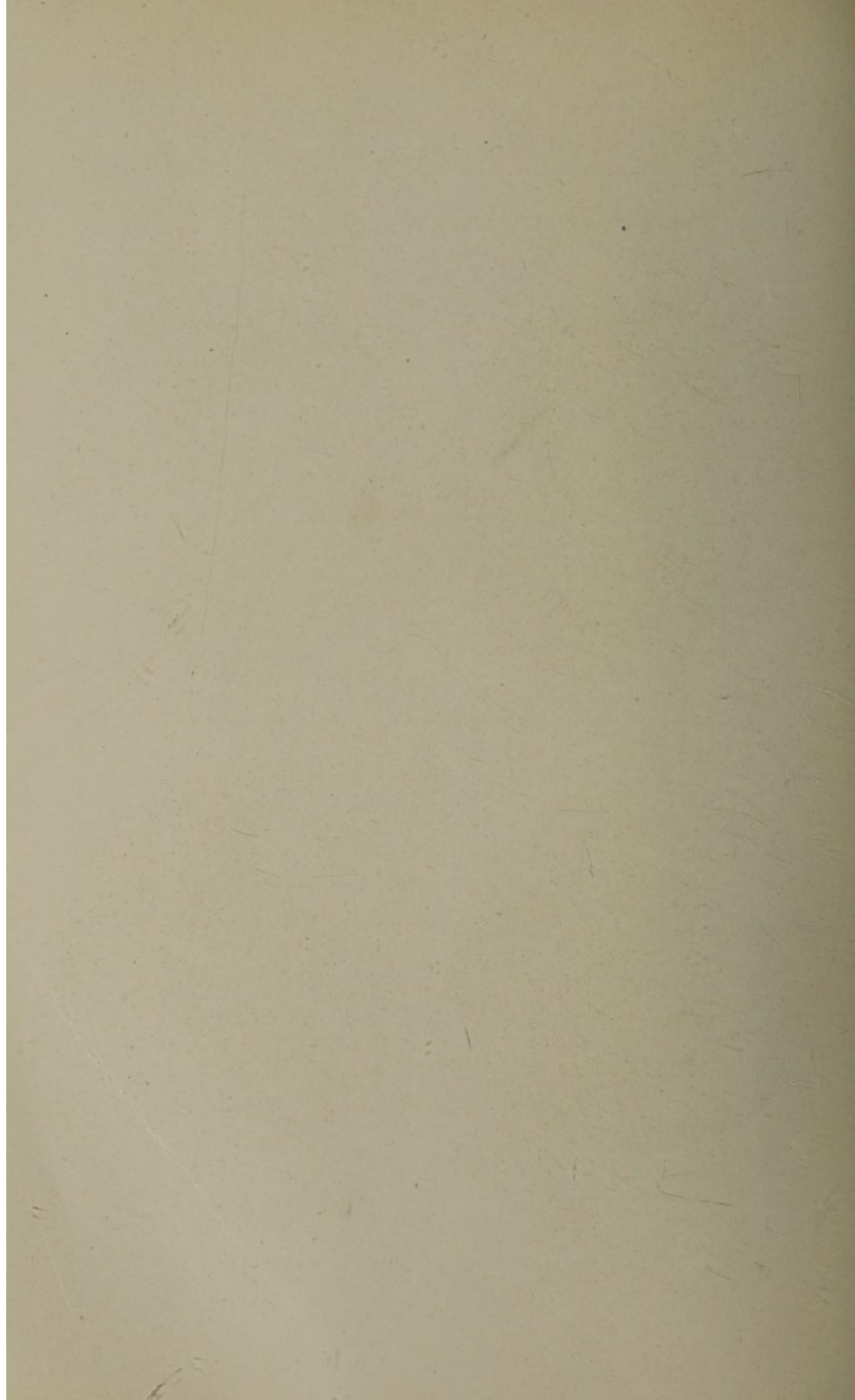


FIG. 264.—Powdered Cocoanut Shells,  $\times 110$







SPICE ADULTERANTS.



FIG. 265.—Powdered Elm Bark,  $\times 110$ .

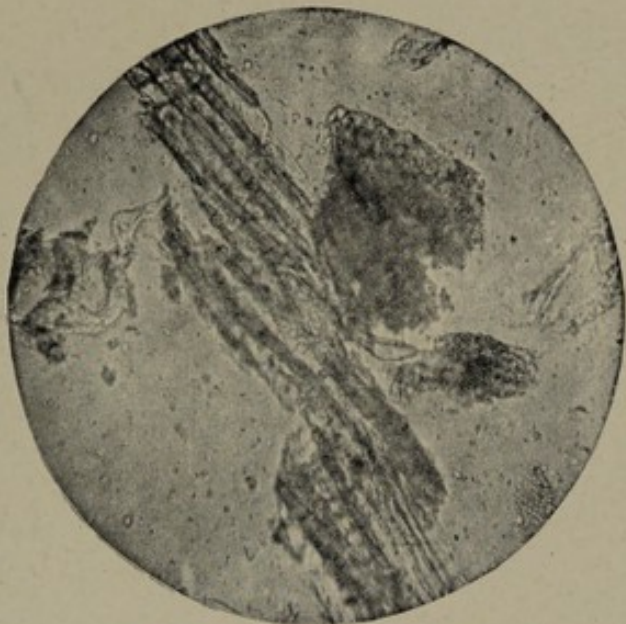


FIG. 266.—Pine Sawdust,  $\times 110$ .  
Finely ground.

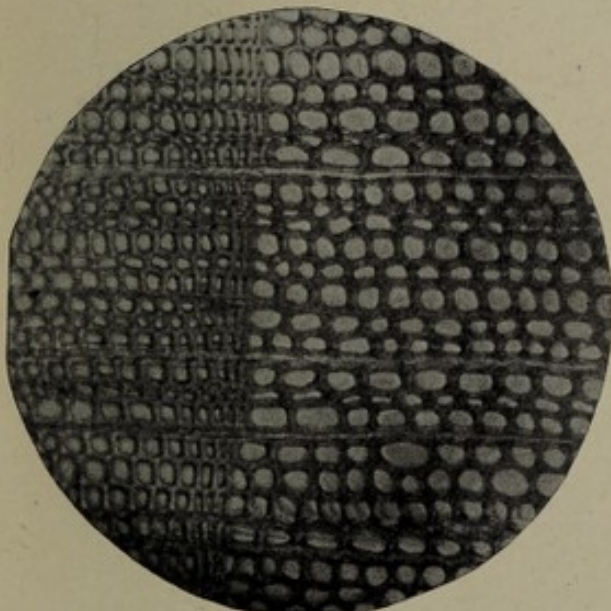
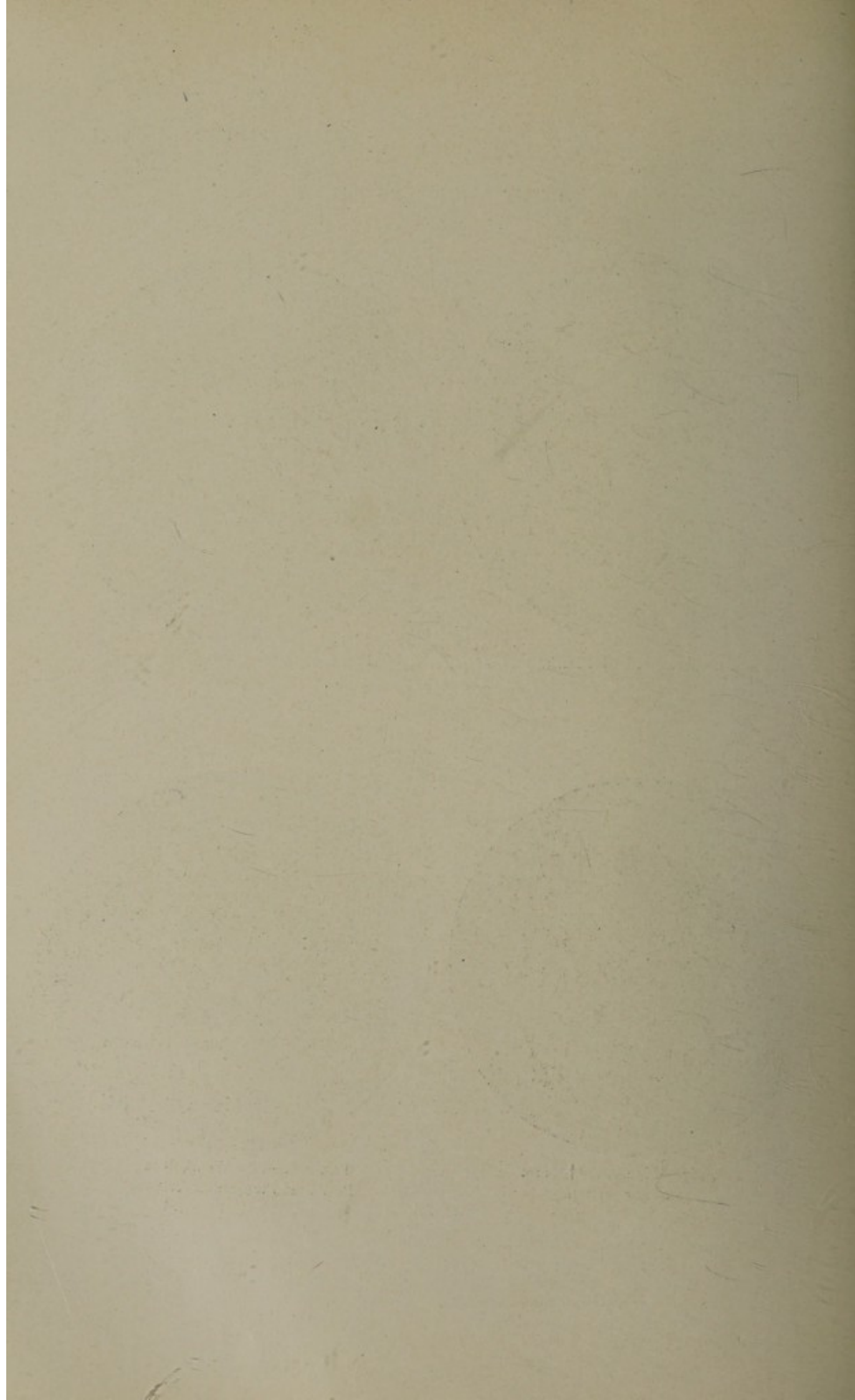


FIG. 267.—Pine Wood,  $\times 110$ .  
Transverse section.



FIG. 268.—Pine Wood,  $\times 110$ .  
Radial and tangential sections.







EDIBLE FATS.

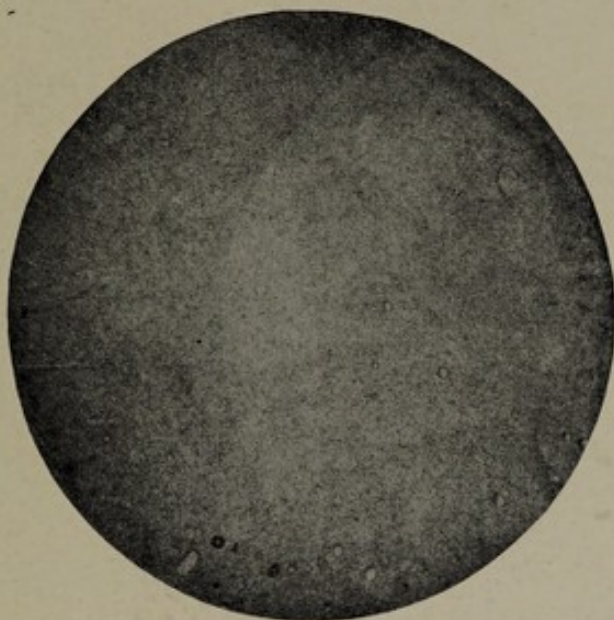


FIG. 269.—Pure Butter,  $\times 25$ .  
With polarized light and selenite plate.

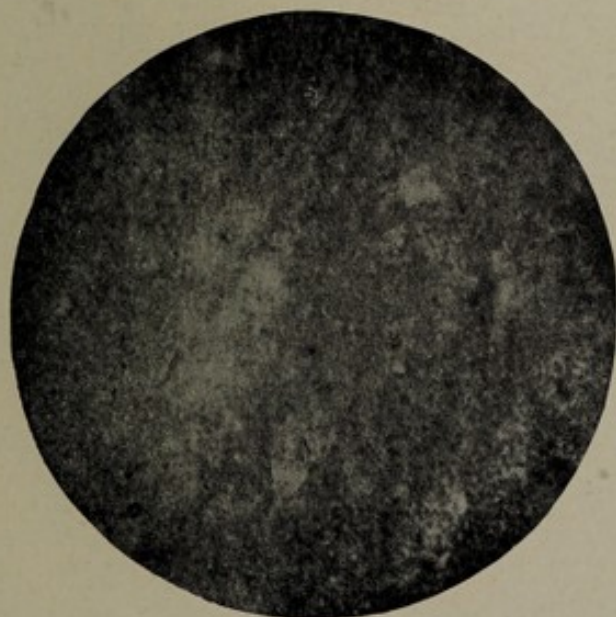


FIG. 270.—Process or Renovated Butter,  $\times 25$ .  
With polarized light and selenite plate.

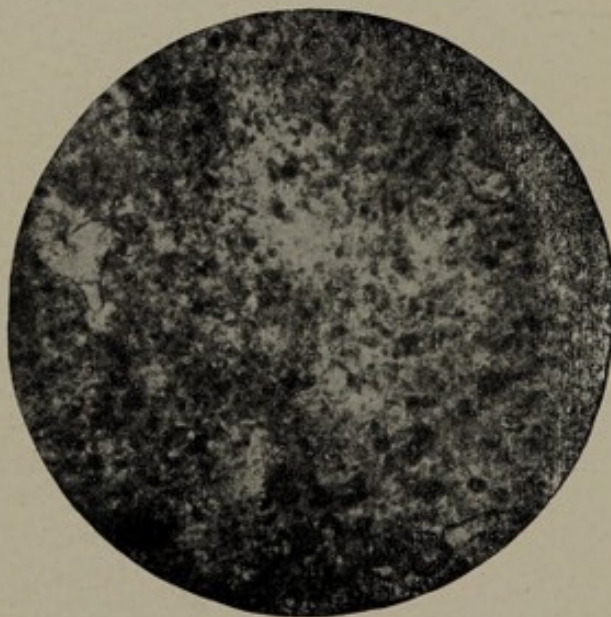
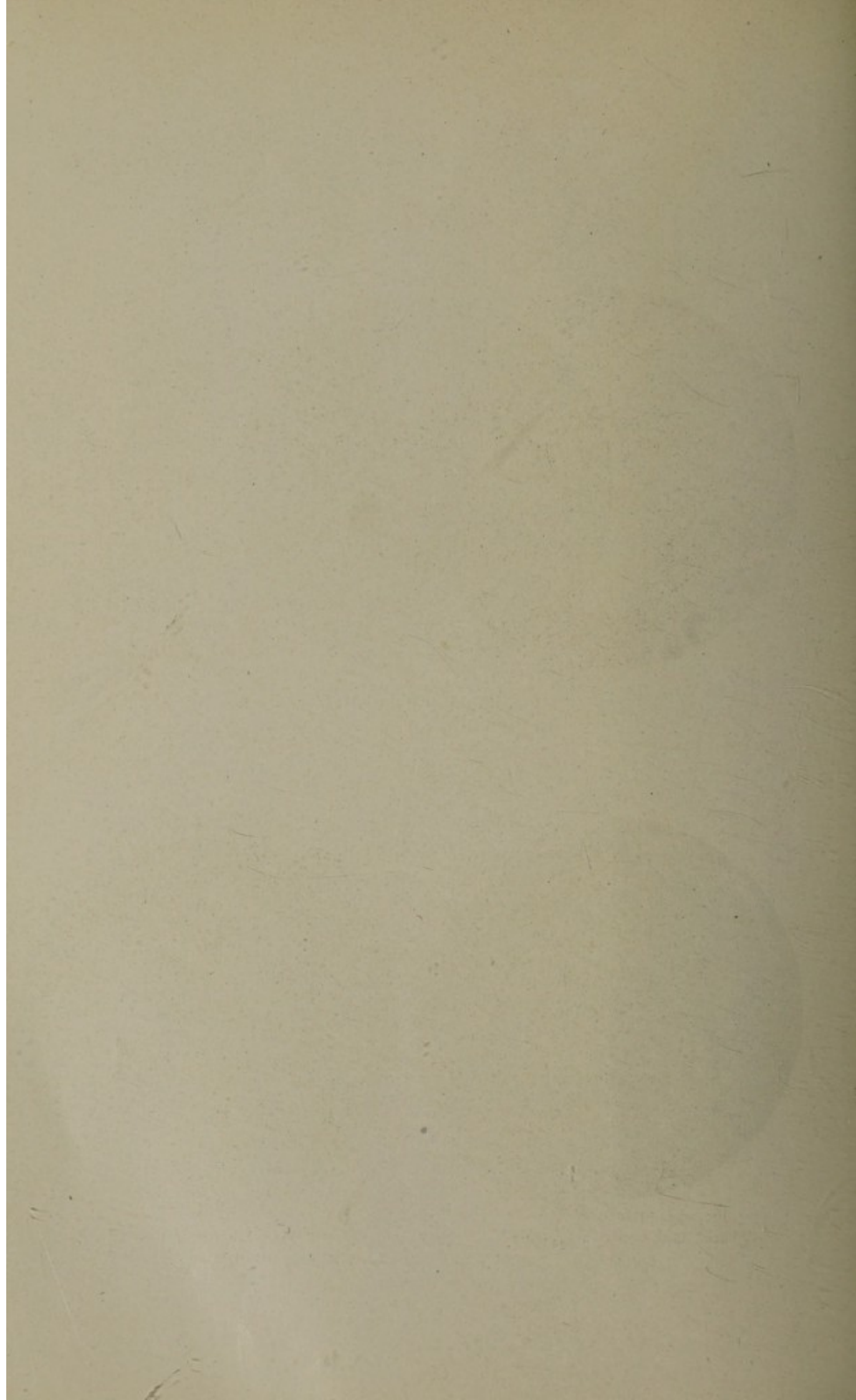


FIG. 271.—Oleomargarine,  $\times 25$ .  
With polarized light and selenite plate.







EDIBLE FATS.

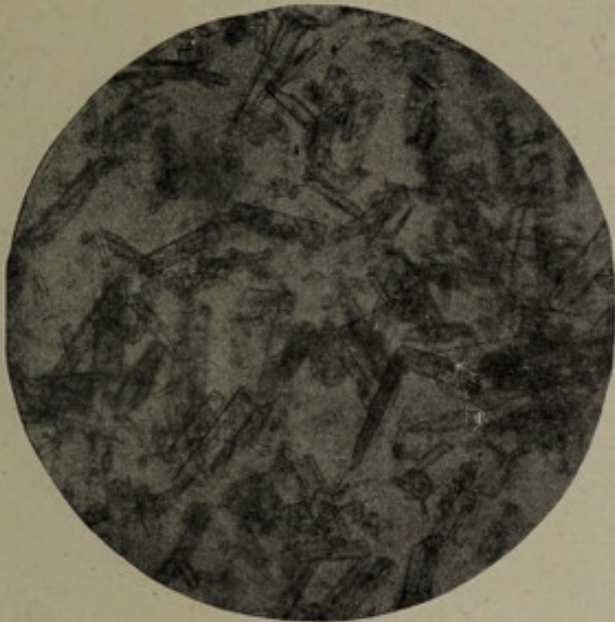


FIG. 272.—Lard Stearin,  $\times 110$ .  
Leaf lard, crystallized from ether.

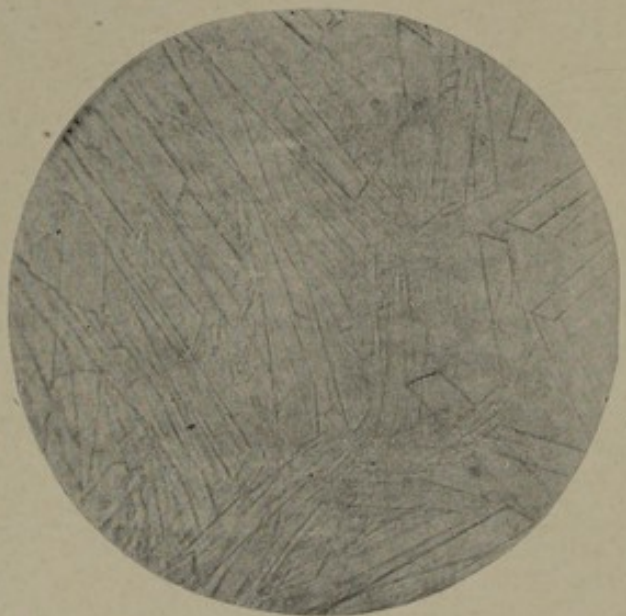


FIG. 273.—Lard Stearin,  $\times 220$ .  
Leaf lard, crystallized from ether.

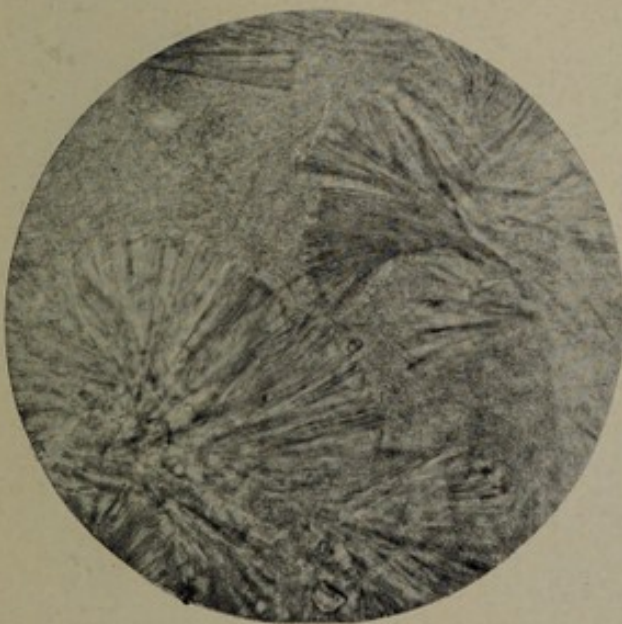
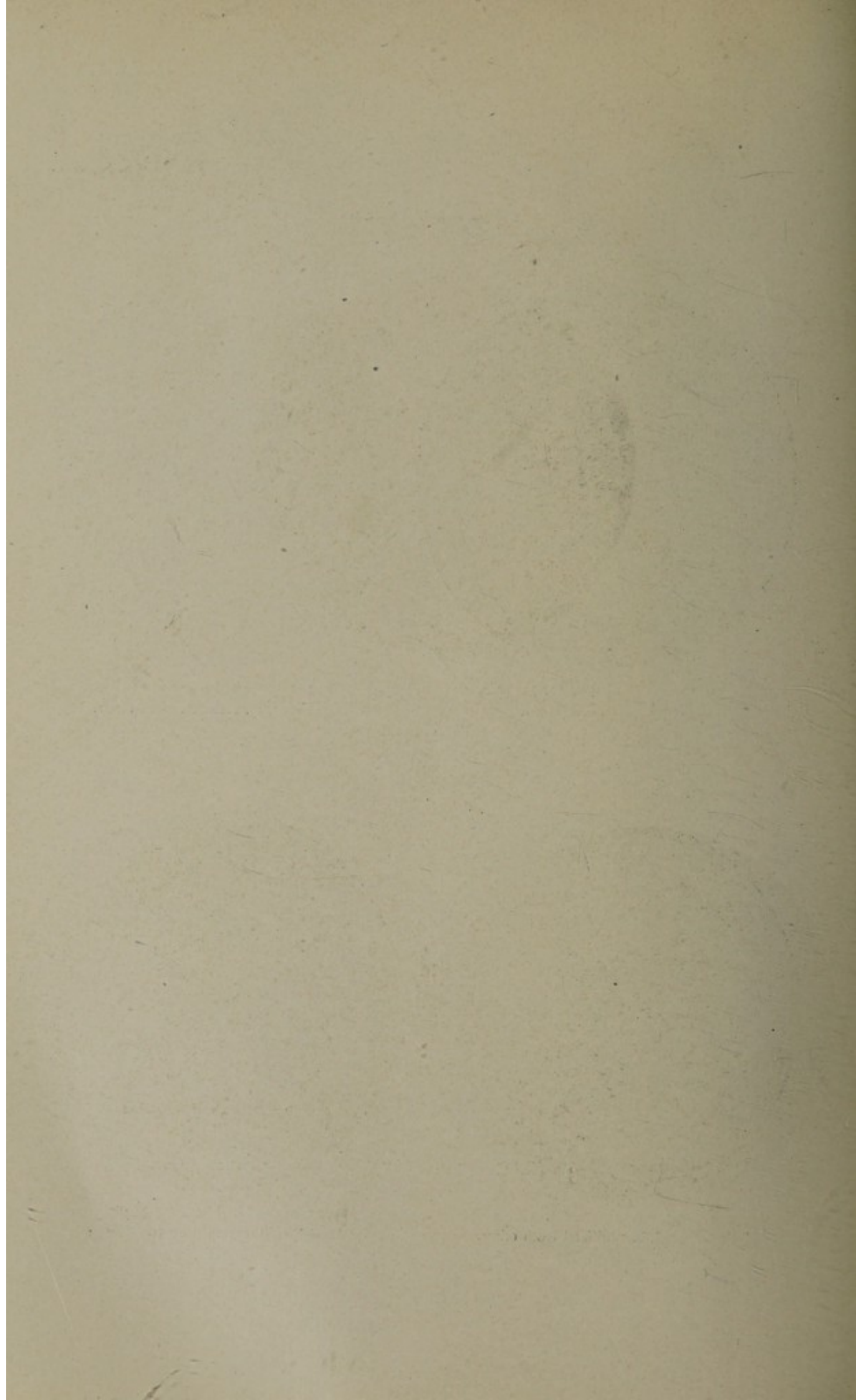


FIG. 274.—Lard Stearin,  $\times 220$ .  
"Back" lard, crystallized from ether.



FIG. 275.—Lard Stearin,  $\times 480$ .  
"Back" lard, crystallized from ether.







EDIBLE FATS.

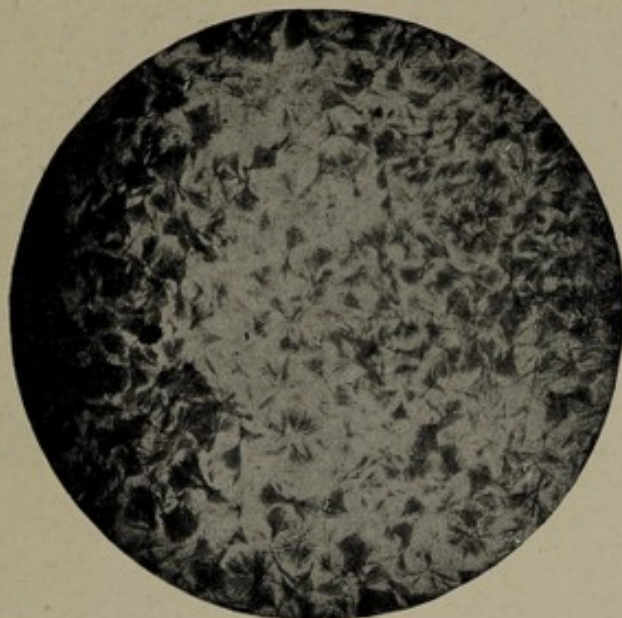


FIG. 276.—Beef Stearin,  $\times 35$ .  
Crystallized from ether.

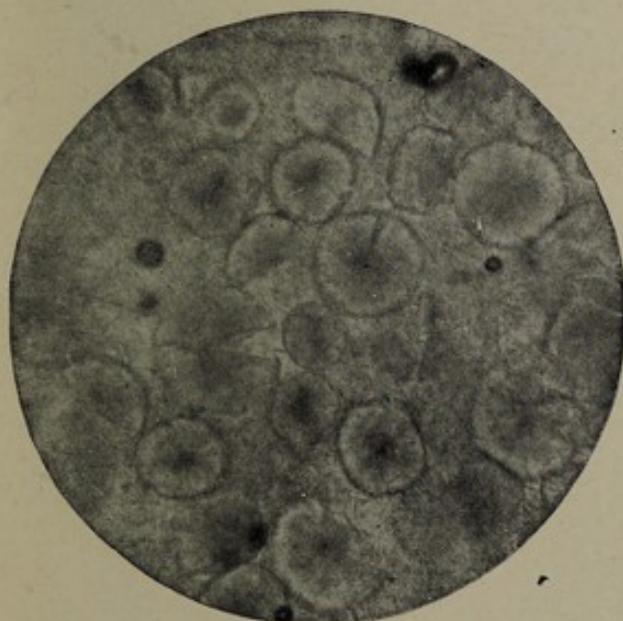


FIG. 277.—Beef Stearin,  $\times 110$ .  
Crystallized from ether.

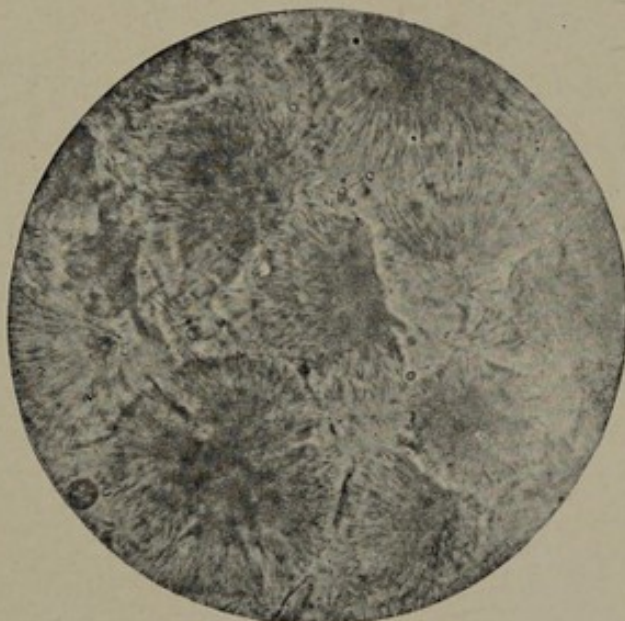
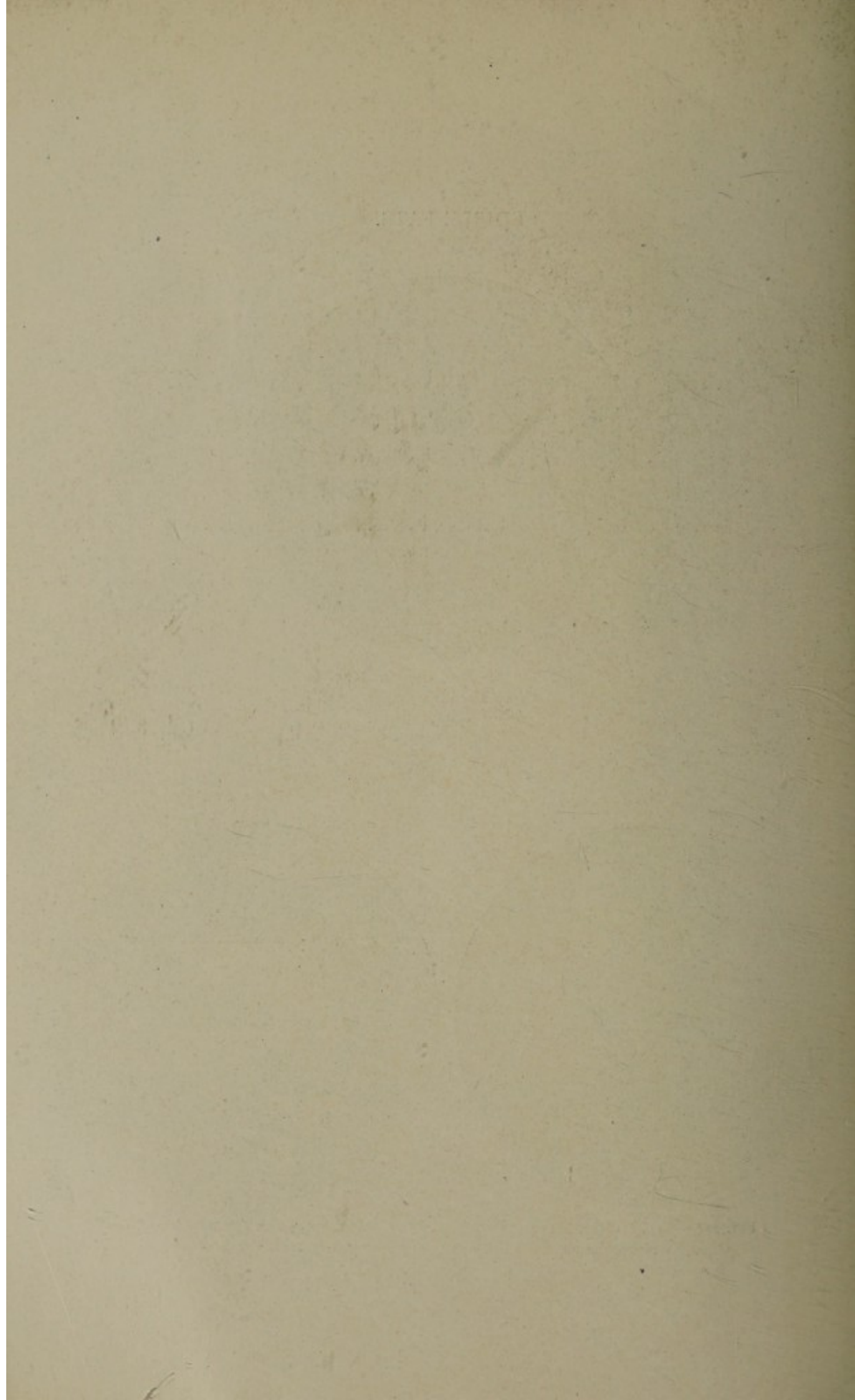


FIG. 278.—Beef Stearin,  $\times 220$ .  
Crystallized from ether.







MINIMUM PERCENTAGES OF ALCOHOL IN WINES CORRESPONDING TO HALPHEN RATIOS.

PLATE XLI.

