Aids to the analysis of food and drugs / by T.H. Pearmain and C.G. Moor.

AIDS TO THE ANALYSIS OF FOOD AND DRUGS

SECOND EDITION

PEARMAIN & MOOR

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BAILLIÈRE, TINDALL & COX.

AIDS

TO

THE ANALYSIS OF FOOD AND DRUGS.

BY

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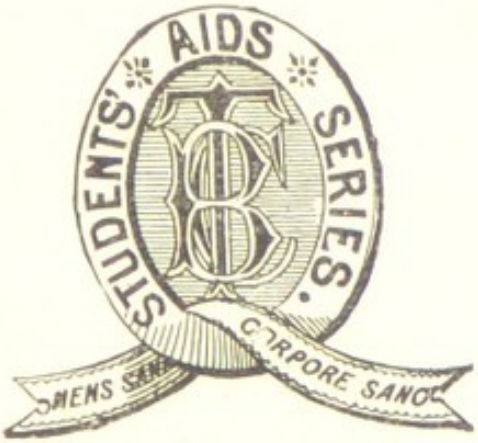
SECOND EDITION.



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PREFACE TO FIRST EDITION.

As no work of moderate size devoted to the analysis of foods and drugs has recently appeared, we venture to hope that this small book may prove of service to those engaged in the examination of foods and drugs. This work is not intended to be used as a cram-book for examinational purposes. We cannot emphasize too strongly the fact that food analysis is not to be taught in a few weeks, as is frequently attempted in the interest of public health students. A competent knowledge of the analysis of food and drugs is only to be attained by some years of active practical laboratory work.

T. H. PEARMAIN.
C. G. MOOR.

September 30, 1895.

PREFACE TO SECOND EDITION.

In preparing the second edition of this little work we have endeavoured to introduce as much recent information as possible on the articles dealt with, and have added a few others which seem to us to come within the scope of the work. Many of the articles have been entirely rewritten and enlarged, but the portion devoted to drugs has been curtailed for the reason given in the text. For further information on this subject we would refer to our larger work on 'The Analysis of Food and Drugs,' now in course of issue.

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4, Dane's Inn,
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April 20, 1899.

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AIDS

TO THE

ANALYSIS OF FOOD AND DRUGS.

MILK.

Milk may be defined as the fluid which is secreted by the lacteal glands of all female mammals for the nourishment of their young. In the case of cow's milk, more particularly, it should be understood to mean the whole milk of perfectly healthy, properly-fed cows.

It is the only article occurring in Nature which combines in the right proportions all the necessary elements requisite to secure the proper nutrition. For children it is rightly considered the ideal food; but it is too voluminous to serve for the sole diet of adults. Milk differs somewhat according to the animal it is obtained from, but its general characteristics are the same in all cases.

Genuine milk is a dense opalescent white liquid, often having a yellowish tinge, which has a specific gravity ranging between 1028·5 to 1035·0. It has a sweetish, bland taste. The colour is due essentially to suspended fatty globules, which occur under conditions which prevent spontaneous coalescence. Milk consists of water, fat, casein, albumin, milk-sugar, and mineral salts, the latter consisting chiefly of the chlorides of potassium and sodium, together with the phosphates of potassium, calcium, and magnesium, and traces of sulphates.

The fat is suspended in the aqueous portion of the milk in the form of minute globules, forming an almost perfect example of a natural emulsion. When examined under the microscope, milk will be seen to consist of a turbid fluid, containing fatty globules of different sizes, from 0·00005 to 0·0004 of an inch in diameter. Small particles of epithelium are frequently present.

When milk is allowed to stand for some time, a separation of the fat takes place. This is known as the rising of the cream, the larger fat globules of the milk naturally rising first. Absolutely fresh milk is amphoteric in reaction—that is to say, blue litmus is turned red, and red litmus blue ; but usually by the time milk reaches the consumers it is faintly acid, owing to some slight fermentative changes taking place, resulting in the production of lactic acid.

When allowed to stand still longer, milk undergoes fermentative changes, due to micro-organisms, usually the *Bacillus acidi lactici.* This decomposes the milk-sugar, resulting in the production of lactic acid, which eventually causes the milk to become sour. This change is largely dependent upon the time the milk has been allowed to stand, but more particularly upon the temperature ; milk, of course, becomes sour more quickly in the summer than in the winter. When the fermentative change has resulted in the production of about 0·4 per cent. of lactic acid, the milk can be distinctly recognised by the taste to be sour. Finally, when the acidity reaches 0·6 per cent., the milk curdles, whereby it spontaneously separates into a solid, known as 'curd,' which consists of the fatty and proteid constituents of the milk, and a clear liquid known as 'whey,' which consists essentially of a solution of milk-sugar and mineral salts. This change is also brought about artificially, as in the manufacture of cheese, by the addition of rennet. The further changes which take place in the composition of the milk after curdling depend upon the nature of the bacteria which have gained access, various organisms giving rise to different fermentations.

As would be expected from its origin, milk is subject to great variations in its composition, on account of the age, breed, food, changes in health, and the general conditions of the animal yielding it. The average composition of genuine cow's milk is as follows :

Water	87·6 per cent.
Fat	3·6 „
Milk-sugar	4·8 „
Proteids	3·3 „
Mineral matter	0·73 „

Milk-fat.—The fat of milk is very complex in its chemical constitution. This will, however, be fully considered under butter-fat, with which it is of course identical.

Milk Proteids.—The proteids of milk consist largely of casein, together with about 0·4 to 0·5 per cent. of albumin, of the variety known as lactalbumin, with traces of globulin. Much work is required to be done on these bodies before their exact nature and their relation to one another can be properly determined. It is these bodies that give to milk its property of curdling when rennet or an acid is added. The albumin is coagulated by boiling. The form in which casein exists in milk is known as caseinogen. It is not known with any degree of certainty in what form the casein exists, but it is possible that it is in combination with the calcium and other phosphates. Acid destroys the combination, precipitating the casein in an insoluble condition; but heat alone will not produce this change.

Very small proportions of other nitrogenous bodies are present, including those to which are due the characteristic flavour and smell of milk. An unorganized ferment or enzyme, which converts starch, is also present in milk.

By boiling, the taste and smell of milk are substantially altered, but the chemical changes which take place are at present obscure.

Milk-sugar, or Lactose ($C_{12}H_{22}O_{11}+H_2O$).—This variety of sugar, which is an isomer of cane-sugar, is peculiar to the milk of the Mammalia. It is the most constant constituent of milk, from 5 to 6 per cent. being present in human milk, and 4 to 5 per cent. in the milk of most of the herbivora. As milk-sugar is the most constant constituent of milk, the variations in the specific gravity of the whey or serum of genuine milk falls within much narrower limits than in the case of the specific gravity of milk itself. Lactose has only a faint degree of sweetness, and is gritty when chewed. It is much less soluble than cane-sugar, being soluble in six parts of cold and three of boiling water. Milk-sugar is convertible by the action of dilute acids into 'galactose' and 'lactoglucose'; these may be reunited to form lactose. Lactose is dextro-rotatory to a ray of polarized light, and resembles dextrose in its reducing action on Fehling's solution.

Mineral Matter.—The mineral matter or ash, obtained by the careful ignition of the solids of milk, consists of the chlorides of potassium and sodium, the phosphates of calcium, magnesium, and potassium, with traces of sulphates. The ash of milk varies from 0·65 to 0·85 per cent.

According to König, the average composition of the ash of milk is as follows :

Potash as K_2O	24·0 per cent.
Soda as Na_2O	8·2 ,,
Lime as CaO	22·4 ,,
Magnesia as MgO	2·6 ,,
Phosphoric acid as P_2O_5 ...	26·3 ,,
Chlorine	13·9 ,,
Sulphuric acid as SO_3 ...	2·5 ,,
Iron	traces.

Human Milk.—The composition of human milk, as in the case of that of cows, is largely dependent upon the health and condition of life of the individual yielding it. The specific gravity of human milk varies from 1029 to 1035. Abnormal variations occur between the limits of 1017 and 1036. The amount of mineral matter does not vary so much as in the case of cow's milk. The proportion of sugar is very nearly constant, but the fat and proteids vary greatly in quantity, depending very much upon the stage of the nursing.

The following table shows the variations in the composition of different kinds of milk as compared with the average composition of human and cow's milk :

Kind of Milk.	Water.	Total Solids.	Fat.	Sugar.	Proteids.	Ash.	Specific Gravity.	Analyst.
Human	87·3	12·3	3·4	6·4	1·7	0·2	1031·3	
Cow ...	87·6	12·4	3·6	4·8	3·3	0·7	—	
Goat ...	86·7	13·3	3·8	4·5	4·1	0·9	—	Richmond.
Ewe ...	75·2	24·8	11·3	3·6	8·8	1·1	1039·3	J. Bell.
,, ...	81·3	18·7	6·8	4·8	6·3	0·8	—	Vieth.
Ass ...	89·8	10·2	1·2	6·8	1·7	4·5	—	Richmond.
,, ...	89·6	10·4	1·6	6·1	2·2	0·5	—	Vieth.
Sow ...	89·6	10·4	4·8	3·4	1·3	0·9	—	,,
Gamoose	84·2	15·8	5·5	5·4	3·8	1·0	1035·4	Richmond.

From the table, it will be seen that, taking cow's milk as a standard for comparison, human milk shows an increased proportion of sugar, but all the other constituents are materially less. The milk of sheep is notable for its enormous percentage of fat; it is not employed to any extent in this country, but abroad it is largely used in the manufacture of Roquefort cheese. The milk of asses is much lower in fat and solids-not-fat than cow's milk; the casein is not readily curdled, on which account it is sometimes used as a substitute for human milk.

As already stated, the health, age, food, etc., of the animal yielding the milk have a great influence upon its composition and quality. The following are the chief circumstances on which these variations in the composition of cow's milk depend, which of course apply equally to all other animals yielding milk:

1. *The Breed of the Cow.*—Some breeds yield quantity, others quality. Alderney and Jersey cows yield the most fat; Shorthorns give the most casein and sugar. The average capacity of a cow's udder is about 5 pints, and the average annual yield of milk about 600 gallons.

2. *The Time and Stage of Milking.*—Cows are usually milked twice a day, the morning milk as a rule being the larger in quantity and the poorer in quality. The milk which is first drawn, known as the 'fore milk,' contains very much less fat than that last drawn, known as the 'strippings.' This is due to a partial creaming taking place in the udders. Dishonest dealers have often taken advantage of this fact in adulteration cases, by having the cows partially milked in the presence of ignorant witnesses, so that the resulting milk consisted largely of the 'fore milk.'

3. *The Age of the Cow.*—Young cows give less milk, while cows from four to seven years old give the richest milk, and less milk is given with the first calf. Cows usually become milkers in the third year. They give the largest yield, according to Fleischmann, after the fifth until the seventh calf. After the fourteenth calf they yield, as a rule, no more milk.

4. *The Time of Year.*—The poorest milk is yielded during the spring and early summer months; the richest, during the autumn and early winter.

5. *The Mental and Physical Conditions under which the Animal is kept.*—If the cows are worried or driven about, the quality and yield of the milk are reduced. If they are kept warm and well fed, the quantity and quality of the milk are naturally increased.

6. *The Food.*—The natural food, fresh grass, is the best ; failing this, hay is the next preferable food. The greater the proportion of the nitrogen in the food, the greater is the yield of the milk, the proportion of fat being especially high. Beets, carrots, and swedes increase the proportion of milk-sugar. Feeding cows with brewers' grains depreciates the quality by lowering the total solids of the milk. This objectionable practice has been made illegal in the State of Wisconsin, U.S.A.

7. *The Health of the Animal.*—As would be expected, the health of the animal has a profound effect on both the quantity and composition of the milk. In the case of diseased udders, the milk is likely to be contaminated with blood, pus cells, and other morbid products. Milk itself is liable to a number of diseases, particularly those of a bacillary nature.

The Variations in the Composition of Cow's Milk.—For our knowledge of the composition of milk, we are indebted in this country mainly to the elaborate and prolonged researches of Adams, Bell, Hehner, Richmond, Stokes, Vieth, and others. As the result of many thousands of analyses, it is found that genuine cow's milk does not normally differ materially from the analysis given as typical on p. 8.

It is only on the rarest occasion that milk contains less than 3·0 per cent. of fat, while the average is 4 per cent. The amount of solids-not-fat may fall from such causes as ill-health, old age, length of time after calving, and injudicious feeding. Foods such as brewers' grains, maize, and certain roots will increase the quantity of milk given, at the expense of its quality, unless the diet is supplemented in a suitable manner.

The evidence that genuine milk contains upwards of 3·5 per cent. of fat, and 8·5 per cent. of solids-not-fat, is overwhelming. It is not surprising that the adulteration of milk is so universal, seeing that it is possible to add about 20 per cent. of water, or 30 per cent. of skimmed milk, to milk of average quality, without the resulting

mixture falling below the present standard. Some analysts make it a practice to adopt a higher standard—namely, 3 per cent. of fat—and report as adulterated all milks which contain less than 3 per cent. of fat. This course is highly to be commended, and it is to be hoped that a legal standard will shortly be fixed.

The following figures were published by Dr. James Bell, of the Inland Revenue Laboratory, Somerset House, in 1881 (Bell, 'Chemistry of Foods,' vol. ii.). The analyses of the milk of 236 individual cows are given; all these are said to have been thoroughly milked in the presence of a responsible person, and the genuineness of the sample was in each case authenticated. The results were as follows :

	Highest.	Lowest.	Average.
Specific gravity at 15·5 C.	1036·9	1026·7	1032·1
Total solids	16·3%	10·3%	12·8%
Fat	6·9	1·9	3·8
Solids-not-fat ...	11·3	8·0	9·0
Ash	0·87%	0·62%	0·71%
Volume of cream after 24 hours	26%	2%	10%

In addition to the above figures, Dr. Bell also published the analytical results of twenty-four dairy samples (mixed milk from herds of cows). These results were as follows :

	Highest.	Lowest.	Average.
Total solids	14·7%	11·8%	13·2%
Fat	5·1	3·2	4·1
Solids-not-fat ...	9·9	8·5	9·0
Ash	0·78%	0·63%	0·72%

At the suggestion of the Local Government Board, a further systematic investigation was made in 1892 in the Inland Revenue Laboratory, Somerset House, under the

direction of Dr. James Bell, to inquire into the composition of genuine milk. The investigation embraced the milk of 273 individual cows, and the mixed milk of fifty-five dairies. In each case the cow was thoroughly milked in the presence of one of the assistant analysts of the department, so that the genuineness of the sample was substantiated. The results contained in the report show a sensible improvement in the quality of the milk as regards the proportion of fat since the investigation which was made ten years previously. The samples included the milk of country farms as well as of town dairies, and were collected over large areas. The breed of the cows, as well as the kind of food, was duly noted. The report, which is dated August, 1893, has been very ably criticised by J. F. Liverseege in a paper in the *Analyst* for January, 1895.

The average composition of milk, as shown by the above report, is as follows :

Averages.	Specific Gravity.	Total Solids.	Fat.	Solids-not-Fat.
Milk of 273 single cows	1032·3	12·9%	4·0%	8·9%
Milk of 55 dairies	1031·9	12·9	4·0	8·9

Dr. Vieth, in a series of papers contributed to the *Analyst* between 1880 and 1891, gives the analyses of no less than 120,540 samples of milk. A summary of these results, extending over a period of eleven years, together with curves showing the monthly variations, was published in the *Analyst* for May, 1892.

The average composition of the above 120,540 samples was as follows :

Fat	4·1	per cent.
Solids-not-fat	8·8	,,
Total solids	12·9	,,

The above valuable series of analyses has been continued by H. Droop Richmond, who has published yearly papers in the *Analyst* from 1892, showing the yearly average composition.

The following figures show the greatest variation

between the monthly averages of the above two series of analyses from 1881 to 1897 inclusive:

Total solids...	...	12·3 to 13·7 per cent.
Fat	...	3·5 „ 4·6 „
Solids-not-fat	...	8·6 „ 9·1 „

These samples of milk were taken from healthy cows of all kinds, fed in every kind of manner, during all weathers and times of the year.

The absolute reliability of these figures, representing 226,009 samples of milk, made in the laboratory of the Aylesbury Dairy Company by Dr. Vieth and his successor, H. Droop Richmond, as truly representing the composition of genuine cow's milk, is fully admitted by all analysts of experience, and the information obtainable from them is amply sufficient to justify the immediate formulation of a legal standard. The recent Select Committee upon Food Products Adulteration have, however, made no recommendation in this respect, but state that the fixing of a standard would be 'one of the most important duties that would engage the attention of the proposed Court of Reference.'

Milk Standards.—There is a very urgent need for a proper legal standard of purity for milk in this country, and it is to be hoped that, in the interests of purchasers, honest tradesmen and public analysts generally, one will soon be adopted. The standard of purity for milk, generally understood to be adopted by the chiefs of the Inland Revenue Department Laboratory, who are the referees under the Sale of Food and Drugs Acts, is as follows:

Total solids	...	...	11·25 per cent.
Fat	...	...	2·75 „
Solids-not-fat	...	...	8·5 „

This standard is founded upon the poorest quality milks that have been known to be yielded by cows. There is a certain amount of evidence to show that milk as poor as this has been yielded by separate cows, who were possibly in ill health, or otherwise abnormal owing to improper feeding or other causes; but it is grossly unfair to judge the quality of milk by the standard of these inferior samples. Milk from single cows is rarely if ever met with in commerce; almost all the milk which finds its

way into the market is the mixed product of two or more animals, which should be above the meagre standard in question. It is only fair for a purchaser of milk to expect this article to be of at least, or not materially below, the average quality. The insufficiency of this standard will be obvious when it is compared with other standards, for it will be seen to be lower than any other with which we are acquainted.

The following table shows some of the standards adopted for milk in various places :

State, Town, Authority, etc.	Percentage by Weight of Solids.		
	Fat.	Non-fatty.	Total Solids.
Society of Public Analysts ...	3·0	8·5	11·5
Inland Revenue Department ...	2·75	8·5	11·25
Central and Associated Chamber of Commerce (proposed standard) ...	3·0	9·0	12·0
Canada (proposed standard) ...	3·5	8·5	12·0
Paris	4·0	9·0	13·0
Berne	3·5	9·0	12·5
Treasury Department (U.S.A.) ...	3·5	9·5	13·0
Pennsylvania	3·0	9·5	12·5
Philadelphia	3·5	8·5	12·0
New York	3·0	9·0	12·0

Skimmed and Separated Milk.—*Bonâ-fide* skimmed milk, in which the cream had been partially skimmed by hand, has ceased to be sold as such, and has now practically disappeared from commerce. Next to actual watering, partial skimming of whole milk, or, what amounts to the same thing, the addition of separated milk to whole milk, constitutes the most frequent form of adulteration of milk.

Every 10 per cent. of the fat removed from whole milk represents approximately the loss of half an ounce of cream from the quart of milk, the value of which is about a halfpenny, thus proving a very profitable adulteration to the dishonest vendor.

All cream is now separated from milk by the various

mechanical centrifugal separators, the best forms of which do not leave much more than 0·1 per cent., or at most 0·2 per cent., of fat in the separated milk. The solids-not-fat are of course, owing to the removal of the fat, proportionally higher than in whole milk. Separated milk containing less than 8·7 per cent. of solids-not-fat should certainly be regarded as adulterated.

Koumiss.—Koumiss is a preparation of mares' or asses' milk in a partly-fermented condition, which is largely used in Russia. It is prepared as follows : The milk is allowed to cool, and is then deprived of a part of its cream ; a little yeast is then added ; this sets up a slow fermentation, the milk-sugar being converted into alcohol and lactic acid.

Koumiss is prepared in this country from cows' milk. The following analyses of Russian koumiss by Drs. Bell and Hautier respectively will give an idea of the general composition of this preparation :

	Bell.	Hartier.
Lactic acid	1·96%	1·15%
Casein	2·11	1·12
Sugar	0·40	2·20
Fat	1·10	1·20
Alcohol	2·12	1·65
Ash	0·34	91·83
Water	91·97	

Kephir.—Kephir is a preparation of fermented cows' or goats' milk, very similar to koumiss, and is prepared by means of a special ferment. It is used largely by the tribes of the Caucasus.

We shall now proceed to describe in detail the methods of analysis ordinarily applied to milk :

Specific Gravity.—Directly the samples are received in the laboratory, the gravity should be taken, each sample being carefully shaken first, to mix in any cream that has risen, but avoiding the creation of a quantity of air-bubbles.

The specific gravity may be taken either by a delicate hydrometer or (more exactly) by means of a Westphal balance.

When milk is sampled, it is important to stir up thoroughly the whole quantity contained in the vessel,

so as to ensure a complete distribution of the cream throughout the body of the milk. This is frequently neglected by Inspectors under the Acts, and they should be repeatedly reminded of its necessity. If a sample is to be divided into three parts, one pint should be purchased and poured into bottles of such a size as to be filled almost, but not quite, to the cork. If they are filled entirely, it is difficult to shake them so as to mix in the cream ; while if they are not filled up to the neck, the shaking of the milk in transit may cause some of the cream to be churned into butter, in which case there is no alternative but to evaporate the entire quantity, and apply Bell's method of analysis. The term 'Recknagel's phenomenon' is applied to the increase observed in the specific gravity of milk, which takes place some time after milking. H. Droop Richmond (*Analyst*, 1894, p. 76) gives an example of this, the results of which were as follows :

Specific gravity		$1\frac{1}{4}$	hours	after	milking	...	1031·0
,,	,,	$3\frac{1}{4}$	,,	,,	,,	...	1032·2
,,	,,	18	,,	,,	,,	...	1032·5

This rise in the specific gravity must not be confounded with a similar rise when frothy milk is allowed to stand.

The samples should be shaken gently just before taking the gravity, so as to mix in the cream, but care must be taken to avoid the creation of a quantity of air-bubbles. The specific gravity of genuine milk generally falls between 1029·0 and 1034·0 at 15·5° C. Tables may be used to correct the gravity taken at temperatures near 15·5° C. to what it would have been if the milk had been exactly at 15·5° C., but in practice it is easy to bring them to the correct temperature by standing the bottles in water.

Milk samples should be examined at the earliest opportunity, as curdling takes place very rapidly in summer, and directly the milk is at all curdled it is impossible to estimate the gravity accurately. If the curdling has not proceeded too far, it is possible to obtain a uniform mixture again by adding a few drops of ammonia to the sample and shaking. This will enable us to measure out the required amounts by pipette, instead of resorting to the more lengthy operation of weighing The specific

gravity of milk is raised by the abstraction of fat, and lowered by the addition of water; hence, by partial skimming and watering an adulterated sample may possess the same gravity as that of genuine milk. Whether, therefore, the specific gravity is normal or otherwise, it will be necessary to estimate the fat and the total solids.

Total Solids.—The total solids in milk are estimated by drying 5 grammes in a shallow dish upon a water-bath until constant in weight. A platinum dish is best for this purpose; but whatever kind of dish is used, it will generally be necessary to dry for three hours on the water-bath, and then for two hours inside a water-oven. Where rigid accuracy is not required, 5 cc. may be taken for the solid determination, correction afterwards being made for the weight from the specific gravity of the sample.

Care should be taken to ascertain that the temperature inside the water-oven really reaches 100° C., as some water-ovens give a much lower temperature than this. Some operators prefer to take only 2·5 grammes of milk, and add a drop of very dilute alcoholic acetic acid, which, of course, is driven off by evaporation afterwards; this addition curdles the milk and prevents the formation of a skin over the surface, which retards the drying.

The milk solids should be weighed at once after drying, as they absorb moisture from the air very readily; moreover, as is well known, an increase of weight takes place with all platinum articles on cooling, due to the occlusion of gases on their surface.

The total solids of genuine milk rarely fall below 12·5 per cent.

The total solids of milk being on the average 12·5 per cent., and the fat 3·5, it follows that the solids-not-fat will be 9·0 per cent.; of this, about 0·75 is mineral matter, or ash; about 3·5 per cent. albuminoids, and the remaining 4·75 per cent. milk-sugar.

Estimation of the Ash.—The residue, after the determination of the total solids, is heated cautiously at a temperature as low as possible over a Bunsen burner until a white ash is left. Overheating will cause the result to be too low, owing to loss of sodium chloride. The ash of normal milk is about 0·75 per cent., and is

slightly alkaline. If the ash is found to be materially less than this, it would point to watering. A marked degree of alkalinity and effervescence with hydrochloric acid would suggest the addition of a carbonate to the milk.

The ash of milk is of but little value as an indication of purity, owing to the almost universal practice of adding varying quantities of borax or boric acid to the milk—at least, during the summer months.

The most constant figure in normal milks is the proportion of ash to solids-not-fat, which averages 8·3 per cent. of the solids-not-fat. It very rarely falls beyond the limits, 8·0 to 8·5. In cases of abnormally low solids-not-fat the ash generally bears a higher ratio to them than the normal.

Estimation of the Fat.—There are several different methods of determining the fat, which may be classified as follows :

1. Estimation of the fat by reading off the volume of fat liberated after treatment by chemical means and the employment of centrifugal force. On this principle all the mechanical methods are based.

2. Estimation by simple extraction, by means of a solvent, of the milk dried (*a*) without addition (Bell's method); (*b*) on blotting-paper (Adams' method).

3. Estimation by extraction of the fat by ether, after destruction of the casein by acid (Werner-Schmidt).

Mechanical Methods.—There are several forms of centrifugal apparatus in use—namely, the Leffmann-Beam machine, the Gerber, etc. Perhaps the one at present most commonly used is the Leffmann-Beam machine, which is capable of giving results, in skilled hands, precisely identical with the Adams process. All these methods depend on the liberation of the fat from the milk by treatment with sulphuric acid. The application of centrifugal force then causes the heavier liquid to force the fat to rise up into the neck, which is graduated into divisions corresponding to a tenth of a per cent. of fat by weight.

Leffmann-Beam Process.—Small flat-bottomed flasks are supplied for use with this machine, holding about 40 cc., and graduated on the neck into eighty divisions, ten of these divisions corresponding to 1 per cent. of fat by weight on 15 cc. of milk.

The procedure is as follows : 15 cc. of milk are run into the bottle, and 3 cc. of a mixture of equal parts of fusel oil and hydrochloric acid added, the bottle well shaken, and then 9 cc. of sulphuric acid (95 per cent.), which is added slowly with agitation. This fills the bottle to within 3 or 4 cc. of the graduations ; the liquid is deep brown or nearly black, and some fat can be seen already separated. Sufficient of a hot mixture of equal parts of sulphuric acid and water is added to bring the top of the liquid nearly up to the zero mark, and the bottle is then whirled in the machine. If there are not as many samples to be analyzed as there are places for, the vacant spaces must be filled by bottles full of water to balance the machine, which would otherwise vibrate.

After two minutes' whirling the machine is stopped, and the bottles examined. If the fat and acid liquid are both quite clear, then the fat column is read off ; if the fat or liquid is cloudy, the sample must be whirled again.

There is no need to get the top of the column exactly at the zero mark so long as the fat column is within the graduations. The reading is made from the *extreme top* to the extreme bottom of the fat column.

If there is a cloudy layer between the fat and the acid liquid, it is generally due to careless mixing of the milk and acid. The readings ought to agree within 0·15 per cent. with the Adams figure ; if they are much higher the fusel oil is likely to be in fault, and this may be the cause of serious errors, some samples of fusel oil having been found to give readings as much as 0·4 per cent. in excess of the truth.

The theory respecting the use of fusel oil is that it assists the collection of the fat globules, but does not itself cause any increase in the fat indicated, as it dissolves in the acid liquid.

This method of fat estimation is most useful on account of its rapidity and, in practised hands, its exactness.

In summer, when milks curdle soon after they are received, it is a great advantage to be able to deal with a considerable number, and to ascertain within a short time which of them are suspicious and will require further work.

Gerber Process.—The Gerber apparatus consists of a hollow disc about 18 inches in diameter, on which are

clips for holding the glass tubes; these, when placed in position, are covered by a plate that fits over the disc, forming a hollow box. The disc and its contents are rotated by giving fifteen to twenty sharp pulls with a string, after the manner of a top, and the apparatus, which runs in ball-bearings, then attains a velocity of 2,000 revolutions per minute, and will run by itself for the three minutes which suffice to separate out the fat completely. The procedure, abstracted from the 'Working Instructions' furnished with the machine, is as follows: 10 cc. of sulphuric acid, from 1·820 to 1·825 specific gravity, are placed in the bottle; then 1 cc. of amyl alcohol is added, and finally 10 cc. of the milk to be tested. The bottle is then closed with the indiarubber stopper, and shaken till all the ingredients are thoroughly mixed, and the solution changes to a dark brown. It is then rotated in the machine for three minutes, and the fat is read off. If the top of the fat column does not precisely correspond to one of the graduations, it is readily made to do so by moving the indiarubber stopper slightly.

When skimmed or separated milk is used, the milk must be shaken for two to three minutes, and, after rotating, the test-bottle is to be placed in hot water for a few minutes; this rotating and warming is to be repeated three separate times. The same procedure applies to condensed milk, working on a 10 per cent. solution. Other milk products are tested thus:

1. Cream: Take from ·5 to 1 gramme, add 6 cc. hot water, then 1 cc. amyl alcohol, 6·5 cc. of sulphuric acid, and shake well; add 6 cc. hot water (60° – 70° C.), and rotate in the machine; after a few minutes rotating place in hot water for a short time, and read off.

2. Butter: Weigh out from ·5 to 1 gramme, add 12 cc. cold water, 1 cc. amyl alcohol, and 6·5 cc. of sulphuric acid; shake well and rotate. Place in hot water for a few minutes, and read off.

3. Cheese: If the cheese is soft, pound it in a mortar; if hard, pare it finely. Weigh out ·5 to 1 gramme, add 6 cc. hot water, 6·5 cc. of sulphuric acid, and shake well; add a further quantity of 7 cc. of hot water, then run in about 5 drops of amyl alcohol, close the bottle, and shake well. After having rotated, add 1 cc. amyl alcohol; then

shake gently, and place for a few minutes in hot water (60° to 70° C.). Rotate a second time, place in hot water, and read off. If skimmed cheese is being examined, rotate three times, and make the second addition of hot water 8 cc. instead of 7.

(*a*) **Bell's Method.**—This method (used by the referees) is particularly applicable to curdled milks. In such cases the *entire contents* of the sample bottle should be poured out into a large platinum dish and evaporated nearly to dryness. When nearly dry, the milk solids are stirred with a glass rod, so as to bring them into a state of fine division. The solids are finally ground with ether, poured on to a filter, and washed with the solvent till free from fat. The ether having been distilled off, the fat is weighed.

This is without doubt the best way of treating curdled milks, in which much separation of the curd and serum has often taken place, thus rendering it impossible to work satisfactorily on a part of the sample.

(*b*) **Adams' Method.**—5 cc. of the milk are spotted on to an Adams' paper, which is allowed to dry and is then rolled up. One and a half strips of the specially fat-free Adams' paper will absorb 10 cc. of milk, but it is better not to put more than 5 cc. on one paper. After the paper has dried in the air, it is placed in the bath for a few minutes' final drying, and extracted in a Soxhlet extractor. A very handy form of condenser is the hollow metal ball, which is more efficient than a 3-foot tube condenser. The fat flask should have a short wide neck, and weigh about 15 grammes; its weight to two places of decimals should be marked on it with a diamond. Sufficient ether (specific gravity ·720) should be used to fill the Soxhlet one and a half times, and it should be made to siphon over twelve times at least. It is advisable to place a light screw of paper in the top of the condenser tube, to limit as far as possible the entrance of air, which would deposit moisture inside the condenser and wet the ether. Dry ether has no solvent action on milk-sugar, so that nothing but fat will be extracted if the ether is kept dry; but if it contain water, milk-sugar will come out with the fat, and if much moisture gets into the ether it may cause the coil to become damp, and then there may be an error in either direction—*i.e.*, an excess

owing to milk-sugar being weighed with the fat, or a loss from fat remaining in the damp coil.

This is certainly the best method of fat extraction for reference work where absolute figures are required. The Adams process is the official method of the Society of Public Analysts.

(*c*) **Werner-Schmidt Method.**—The Werner-Schmidt process was first introduced into this country by A. W. Stokes, and is briefly described in the *Chemical News*, vol. lviii., p. 197. In the same paper (vol. lx., p. 214) A. W. Stokes published in detail the exact procedure he recommends, together with a table showing the comparison of estimations made by it, with the amount of fat calculated from the total solids and gravity. We quote the description in his own words :

'Into special tubes (to be obtained of Messrs. Townson and Mercer), which are partly graduated up to 50 cc., pipette 10 cc. of the milk, if fresh, and then pour direct from its bottle some HCl roughly to the 20 cc. mark. If the milk is a sour sample, weigh out 10 grammes, and with a small wash bottle containing strong HCl wash the milk into the tube till it is full to about the 20 cc. mark. Now boil the mixture, with frequent shaking, till it turns brown. Merely heating it to the boiling-point of water by immersion in a water-bath is not sufficient. Leave for about three minutes to stand ; the colour will darken considerably, while thus standing, without further heat. Cool by immersion in water ; fill up roughly to about the 50 cc. mark with ether. It is not necessary that the ether should have been previously washed with water, unless it contains more than 3 per cent. of alcohol. Cork the tube and shake the mixture for half a minute ; let settle for five minutes. Accurately pipette off 20 cc. of the supernatant ethereal solution into a tared beaker, evaporate off the ether, dry in air-bath, and weigh the residual fat. It is advisable to take at least 20 cc. of the ethereal solution, so as to avoid the errors of high multiplication. It is perfectly easy to pipette off accurately 20 cc. of the ethereal solution ; the presence of the fat in the ether prevents the difficulty that is found in pipetting off ordinary ether. Now notice how many cc. of ethereal solution are left in the tube. Here there is

sometimes a slight difficulty, since above the sharp line that separates the brown mixture of HCl and milk from the colourless ethereal solution sometimes there floats a fluffy narrow stratum of casein. If, however, three-quarters of this stratum be assumed to be ether, a correct reading will be made. From the whole quantity of ethereal solution originally present the percentage of fat in the milk is now calculated.

'The whole process, doing at the same time a number of samples, need not take more than twenty minutes. Its accuracy is not excelled by any other process; the process is simple; the reagents are those found in every laboratory; almost all the ether can be recovered, if thought worth while; the only special apparatus needed is a cheap calibrated tube.

'The HCl and the milk in the process should not be boiled together more than two minutes, else the ether will take up a caramel-like substance. Very highly watered milks do not turn a dark brown because of the small amount of milk-sugar present, while condensed and sugared milks become almost black.

'An example may show the calculation required:

'10 cc. of a milk having the specific gravity 1031, and giving 12 per cent. by weight of total solids, when treated thus, gave, in 20 cc. of ethereal solution, 0·277 gramme of fat. There were left in the tube 6·5 cc. of ethereal solution, making a total of 26·5 cc. Then, $\frac{0{\cdot}277 \times 26{\cdot}5}{2} = 3{\cdot}67$ per cent. in 100 cc. of the milk. Dividing this by the specific gravity 1031, we get 3·55 per cent. by weight of fat in the sample. Calculating from the specific gravity and total solids, the fat should be 3·54 per cent.'

Calculation Method.—Seeing that the presence of fat in milk tends to lower its specific gravity, while the presence of the solids-not-fat tends to raise it, it is evident that there is a relation between the gravity, fat, and total solids, which will enable us to calculate the third factor, if the other two are known.

Formulæ have been devised whereby, knowing two of the factors, the third is easily calculated. The formula of Hehner and Richmond is the one now exclusively

used, this having been based on a very extensive series of observations, in conjunction with perfect methods of analysis.

The formula is as follows :

$$F = \cdot 859T - \cdot 2186G.$$

F represents the fat, T the total solids, and G the last two units of the specific gravity, together with any decimal.

The above formula suffices for ordinary milks, but for skim milks it has been found necessary to slightly modify it as under :

$$F = \cdot 859T - \cdot 2186G - \cdot 05\left(\frac{G}{T} - 2 \cdot 5\right).$$

This correction need only be applied when G divided by T exceeds 2·5. Extended tables are published (see the *Analyst*, vol. xiii., p. 26) to facilitate calculation founded on the above formulæ, but a still more ready method of applying the formulæ is by means of the invaluable instrument described below.

Richmond's Slide Rule.—This consists of a wooden rule, part of which is made to slide, and by its aid we can calculate the fat from the total solids and gravity, or the gravity from the total solids and fat, or the total solids from the fat and gravity. The rule has three scales, two of which are for total solids and fat respectively, which are marked on the body of the rule, while that for specific gravity is placed on the sliding part. The divisions are as follows : Total solids, 1 inch divided into tenths; fat, 1·164 inches divided into tenths; specific gravity, each division = 0·254 inches. These numbers show the relation according to the formula

$$T - \cdot 254G = 1 \cdot 164F.$$

For example, supposing that a certain milk has a specific gravity of 1032, and is found to contain 3·5 per cent. of fat, the sliding portion of the rule is adjusted so that the arrow points to 3·5 per cent. of fat. The figure representing total solids will now be exactly below the gravity figure, and in this particular case will show the total solids to be 12·2 per cent., and if we made an actual estimation by evaporation, the figure obtained should vary but little from this. Having obtained the total solids by the slide

rule, we subtract the fat, and if the solids-not-fat do not fall below 8·5 per cent., an actual estimation of the total solids is not necessary.

Total Proteids.—The best method of determining these is by calculation from the total nitrogen, which is determined by Kjeldahl's process. This method, which occupies but a short time, and does not involve the use of complicated apparatus, has almost entirely replaced the combustion process.

It depends on the conversion of the nitrogenous matter into ammonium sulphate, which is subsequently decomposed by an excess of alkali, the liberated ammonia being distilled off and titrated.

About 5 grammes of the milk are weighed into a round-bottomed, long-necked flask of about 300 cc. capacity. The milk is then evaporated to dryness over a water-bath, the last traces of water being got rid of by drying in a water-oven and by gently blowing warm air into the flask. To the dry solids are then added 20 cc. of concentrated sulphuric acid, and 5 to 10 grammes of potassium sulphate, and the whole is heated for some time over a Bunsen flame. At first frothing takes place and white fumes escape, consisting chiefly of water vapour.

The flask should be placed in a slanting position, so as to encourage condensation of the sulphuric acid vapours in the neck as far as possible. When the liquid has become clear and colourless, or nearly so, the flask is allowed to cool: 200 cc. water are added, and the whole poured into the funnel of the distilling apparatus. A further quantity of about 200 cc. of water is used to rinse out the flask; this also is poured into the funnel, and followed by 75 cc. of 50 per cent. soda hydrate solution. The stop-cock of the funnel is closed and heat applied; the ammoniacal steam is freed from splashings by its passage through the glass 'anti-splasher.' With this apparatus the most troublesome substances can be dealt with.

The apparatus is constructed as follows: A copper flask, capable of holding 500 cc., is fitted with a rubber cork, through which passes a Soxhlet tube, the other end of which is closed by a rubber cork pierced by two holes; through one of these passes the stem of a tapped funnel, and through the other the end of a block-tin tube, $\frac{3}{8}$ inch in diameter, which is carried up about 18 inches, and then

down again, its other end passing through a rubber cork into a tapered glass connector, which dips into 50 cc. of $\frac{N}{10}$ sulphuric acid contained in a 4-oz. flask, kept cool by being placed in a vessel of cold water.

The ammoniacal steam condenses in the tin tube, and is received in the acid. After about 250 cc. of distillate have been collected, the stop-cock is opened and the burner turned out.

The distillate is cooled by placing the flask under the tap, and then titrated with $\frac{N}{10}$ soda hydrate, till the excess of acid is neutralized, methyl-orange being used as indicator.

Each cc. of $\frac{N}{10}$ sulphuric acid neutralized by the ammoniacal distillate corresponds to ·0014 gramme of nitrogen, or ·0017 gramme of ammonia.

Working with ordinary reagents, it will be found that a 'blank' experiment usually requires ·2 cc. of $\frac{N}{10}$ sulphuric acid, hence ·2 cc. should be subtracted from the number of cc. of $\frac{N}{10}$ soda used to neutralize the 50 cc. of acid taken.

The total proteids are estimated by multiplying the amount of nitrogen found by the factor 6·33. This will give a very close approximation to the total amount of proteids present, as they contain on the average 15·8 per cent. of nitrogen.

The Estimation of Sugar by Pavy's Method.—This method is a modification of Fehling's process, where, instead of weighing the copper in the form of cuprous oxide, as in the ordinary method, the modification devised by Dr. Pavy depends on the fact that cuprous oxide dissolves in ammonia, forming a colourless liquid. When the saccharine liquid to be tested is run into the boiling Pavy solution, instead of a bulky red precipitate falling and obscuring the end reaction, the liquid changes from blue to colourless.

To prepare the test solution, dissolve 20·4 grammes of Rochelle salt and the same weight of caustic potash in distilled water; dissolve separately 4·158 grammes of pure cupric sulphate in water by the aid of heat; add the copper solution to that first prepared, and when cold, add 300 cc. of strong ammonia, and distilled water to 1 litre.

If ordinary Fehling solution is to hand, the Pavy solution is most readily made as follows: To 120 cc.

of the Fehling solution is added 300 cc. of ammonia (specific gravity ·880), and 400 cc. 12 per cent. caustic soda solution ; the mixed solutions are then made up to 1 litre. 100 cc. of this solution has the same oxidizing power on glucose as 10 cc. of the ordinary Fehling solution.

Pavy's solution possesses a different oxidizing power on maltose and lactose from that exerted by Fehling's solution. Its reaction on invert sugar under the above-named conditions is only five-sixths of that exerted by Fehling's test. Hence 120 cc. of the latter are employed in making the ammoniacal solution instead of 100, as would be the case if they were strictly equivalent (A. H. Allen).

In working with Pavy's solution, 50 cc. of the reagent (=0·025 gramme glucose, or 0·0481 lactose) should be placed in a flask, and the milk, diluted with fairly strong ammonia, run in from a burette, the nose of which passes through a hole in the cork which closes the mouth of the flask. Another tube, just passing through the cork, is connected to a rubber tube leading the ammoniacal vapour out of the window. The Pavy's solution is brought to a boil and maintained just boiling, and then the diluted milk is run in slowly till the blue colour is discharged. The reaction does not occur so rapidly as when glucose is brought into contact with Pavy's solution.

A suitable dilution for the milk would be five parts of milk made up to 100 cc., with ammonia and water.

Polarimetric Method of Determination of Lactose.—Milk-sugar can be readily determined by determining the rotation of the sample, after the removal of the fat and proteids. The best form of instrument for the purpose is one of the half-shadow type, for use with the sodium monochromatic light. This, destroying all colour, causes a dark shadow to appear when the instrument is used, and so enables colour-blind persons to use the instrument without difficulty.

The specific rotatory power of a body, usually expressed as $[\alpha]^{\circ}$, is the amount of angular rotation in degrees of the ray of polarized light which is produced when a solution of the substance, containing 1 gramme in 1 cc., is examined in a column 1 decimetre long. It is expressed by the following formula :

Let a=the observed angle, c the strength in grammes per 100 cc., and l the length of the tube used in decimetres; then—

$$[a]^{D} = \frac{100a}{c \times l}.$$

The calculation of the amount of sugar corresponding to the observed rotation may be determined by substitution in the formula.

The specific rotatory power of lactose at 20° C. is 52·5° when observed by the sodium flame. The rotatory power is unaffected by the degree of concentration within the limits met with in ordinary analytical work. It is also but slightly affected by temperature, it being decreased by about 0·042° for each successive rise of each degree of temperature.

When milk-sugar is freshly dissolved in water, it exhibits the phenomenon known as 'birotation,' whereby it shows a higher rotation than that given above. By standing, or immediately on boiling and cooling, the rotatory power falls to normal. In preparing solutions of solid milk-sugar, care must therefore be taken to raise them to the boiling-point before making up to a definite volume. This precaution is unnecessary when operating upon milk.

To determine the amount of milk-sugar by means of the polarimeter, the proteids and fat must be removed. This is best done by means of mercuric nitrate, prepared as follows: Mercury is dissolved in an equal weight of nitric acid of specific gravity 1·42; when dissolved, an equal weight of water is added.

The process is as follows: 60 cc. of the milk are placed in a 100 cc. flask, 1 cc. of the mercury solution is added, and water added up to the mark. The solution, after being well shaken, is filtered through a wet filter. The liquid, which should be quite clear, is now ready for examination in the polarimeter tube. Several readings should be carefully made, and the average taken. Correction should be made for the space occupied by the fat and proteids. This is done by finding the volume of the fat by multiplying the weight by 1·075, and that of the proteids by 0·8.

For example : Specific gravity of sample of milk taken 1030·0. Fat, 4 per cent. Total proteids, 4 per cent.

Milk taken $=60\times1{\cdot}03=61{\cdot}8$ grammes.
Weight of fat $=4$ per cent. of $61{\cdot}8=2{\cdot}47$ grammes.
Volume of fat $=2{\cdot}47\times1{\cdot}075=2{\cdot}65$ cc.
Weight of proteids $=4$ per cent. of $61{\cdot}8=2{\cdot}47$ grammes.
Volume of proteids $=2{\cdot}47\times{\cdot}8=1{\cdot}97$ cc.

$\therefore$ The actual bulk of the sugar-containing liquid is :

$$100-(2{\cdot}65+1{\cdot}97)=95{\cdot}38 \text{ cc.}$$

In order to avoid the calculation involved in taking 60 cc. of sample as above, an amount may be employed which is a simple multiple of the standard amount of the polarimeter to hand. In case of instruments adjusted so that 16·19 grammes of sucrose (=20·56 grammes of milk-sugar) in 100 cc. of the solution produces 100 degrees on the per cent. scale. Therefore 61·68 (=20·56 grammes $\times3$) grammes of the sample are weighed out, treated with mercury solution, and made up to 100 cc. The joint volume of the fat and proteids is calculated as above, and the same volume so found is added to the 100 cc. The flask is well shaken and filtered as before. The bulk of the sugar-containing liquid will now be exactly 100 cc., and the polarimeter readings divided by 3 (if a 200 mm. tube be used) will give the percentages of hydrated lactose.

Detection and Estimation of Preservatives in Milk.—The following are those which are most commonly employed : (1) Borax and boric acid ; (2) formaldehyde ; (3) salicylic acid.

This is the order in which we have most frequently met with them, but there is no doubt that formaldehyde, being the most effective, when it becomes more generally known, will supersede boric acid as a preservative. Mixtures of the above are sold to farmers and dairymen under protected names, with directions how, and in what quantity, they are to be added.

Borax and Boric Acid, $Na_2B_4O_710H_2O$; H_3BO_3.—The presence of either may be demonstrated by ashing a few cc. of the milk, and, after moistening with strong sulphuric acid and alcohol, applying a light, when a green colour, which is more easily seen by placing the dish in the dark, is imparted to the flame, if either boric acid or

borax be present. This reaction is fairly delicate, for as small a quantity as 0·01 per cent. can be detected in this manner.

Another and slightly more delicate mode of testing for these is by treating the ash with dilute hydrochloric acid; on placing a small piece of freshly-prepared turmeric-paper in the dish, it will turn reddish-brown, becoming bluish-black when treated with a little sodium carbonate solution.

Boric acid in milk may be estimated by Thomson's method as follows: One to two grammes of sodium hydrate are added to 100 cc. of milk, and the whole evaporated to dryness in a platinum dish. The residue is thoroughly charred, heated with 20 cc. of water, and hydrochloric acid added drop by drop until all but the carbon is dissolved. The whole is transferred to a 100 cc. flask, the bulk not being allowed to get above 50 to 60 cc., and 0·5 gramme dry calcium chloride added. To this mixture a few drops of phenolphthalein solution are added, then a 10 per cent. solution of caustic soda, till a permanent slight pink colour is perceptible, and finally 25 cc. of lime-water. In this way all the phosphoric acid is precipitated as calcium phosphate. The mixture is made up to 100 cc., thoroughly mixed and filtered through a dry filter. To 50 cc. of the filtrate (equal to 50 grammes of the milk) normal sulphuric acid is added till the pink colour is gone, then methyl-orange, and the addition of the acid continued until the yellow is just changed to pink. Fifth-normal caustic soda is now added till the liquid assumes the yellow tinge, excess of soda being avoided. At this stage all acids likely to be present exist as salts neutral to phenolphthalein, except boric acid, which, being neutral to methyl orange, exists in the free condition, and a little carbonic acid, which is expelled by boiling for a few minutes. The solution is cooled, a little phenolphthalein added, and as much glycerine as will give at least 30 per cent. of that substance in the titrated solution, and titrated with fifth-normal caustic soda till a distinct permanent pink colour is produced; each cc. of fifth-normal soda is equal to 0·0124 gramme of crystallized boric acid. A series of experiments with this process showed that no boric acid was precipitated along with the phosphate of calcium so long as the solution operated

upon did not contain more than 0·2 per cent. of crystallized boric acid, but when stronger solutions were tested, irregular results were obtained. The charring of the milk is apt to drive off boric acid, but by carefully carrying the incineration only so far as is necessary to secure a residue which will yield a colourless solution, no appreciable loss occurs.

A modification of Gooch's method is recommended by C. E. Cassal (*Analyst*, 1890, p. 231), and is performed as follows : In the case of cream about 50 grammes, and in that of milk about 100 grammes, of the sample are rendered alkaline with caustic soda, evaporated to dryness and incinerated. The ash, which need not of course be burnt white, is ground up and transferred, washing in, with a little methyl alcohol and a few drops of water, to a conical flask of from 200 cc. to 300 cc. capacity, provided with a doubly-perforated caoutchouc cork, through which pass a stopcock-funnel tube and a delivery tube. The flask is attached to an ordinary condenser by means of a flexible joint, in such a way as to admit of its being shaken round occasionally, and is placed in an oil-bath. The mixture in the flask is acidified with acetic acid, and 5 cc. of methyl alcohol are run in from the funnel tube and distilled. The distillate is received in a weighed amount of pure lime (ignited to constant weight). About 1 gramme is a convenient quantity. The lime is contained in a platinum dish capable of holding about 70 cc., which is placed in a glass receiver.

This receiver may be made from a ground-edged glass vessel, covered with a perforated ground-glass plate. The end of the condenser passes through a grooved or closely-fitting cork in the perforation of the plate, and terminates just above the lime in the platinum dish.

Distillation with 5 cc. of methyl alcohol is repeated several times, ten such treatments being ample for all likely cases ; fewer are generally quite sufficient. A gelatinous mixture is obtained in the platinum dish ; it is well stirred, and allowed to stand for some minutes ; it is then evaporated in an oil-bath, and finally heated over the blowpipe flame to constant weight. The increase of weight is boric acid, and the error does not in any case amount to more than 1 milligramme *plus*, when dealing with from 0·1 to 0·25 or 0·3 gramme of anhydrous boric

acid. To make sure that the acid has been completely volatilized, the residues in the distillation-flasks are tested with turmeric-paper.

The few drops of water added before the first distillation greatly increases the rapidity of the removal of the boric acid.

Formaldehyde, CH_2O.—Formaldehyde is, comparatively speaking, a new food preservative, and is by far the most effective ; consequently, a smaller amount being required to preserve the milk, its detection and estimation is a somewhat difficult matter.

It is usually added in the form of a 40 per cent. solution, which is called 'formalin.' Two or three drops in a pint of milk keeps it fresh for three or four days, and the addition of 0·05 per cent. preserves milk for months.

In the trade a much more dilute solution of formaldehyde is generally employed, namely, 1 part of formaldehyde to 80 of water. S. Rideal states that a quarter of a pint of such solution added to 17 or 18 gallons of milk keeps it fresh for at least three days, and does not communicate any smell or taste to the milk.

Formaldehyde may be detected by the following methods :

On tasting milk containing formaldehyde, a peculiar sensation is noticed at the back of the throat, and when strong hydrochloric acid is added to it (as in the Werner-Schmidt process), the casein turns yellow, and is less soluble than that of pure milk.

The most reliable test is one pointed out by Otto Hehner. It is based upon the fact that when milk, formaldehyde, and sulphuric acid are mixed together a blue coloration is formed. The best method of applying the test, according to Richmond and Boseley, is to dilute the milk with an equal bulk of water and add sulphuric acid of 90 to 94 per cent. strength. Under these conditions milk, in the absence of formaldehyde, gives a slight greenish tinge at the junction of the two liquids, while a violet ring is formed when formaldehyde is present. This colour is permanent for two or three days. In the absence of formaldehyde a brownish-red colour is developed after some hours, not at the junction of the two liquids, but lower down in the acid. It cannot be

mistaken by anyone who has had any experience with the test for the formaldehyde reaction.

It is stated that 1 part of formaldehyde in 200,000 parts of milk can be easily detected by means of this test, but the blue coloration is not obtained with milks containing over 0·5 per cent. This method is represented to be much more delicate than Schiff's test, which is as follows:

The reagent used is a solution of magenta decolourized by sulphurous acid, care being taken not to have an excess of sulphurous acid. This is added to the whey obtained by coagulating the casein with a little dilute sulphuric acid and filtering, the presence of formaldehyde being indicated by a red coloration.

Otto Hehner prefers to conduct the operation by adding five drops of the reagent to the distillate from 100 cc. of milk (amounting to about 25 cc.), and placing the mixture in a stoppered cylinder; he observes the colour next morning, and then adds a few drops of sulphurous acid solution. After a short time any colour which may be due to oxidation will have vanished, while that due to the presence of an aldehyde remains. There is certainly a difference in the tint produced by colour oxidation, which resembles that of rosaniline, and that of the aldehyde compound, which is violet; and with those small traces with which we have often to deal, only a comparison of the relative colours would allow of anything like a safe conclusion being drawn.

Another test suggested by Otto Hehner is the following, which is equally sensitive as the foregoing:

If to the distillate from a sample of milk, etc., one drop of a dilute aqueous solution of phenol is added, and the mixture poured upon strong sulphuric acid contained in a test-tube, a bright crimson colour appears in the zone of contact. This colour is still readily seen with one part of formaldehyde in 200,000 of water. If there is more than one part in 100,000, there is seen above the red ring a white, milky zone, while in stronger solutions a copious white or slightly pink, curdy precipitate is obtained. This reaction has this advantage over the one above referred to, that it is obtained with formaldehyde solutions of all strengths, while the blue colour is not obtained with milk containing much formaldehyde.

Acetaldehyde also gives a coloration and a precipitate with phenol and sulphuric acid, but it is orange-yellow, not crimson.

R. T. Thompson has recently made experiments with the object of proving the presence of formaldehyde in milks, and has found that a modification of the well-known reaction with ammonia nitrate of silver gives a good indication of its presence. To apply the test 100 cc. of the milk are carefully distilled until (say) 20 cc. of distillate comes over; this is transferred to a stoppered tube, and about 5 drops of ammonia silver nitrate are added. (This solution is prepared by dissolving 1 gramme of silver nitrate crystals in 30 cc. of distilled water, adding dilute ammonia till the precipitate at first formed is redissolved, and then making up to 50 cc. with water.) The mixture of the milk distillate and the silver solution is now allowed to stand for several hours in a dark place (as much as twelve to eighteen hours may be necessary if very little formic aldehyde is present), when, if formic aldehyde is present, a strong black colour or deposit will be produced. A light brown colour should be disregarded; but, so far as his experience goes, the production of a decided black under these circumstances is only brought out by formic aldehyde, but possibly by other aldehydes also. The usual method of heating with the silver solution in order to obtain a silver mirror is of no value with weak solutions of formic aldehyde. It was found that genuine milks from various sources, when tested by the method described, gave no reaction whatever, even when the distillate was left mixed with the silver solution for twenty-four hours, or at most gave a slight brown tinge. When as little as 2 grains of the 40 per cent. formalin were added to 1 gallon of milk (which before the addition gave no reaction with this process), the distillate from 100 cc. gave a decided black colour, or deposit, intense enough to render the mixture quite opaque. As 2 grains per gallon is a quantity of formalin which would be of little value in the preservation of milk, it is evident that this method of testing is quite delicate enough for the purpose. It ought to be noted that, if a milk contains about 2 grains of formalin per gallon, the 20 cc. distillate from 100 cc. of the milk appears to contain all the formic aldehyde that will distil

over, and distillates after that give practically no reaction. A milk containing 7 or 8 grains per gallon of the preservative may require the distillation to be carried on till 30 or 40 cc. are collected before it ceases to show a reaction with the silver solution; but in all cases the reaction can be got by distilling over the 20 cc., or often indeed 10 cc.

Salicylic Acid, $C_7H_6O_3$.—The use of this acid as a milk preservative is gradually diminishing in this country, though it is still largely employed on the Continent.

It is best detected by H. Pellet's method as given in Allen's 'Commercial Organic Analysis,' vol. iii., part i., p. 53. 200 cc. of the milk are diluted with an equal measure of water, then heated to 60° C., and treated with 1 cc. of acetic acid and an excess of mercuric nitrate free from mercurous salt. The salicylic acid is extracted from the filtered solution by agitation with ether, and recognised by evaporating a little of the ethereal solution to dryness, and testing the residue with ferric chloride, which gives a violet coloration with salicylic acid.

Bacteriology of Milk.—Milk and other dairy products are liable to several diseases or taints of bacterial origin, and are frequently a source of infection of several diseases, such as typhoid, tuberculosis, diphtheria, etc., as a result of disease of the animals themselves, or of the unsanitary conditions which surround the milk-supplies in many parts of the country. For a full description of these, and of the methods employed in their investigation, the reader is referred to the following works of the authors: 'A Manual of Applied Bacteriology,' 'Aids to the Study of Bacteriology' (Baillière, Tindall and Cox).

CREAM.

'Devonshire' or 'Cornish' cream is prepared by warming milk in pans for several hours, when the cream rises to the top in a much more coherent layer and more rapidly than if the milk is merely allowed to stand at room-temperature. The cream obtained in this way contains 60 per cent. or more of butter-fat. Owing to the partial sterilization that it has thus undergone, cream prepared in this way keeps sweet without the addition

of preservatives much longer than separated cream would.

Before the introduction of separators, cream used to be prepared either by the above-mentioned method, or by simply allowing milk to stand overnight and skimming off the cream in the morning. Such a rough method as this would of necessity result in a product containing very varying amounts of fat, according to the temperature of the milk and the shape of the vessel. Now, however, there are a number of centrifugal machines in the market, by the use of which milk can be almost entirely denuded of its cream.

The percentage of fat in separated cream is often as high as 65 per cent., but it is rarely sent out for sale at this strength, and the retailers dilute it down considerably before it reaches the consumer, in many cases adding an equal volume of milk.

It is therefore evident that a standard for cream should be fixed, and a reasonable standard would be that it should contain not less than 45 per cent. of fat.

Cream is generally bought and sold on its taste and appearance, not on the amount of fat that it contains. There is thus a certain temptation to thicken it artificially so as to enable a thin cream to sell at the best price, and we recently examined a sample containing only 40 per cent. of fat which had been thickened with gelatine so as to bring it to the same thickness as a sample containing 60 per cent. of fat. The addition of any considerable quantity of gelatine to cream causes it to assume a buttery consistency, and it will no longer pull out into strings as genuine rich separated cream will.

The difference is very apparent on comparing a sample of cream adulterated in this way with a genuine sample.

Detection of Gelatine.—Gelatine in cream may be detected by carefully drying a weighed portion, as in Bell's method for the analysis of milk, removing the fat with ether, and taking up the residue with the least possible quantity of boiling water. On allowing to cool, if gelatine is present, the liquid will set solid.

Another method, for which we are indebted to Mr. A. W. Stokes, depends on the precipitation of gelatine by tannin. Mix a weighed quantity of the suspected sample with warm water, and add acetic acid to pre-

cipitate fat and albuminoids, taking care to avoid excess; filter, and to the clear liquid add a few drops of strong solution of tannin.

A sample of genuine cream should be treated in the same way for comparison. On addition of the tannin solution, a slight precipitate is produced in the case of genuine cream; but in a sample adulterated with gelatine a copious precipitate will be thrown down.

Fat.—The amount of fat in cream is most readily estimated by weighing out 2 grammes into a small dish, thoroughly mixing with about 12 cc. of water, pouring into a Leffmann-Beam bottle, and treating in the same way as ordinary milk. The result obtained is multiplied by the factor, 7·77, as explained in the analysis of cheese. The fat in cream might also be estimated by the Werner-Schmidt method, or by the Adams method; but the process above described is quite accurate and by far the quickest.

Preservatives.—Separated cream almost invariably contains preservatives; one preservative, very commonly used, consists of borax and boric acid, sweetened with saccharine. The use of a preservative is practically a necessity in the cream trade, as separated cream will begin to turn sour in as short a time as four hours in the summer, if no preservative has been added.

CONDENSED MILK.

Condensed milk seems to have been first prepared about the year 1856, and is now an article of great importance, particularly on account of the great quantity used in the feeding of young children.

There are about fifty brands at present on sale in this country, many of which are the same milk under different labels. They may be roughly divided into four classes:

1. Unsweetened milks.
2. Sweetened milks.
3. Sweetened partly-skimmed milks.
4. Sweetened skimmed milks.

In most cases the degree of concentration is that obtained by evaporating three volumes to one; that is,

the addition of two volumes of water (to an unsweetened milk) will produce a strength equal to the original.

1. The unsweetened milks, of which there are at present four different brands, are well prepared, and keep perfectly. They contain the due proportion of fat.

2. This class forms by far the largest and most important part of the whole supply, and for the most part there is nothing to complain of in them, except that the dilutions recommended would in very nearly every case produce a milk very much below standard. A sweetened condensed milk generally contains rather more added cane-sugar than milk solids. The following figures are typical of a good specimen :

Fat	11·0	per cent.
Proteids	10·0	,,
Milk-sugar	14·0	,,
Ash	2·2	,,
Cane-sugar	38·0	,,

3. This class seems now to have almost disappeared.

4. Separated milks (generally miscalled skimmed milks) are largely used by poor and ignorant people for infant-feeding, and there can be no doubt but that great harm results from the practice.

It appears that some of the labels on some of the tins of inferior condensed milk bear the words 'skimmed,' but in such small type that the intimation might be easily overlooked.

In a paper on 'The Composition and Analysis of Condensed Milk,' printed in the *Analyst* for December, 1895, we drew attention to the inadequate disclosure of the contents made on most of the labels, and stated that it ought not merely to be enacted that the words 'skimmed' or 'separated' should be printed on the label in as conspicuous type as the words 'condensed milk,' but that it should be made compulsory to add, 'Skimmed milk is unfit for the nourishment of children.'

In the case of even the best condensed milks, prepared from genuine milk with all its fat, the directions for dilution are very open to criticism.

The following typical analyses of condensed milk are selected from the above-mentioned paper, which con-

tained the results on practically all the brands which were then on the market :

Brand.	Total Solids.	Fat.	Milk-sugar.	Pro-teids.	Cane-sugar.	Remarks.
First Swiss .	36·7%	10·5%	14·2%	9·7%	—	Un-sweetened.
Ideal ...	38·0	12·4	16·0	8·3	—	
Viking ...	34·2	10·0	13·3	9·0	—	
Anglo-Swiss	74·4	10·8	16·0	8·8	37·1%	Sweetened whole milk.
Fourpenny .	76·5	10·4	13·0	9·8	41·3	
Mother ...	72·0	8·8	13·7	7·3	40·5	
Milkmaid ...	76·3	11·0	14·6	9·7	38·7	
Nestlé's ...	77·2	13·7	15·0	9·7	37·2	
Calf ...	58·0	1·0	16·0	7·5	31·9	Sweetened skim milk.
Cup... ...	56·9	1·0	15·4	8·5	30·4	
Goat ...	71·0	1·2	12·0	9·9	45·9	
Handy ...	75·5	0·3	17·0	12·3	44·3	
Lancer ...	67·6	0·3	16·6	12·3	35·8	
Minstrel ...	75·3	0·2	15·4	9·7	48·3	

The Analysis of Condensed Milk.—The analysis of condensed milk should comprise estimations of the total solids, ash, proteids, milk-sugar, and fat. The last four items added together and subtracted from the total solids will give the cane-sugar with fair accuracy. We have examined the ash of a number of milks for tin and lead, but they have been absent in every case. In examining any tinned article for tin or lead, great care must be taken to use a *sharp* tin-opener, or fragments of metal may be torn off and vitiate the result. The tin should be cut open, the contents should be thoroughly mixed, and 10 grammes weighed out, and made up to 100 cc. We have now a 10 per cent. solution, which serves conveniently for the following estimations :

Total Solids.—20 cc. of the solution are evaporated in a platinum dish till constant in weight ; this will take five or six hours.

Ash.—The same quantity will serve for the determination of ash, and should be ignited at as low a temperature as possible. The ash in condensed milk varies, but on the average is somewhat over 2 per cent.

Proteids.—10 cc. of the solution are evaporated to dryness in an 8-oz. oxygen flask, and the nitrogen is determined by the Kjeldahl process ; the proteids are then calculated by the 6·3 factor.

In May, 1893, Richmond and Boseley read a paper before the Society of Public Analysts, entitled 'Points in the Analysis of Condensed Milk,' in which they give methods for the estimation of the casein and the albumen, and point out some of the errors liable to occur in the use of Ritthausen's process for determining proteids. They recommend the Adams process for the extraction of the fat.

Milk-sugar.—10 cc. of the solution are made up to 100 cc. by the addition of 40 cc. of water and 50 cc. of ammonia. This gives us a 1 per cent. solution, which is a convenient strength for the estimation of the sugar by Pavy's method. In applying Pavy's test to milk-sugar, the reagent must be kept boiling briskly while the milk-sugar solution is run in slowly. The reaction takes longer in the case of milk-sugar than in the case of glucose, so that unless the titration is conducted slowly, the milk-sugar solution may be added in excess of the quantity required to complete the reaction. Using ordinary Pavy's solution, it will be found in the case of most condensed milks that about 35 cc. of the 1 per cent. solution of condensed milk will be equivalent to 50 cc. of Pavy's solution, and the calculation will be as follows :

If 35 cc. dilute milk = 50 cc. Pavy's solution,
Or 70 cc. dilute milk = 100 cc. Pavy's solution,
Or ·7 gramme sample = 100 cc. Pavy's solution,
" " = ·05 gramme of glucose,

$$\therefore \text{100 grammes of sample} = \frac{\cdot 05 \times 100}{\cdot 7} = 7\cdot 1 \text{ grammes glucose,}$$

$$\text{And 100 grammes of sample} = \frac{\cdot 05 \times 100 \times 100}{\cdot 7 \times 52} = 13\cdot 7 \text{ per cent. lactose.}$$

Fat.—Two quantities of 5 cc. of the solution are placed on two Adams papers, which are well dried and then extracted with dried ether.

The method of drying with sand or calcium sulphate, and then extracting with ether, is unsatisfactory, as the fat is extracted with great difficulty.

We have attempted to apply the Leffmann-Beam machine to the estimation of fat in condensed milk, and it succeeds fairly well if the following details are adhered to : Place 10 cc. of the 10 per cent. solution in a Leffmann-Beam bottle with 3 cc. of the hydrochloric acid and fusel-oil mixture, shake well, and add 15 cc. of sulphuric acid, 95 per cent., and fill up to the mark to the hot mixture of sulphuric acid and water. The bottle is then whirled for three minutes. The fat will not all come out at once, and after whirling the bottle should be placed in the water-bath and then whirled again, when the entire amount of fat will be separated. The chief objection to this method is the large factor that has to be employed, namely, 15·5, showing that we are working on 1 gramme of the original sample.

It is rarely possible to get the figures representing the separately-determined constituents when added up to agree with the total solids in an ordinary milk, but the error is usually small ; hence, in the case of sweetened condensed milks it is usual to subtract the sum of the ash, fat, proteids, and milk-sugar from the total solids, and to consider the difference added sugar.

With respect to the difficulty just mentioned of getting agreements between the sum of the separately-determined constituents and the total solids, some of the anomalies observed may be due to the fact that milk-sugar may or may not be dehydrated, according to the manner in which the evaporation has been conducted ; or, again, the proteids may have become altered during the process of condensation, causing them to be incorrectly represented by the ordinary factor.

Condensed sweetened milk was at one time used as an adulterant of cow's milk, but it is not now employed for that purpose. Unsweetened condensed milk is sold in large tins, which are made use of by the retailer to supplement his daily supply on occasions when milk is short.

Attempts have frequently been made to replace the fat in separated condensed milk by working in some foreign oil or fat, just as margarine cheese is prepared by adding

margarine fat to skim milk and then curdling in the ordinary way.

Foreign fat in condensed milk would be readily detected by extracting 200 grammes of the sample with ether, after grinding with enough anhydrous sulphate of copper to form a dry powder, and examining the fat when quite free from the solvent by the Valenta or Reichert tests.

A sample of condensed milk may be regarded as genuine which fulfils the following requirements :

1. The fat must not be less than 10 per cent., and must be true butter-fat.

2. The abuminoids, estimated by multiplying the nitrogen figure by the 6·3 factor, must not exceed the figure obtained for fat.

3. The sample must be free from preservatives (except sugar), starch, and all other foreign matters.

Humanized Condensed Milk.—It has long been recognised that, however pure cow's milk may be, the proportion of albuminoids which it contains is necessarily higher than that in human milk, and it must therefore be more difficult of digestion by infants. Various methods have been used for removing a portion of the excess of albuminoids, and the preparations so obtained are called 'humanized milks,' because they closely approximate to human milk in chemical constitution.

After considering these methods, one of us (C. G. M.) found it practicable to produce a milk of similar composition in a condensed form, constituted as follows :

Fat	13·5
Albuminoids...	7·0
Milk-sugar	21·2
Mineral matter	2·0
Water	56·0
	100·0

When diluted with three times its volume of water its composition will closely approximate to that of human milk. It contains no cane-sugar, colouring matter, or preservative.

BUTTER.

By continuously shaking or beating milk at a high temperature, the fat corpuscles can be divided, and their number increased. If, however, the milk is cooled, and is then shaken or beaten, the fat corpuscles adhere together and form *butter*, a thin bluish *butter-milk* remaining behind.

The amount of butter-fat in butter prepared from cow's milk is about 85 per cent., the remainder being water, casein, or curd, and generally added salt.

Butter varies in colour from white to deep yellow, and is more or less granular in character.

The butter-fat is very complicated in composition, consisting as it does of fatty acids in combination with glycerol, forming triglycerides.

The fatty acids that enter into the composition of butter-fat are : butyric, caproic, caprylic, capric, myristic, palmitic, stearic, and oleic acids. The first four are soluble in water, and are therefore known as 'soluble fatty acids' ; the latter, being insoluble, are known as 'insoluble fatty acids.'

Dr. J. Bell has published the following analysis of a sample of butter-fat :

Butyric acid	6·1%
Caproic, caprylic, and capric acids ...	2·1
Myristic, palmitic, and stearic acids ...	49·4
Oleic acid	36·1
Glycerol (calculated)	12·5

The proportion of butyric acid and its immediate homologues produced by the saponification of butter-fat ranges between 5 and 8 per cent.

The amount of glycerol in butter-fat was first determined by Chevreul, who obtained 11·85 per cent. by direct weighing of the isolated glycerol. Benedict and Zsigmondy, by oxidizing the glycerol with permanganate of potash, and determining the oxalic acid so formed, have found from 10·2 to 11·6 per cent. of glycerol to be formed by the saponification of butter-fat. A. H. Allen has confirmed these experiments.

These analytical results show that butter-fat is essentially a mixture of various triglycerides, those of butyric, palmitic, and oleic acids being the leading constituents :

Tributyrin	Tripalmitin	Triolein
$C_3H_5(O.C_4H_7O)_3$	$C_3H_5(O.C_{16}H_{31}O)_3$	$C_3H_5(O.C_{18}H_{33}O)_3$.

Some experiments of Dr. J. Bell indicate that the glycerides contain several acid radicles in the same molecule, and therefore the butyrin cannot be separated by any process of fractional solution from the less soluble glycerides of palmitic and oleic acids. Hence butter-fat probably contains complex glycerides of the following characters :

$$C_3H_5\begin{cases} O.C_4H_7O. \\ O.C_{16}H_{31}O. \\ O.C_{18}H_{33}O. \end{cases}$$

Such a complex glyceride would yield on saponification fatty acids and glycerol in the same proportion as would be obtained from a mixture of butyrin, palmitin, and olein in the ratio of their molecular weights (A. H. Allen, 'Commercial Organic Analysis,' vol. ii.).

On keeping, butter may turn rancid, especially if the butter-milk has not been well washed out : but the changes it undergoes would not, under ordinary circumstances, be sufficient to very seriously invalidate the Reichert, Valenta, or 'soluble' and 'insoluble' fatty acid determinations.

Such decomposition as would occur in the course of two or three months would probably never be sufficient to invalidate the results of analysis.

Margarine.—Margarine, oleo-margarine, butterine, or Dutch butter, as it used to be termed, is prepared by churning melted and clarified animal fats, usually beef or mutton fat, occasionally lard (vegetable oils are now but rarely employed), with skim milk, milk, or cream ; in this way the curd or casein found in the margarine contracts more or less the flavour of genuine cow's butter. When margarine is carefully prepared and duly coloured, it is not easy to tell the same from pure butter by taste or smell.

Any fictitious butter can now only be legally sold in this country under the term 'margarine,' and must be so

marked by a label bearing this name in letters not less than 1½ inches high. If unlabelled, an inspector may require the shopkeeper to supply him with the article as butter, and convictions are often obtained in this manner (see the Margarine Act).

The Analysis of Butter.—Margarine differs from butter in yielding only traces of 'soluble' fatty acids. It consists mainly of the glycerides of oleic, stearic and palmitic acids. The absence of glyceryl butyrate, which is the chief characteristic of butter-fat, is the most valuable means of distinguishing between butter and margarine. On this difference depend the Reichert, Valenta, and Hehner tests, the three most trustworthy tests we have for butter.

Water.—Ordinary good butter should contain about 12 per cent. of water; anything over 16 per cent. should be held to be adulteration. Out of 1,500 samples of English and foreign butter examined by Vieth, Richmond, Bell, and others, only 0·6 per cent. contained over 16 per cent. of water; the larger number contained between 11 and 13 per cent. of water. Where the amount exceeds 16 per cent., it has either been left in by careless manufacture or fraudulently incorporated. Professor Tichborne is apparently in favour of allowing more than 16 per cent. of water in Irish salt butters, but we cannot believe that they could not be made so that the water should not exceed 16 per cent.

The amount of water can be best estimated by drying 10 grammes in a platinum dish at 105° C. until practically constant in weight; this will generally be when no crackling noise can be heard when the ear is brought near the dish.

Salt.—The residue, after burning off the fat from the above, can be taken as salt for all practical purposes. The salt in butter may amount to, but does not often exceed, 10 per cent. Dr. Bell found as much as 15 per cent. in one sample of English butter.

Casein or **Curd** can be estimated by drying the butter as above, transferring to a filter, and washing with ether until fat-free, the residue, which consists of casein, being weighed. The casein varies from 0·3 to 4 per cent.

Examination of the Fat.—The sample is put into a beaker and placed in the water-bath for a short time,

when the water and curd will settle to the bottom. The fat is then decanted and filtered through a dry filter-paper. If this is carefully done, the fat will be quite clear and bright.

The fat is now examined by the Reichert-Meissl and the Valenta acetic acid test. The indications given by these two tests are generally all that is necessary for all ordinary purposes, but in the case of suspicious or adulterated samples it may be desirable to determine the soluble and insoluble fatty acid by the Hehner method, and also to take the specific gravity of the fat.

The Reichert Method of estimating foreign fat in butter is as follows: 5 grammes of the fat at as low a temperature as it will keep fluid are weighed into a flask, and 2 cc. 50 per cent. NaHO and 30 cc. rectified spirit are then added and the flask attached to a reflux condenser. The flask is then heated over a water-bath, and the contents allowed to boil briskly for twenty minutes. The flask is then detached from the condenser and the alcohol boiled off; the last traces are removed by gentle blowing with the bellows; 100 cc. of hot water are then added to the flask and shaken until the soap is entirely dissolved. 40 cc. dilute sulphuric acid (40 grammes to the litre) is then run in together with a small piece of pumice-stone to prevent explosive boiling. The flask is quickly attached to an ordinary condenser, and heated with a naked Bunsen flame until 110 cc. have distilled over; the distillation should last thirty minutes. The distillate is mixed and filtered. 100 cc. is then titrated with $\frac{N}{10}$ soda or baryta, using phenolphthalein as indicator. As 110 cc. was distilled and only 100 cc. titrated, we have to add $\frac{1}{10}$ to the number of cc. of $\frac{N}{10}$ alkali required.

Leffmann and Beam have published a process wherein glycerine is used instead of alcohol to perform the saponification. This process is much quicker than the alcohol method, with which it gives concordant results. Special flasks with a bulb blown in the neck, which can be placed on the fine balance, can be obtained, which are very much more convenient than using a flask into which the fat has to be weighed by subtraction. Genuine butter-fat requires from 24 cc. to 32 cc. $\frac{N}{10}$ alkali for neutralization.

Margarine-fat and the vegetable oils, when tested by

this process, give distillates which only require from 0·2 to 1·0 cc. $\frac{N}{10}$ alkali.

From the above data we can calculate approximately the amount of butter-fat in a mixture. Many chemists use half the above quantities; that is, they work on 2·5 grammes of fat, and obtain a distillate of 50 cc., which is titrated direct without filtering. Hence, in speaking of the Reichert figure, it is important to know which procedure has been employed. Of course, the amount of alkali required by the distillates in this case is only about half that in which 5 grammes of fat were saponified.

The Valenta Acetic Acid Test.—This test depends on the intermiscibility of butter-fat and strong acetic acid at a low temperature, whereas animal and vegetable fats do not form a clear mixture, except at a much higher temperature. The acetic acid used must be about 99 per cent. This will give a turbidity with genuine butter-fat at from 32° to 36° C. It is best to set the acid against a sample of butter-fat of known purity rather than by titration.

This test is carried out as follows: A test-tube is graduated by running two quantities of 3 cc. of water from a burette or pipette; file scratches are made exactly opposite the dark line forming the meniscus. The tube is then dried, and 3 cc. each of the fat and acetic acid poured into the tube. The mixture is warmed to 40° C., when, if the sample is butter-fat, the contents of the tube will become clear, whereas margarine will not dissolve in the acid at a lower temperature than 75° C. Mixtures of butter and margarine will of course require intermediate temperatures. In practice we do not note the temperature at which solution takes place, but the point at which turbidity (the reverse of solution) begins to appear on removing the source of heat. This is done by stirring the mixture, previously heated till clear, with a thermometer, and noting the temperature as soon as the point of turbidity is reached. It makes its first appearance as a tail following the thermometer bulb. (See a paper on 'The Acetic Acid Test,' by the authors, *Analyst*, July, 1894.)

The Determination of the 'Soluble' and 'Insoluble' Fatty Acids (Hehner's Method).—This method was

first devised by Angell and Hehner, but the original process has been modified and improved by Allen, Muter, and others.

The following procedure is recommended as the most satisfactory by A. H. Allen in his 'Commercial Organic Analysis' (vol. ii.).

Before commencing the operation, the following standard solutions must be prepared :

(*a*) Dissolve 14 grammes of good stick-potash in 500 cc. of rectified spirit, or methylated spirit which has been redistilled with caustic alkali, and allow the liquid to stand till clear. This solution will be approximately seminormal.

(*b*) A standard hydrochloric or sulphuric acid of approximately seminormal strength.

(*c*) Accurately prepared decinormal caustic soda. Each 1·0 cc. contains ·0040 gramme of NaHO, and neutralizes ·0088 gramme of butyric acid, $C_4H_8O_2$.

A quantity of the butter-fat is melted in a small beaker, a small glass rod introduced, and the whole allowed to cool and then weighed. It is remelted, stirred thoroughly, and about 5 grammes poured into a strong 6-oz. bottle. The exact weight of fat taken is ascertained by reweighing the beaker containing the residual fat.

By means of a fast-delivering pipette, 50 cc. measure of the alcoholic potash (solution *a*) is run into the bottle, and the pipette drained exactly thirty seconds. At the same time another quantity of 50 cc. is measured off in an exactly similar manner into an empty flask.

The bottle is fitted with an indiarubber stopper, which is tightly wired down, and is placed in the water-oven, and from time to time removed and agitated, avoiding contact between the liquid and the stopper. In about half an hour the liquid will appear perfectly homogeneous, and when this is the case the saponification is complete, and the bottle may be removed. When sufficiently cool, the stopper is removed, and the contents of the bottle rinsed with boiling water into a flask of about 250 cc. capacity, which is placed over a steam-bath, together with the flask containing merely alcoholic potash, until the alcohol has evaporated.

Into each of the two flasks is now run about 1 cc. more seminormal acid (solution *b*) than is required to neutralize

the potash, and the quantity used accurately noted. The flask containing the decomposed butter-fat is nearly filled with boiling water, a cork with a long upright tube fitted to it, and the whole is allowed to stand on the water-bath until the separated fatty acids form a clear stratum on the surface of the liquid. When this occurs, the flask and its contents are allowed to become perfectly cold.

Meanwhile, the blank experiment is completed by carefully titrating the contents of the flask with the decinormal soda, a few drops of an alcoholic solution of phenolphthalein being added to indicate the point of neutrality.

The fatty acids having quite solidified, the resultant cake is detached by gently agitating the flask, so as to allow the liquid to be poured out, but avoiding fracture of the cake. The liquid is passed through a filter to catch any flakes of fatty acid, and is collected in a capacious flask. If any genuine butter be contained in the sample, the filtrate will have a marked odour of butyric acid, especially on warming.

Boiling water is next poured into the flask containing the fatty acids, a cork and long glass tube attached, and the liquid cautiously heated till it begins to boil, when the flask is removed and strongly agitated till the melted fatty acids form a sort of emulsion with the water. When the fatty acids have again separated as an oily layer, the contents of the flask should be thoroughly cooled, the cake of fatty acids detached, and the liquid filtered as before. This process of alternate washing in the flask by agitation with boiling water, followed by cooling and filtration of the wash-water, is repeated three times, the washings being added to the first filtrate. It is often difficult or impossible to obtain the wash-water wholly free from acid reaction, but when the operation is judged to be complete, the washings may be collected separately and titrated with decinormal soda. If the measure of this solution required for neutralization does not exceed 0·2 cc., further washing of the fatty acids is unnecessary.

The mixed washings and filtrate are next made up to 1,000 cc., or some other definite measure, and an aliquot part carefully titrated with decinormal soda (solution *c*). The volume required is calculated to the whole liquid. The number so obtained represents the measure of deci-

normal soda neutralized by the soluble fatty acids of the butter-fat taken, *plus* that corresponding to the excess of standard acid used. The latter will have been previously ascertained by the blank experiment. The amount of soda employed in this is deducted from the total amount required by the butter-fat quantity, and the difference is the number of cubic centimetres of standard soda corresponding to the soluble fatty acids. This volume multiplied by the factor 0·0088 gives the butyric acid in the weight of butter-fat employed. Thus, suppose an experiment to have given the following figures: Weight of butter-fat taken, 5·120 grammes; decinormal soda required in the blank experiment, 3·90 cc.; decinormal soda required to neutralize one-fifth of the solution of the soluble fatty acids, 6·25 cc. Then,

$$\frac{\cdot0088 \times (31\cdot25 - 3\cdot9) \times 100}{5\cdot120} = 4\cdot70 \text{ per cent.}$$

The flask containing the cake of insoluble fatty acids is thoroughly drained, and then placed on the water-bath to melt the contents, which are poured as completely as possible into the (wet) filter through which the aqueous liquid was previously passed. The fatty acids are washed on the filter with boiling water, to remove the last traces of sparingly soluble acids. The funnel, with the filter containing the fatty acids, is then placed in a small beaker and put into the water-bath until all the fatty acid that will has run through.

The flask, funnel, and filter-paper are well washed with ether, and the washings evaporated in a flask to obtain the last traces of fatty acids. The beaker containing the bulk of the insoluble fatty acids, together with the smaller quantity recovered from the ether washings, are dried to constant weight at 100° C. and the two weights added; the sum gives the weight of the insoluble fatty acids contained in the weight of butter-fat taken.

The soluble fatty acids, calculated as butyric acid, should amount to *at least* 5 per cent., any notably smaller proportion being due to adulteration. The insoluble fatty acids from genuine butter-fat rarely exceed 88½ per cent., though occasionally reaching 89 per cent., but a sample ought scarcely to be regarded as certainly adulterated unless the insoluble acids exceed 89½ per cent. As a standard for

calculation, 88 per cent. of insoluble acids may be regarded as a fair average, the soluble acids being taken at $5\frac{1}{2}$ per cent.

According to J. Bell, the proportion of soluble acids, calculated as butyric acid, not unfrequently falls as low as 4·5, and the percentage of insoluble acids sometimes slightly exceeds 89·0 per cent.

The percentage of adulterant in a butter-fat may be calculated from the following formula, in which F is the percentage of foreign fat, and I that of the insoluble fatty acids:

$$F = (I - 88) \times 13{\cdot}3$$

Or each 0·1 per cent. of soluble acids above 0·5 may be regarded as showing the presence of 2 per cent. of butter-fat.

The Specific Gravity of Butter-fat.—The method of taking the specific gravity at the ordinary temperature is now but seldom employed, it being more convenient to take the density at temperatures at which the fat is in a molten condition. The temperatures that give the best results, and those that are generally employed, are 37·8° C. (=100° F.) and 99° to 100° C.

Dr. James Bell prefers the former temperature. The fat at about 110° F. is poured into an ordinary specific gravity bottle, which is allowed to stand in water at exactly 100° F. for a few minutes. The stopper is then pushed well home, the bottle wiped dry, cooled, and weighed. The weight is compared with water as 1,000 at the same temperature. As the result of the examination of a great number of samples, pure butter-fat is found to range from 910·7 to 913·5, the greater number falling between 911 and 913. Margarine examined under the same conditions ranges from 901·5 to 906·0.

Allen, Estcourt, and others, take the specific gravity of butter-fat at the temperature of boiling water (98° to 100° C.), comparing it with water at 15·5° C. as unity.

This is best done with the Sprengel tube. The tube is filled with the melted fat by sucking the contracted end of the tube, the wider end being immersed in the fat. The tube is then placed in water in a state of rapid ebullition, contained in a beaker of such a size that the capillary ends are only just free of the boiling water. When the ex-

pansion ceases, the tube is set to the mark by the application of filter-paper to the capillary orifice. The tube is then withdrawn, dried, cooled, and weighed. The weight of the Sprengel tube and the weight of the water contained in it at 15·5° C. being known, the weight of fat contained at 99° C. divided by the weight of water at 15·5° C. will give the density of the fat at the temperature of boiling water.

The following are the limits for butter-fat and margarine at a temperature of about 99·5° C. compared with water at 15·5° C. taken as 1000 :

Butter-fat	865·3 to 866·8
Margarine	856·0 to 860·0

The above method may be replaced by the Westphal balance, as recommended by Estcourt. The oil or fat is contained in a wide test-tube, which is immersed in boiling water ; the tube is arranged with a collar, or some other device, to protect the balance from the steam. The plummet is then dipped into the fat, and the specific gravity found as soon as the maximum temperature is reached.

The best form of apparatus with which to carry out this method is that devised by Charles Estcourt, a full account of which will be found in Allen's ' Commercial Organic Analysis ' (vol. ii., p. 16).

The **Koettstorfer Saponification Equivalent** of butter-fat varies from 242 to 253, the mean figure for margarine being about 284. For the method of determining this figure, see under ' Oils and Fats.'

The **Iodine Absorption** of butter-fat ranges from 23 to 38 per cent., margarine giving from 40 to 55 per cent. of iodine absorbed. These results, though interesting in themselves, are not of much value when determining the amount of foreign fat in butter. For the method of determining the iodine absorption, see under ' Oils and Fats.'

The **Oleorefractometer** is used by several workers to ' sort ' samples of butter, but in our opinion it is not to be compared for this purpose with the easily performed Valenta test. For the description of this apparatus, see under ' Oils and Fats.'

As the result of the examination of several hundred of samples examined in this instrument, pure butter-fat

gives a deflection of from $-35°$ to $-23°$, margarine deflecting from $-18°$ to $-10°$.

Microscopical Examination of Butter.—Mr. A. W. Stokes uses the microscope for sorting the samples of butter. He cuts a piece off the sample to obtain a clean surface ; this surface is *lightly* scraped all along so as to get as representative a portion as possible. The scrapings should not measure more than about one-tenth of the size of an ordinary pea. This is transferred to a small microscopical cover-glass, which is then pressed down on to an ordinary microscopical slide, so that the butter lies in the form of a wedge between the two glasses, one side being pressed very thin, while the opposite side is left very thick. The slide thus prepared is placed under the 1-inch power of the microscope; the microscope must be provided with a good polarizing apparatus. The thinnest edge of the preparation should be so arranged that it cuts across the middle of the field of view, one-half of the field being left blank. Gas-light should be used.

Now, on rotating the Nicol's prisms, so that the two prisms are at right angles, the whole of the field will remain equally dark if the sample be pure butter. If, however, as much as 20 per cent. of margarine be present in the sample, it will be impossible, however the prisms may be placed, to darken the side of the field occupied by the sample. Margarine gives, when thus viewed, a similar appearance to that of a cloud in a dark sky.

No selenite plate should be used. The butter should not be melted, and since most of the adulterated samples of butter contain over 20 per cent. of margarine, this microscopical method will rarely pass an adulterated sample. After an experience on many thousands of samples during the past twenty years, Mr. Stokes still believes in the efficiency of this simple and rapid method in the hands of a competent microscopist.

Butter has occasionally been adulterated with sesame oil, which is readily detected by the use of Baudouin's test, which is as follows : Dissolve 0·1 gramme of cane-sugar in 10 cc. of hydrochloric acid of specific gravity 1·2; add to this 20 cc. of the oil to be tested ; shake thoroughly, and allow to stand. In the presence of even 2 per cent. of sesame oil the aqueous liquid will become of a crimson colour.

The addition of hydrochloric acid alone to butter-fat sometimes produces a pink colour owing to the presence of a tar-dye, so that care must be taken not to put down to the presence of sesame oil a colour due to a dye.

It occasionally happens that a butter is found which gives a positive reaction with the silver test for cotton-seed oil ; but if this reaction is obtained, it must not be taken for granted that cotton oil has been added to the butter, as we found by experiment that when a cow, whose butter normally gave no reaction with the silver test, had half a pound of cotton-seed cake added to her daily food, the butter at once gave an unmistakable darkening with the silver reagent.

Colouring Matter in Butter.—Artificial colouring matters are very frequently added to butter, among which may be mentioned the following : annatto, turmeric, saffron, saffronette, marigold, and the azo-dyes, the last being of somewhat recent introduction for this purpose. The addition of artificial colouring to butter is not regarded as an adulteration in this country. It is worth noting that the butter from some Jersey cows often has a very deep yellow colour.

If the colouring matter of a butter can be extracted with alcohol, foreign colouring is undoubtedly present, as the natural colouring matter is not soluble in alcohol.

Butter-fat, on exposure to light and air, loses its yellow colour, and acquires the smell and colour of tallow.

Preservatives in Butter.—Boric and salicylic acids are sometimes added to butter. We have found boric acid recently in a large number of samples, especially in those coming from abroad.

Boric Acid is detected in the ash, as given under 'Milk.'

Salicylic Acid may be estimated by the method used in the Paris Municipal Laboratory : 20 grammes of the sample are repeatedly exhausted by a solution of bicarbonate of soda, which converts the salicylic acid into the soluble sodium salicylate. The aqueous liquid is acidulated with dilute H_2SO_4, and extracted with ether ; on the addition of a little mercurous nitrate to the residue after evaporating off the ether, a precipitate nearly insoluble in water is obtained. This is filtered off, washed, and decomposed by dilute H_2SO_4, free salicylic acid again resulting. It is redissolved in ether, the solvent evaporated off,

and the residue warmed from 80° to 100° C. until nearly dry. In order to remove any other acid present, the residue is extracted with neutral petroleum ether; the ethereal solution is diluted with an equal volume of 95 per cent. alcohol, and titrated with $\frac{N}{10}$ alkali, using phenolphthalein as indicator (1 cc. of $\frac{N}{10}$ NaHO=0·0138 of salicylic acid).

For further identification, the salicylic acid may be liberated again with a corresponding amount of $\frac{N}{10}$ HCl, and the liquid tested with a drop of Fe_2Cl_6 solution, when a violet coloration should be obtained.

CHEESE.

The manufacture of cheese by empirical methods has been carried on from the earliest times, and was a means of storing and preserving milk many centuries before any other method was devised.

Cheese is made from the milk of the cow, goat, and sheep, though cow's milk alone is used for cheese-making in this country. Cheese is made from whole milk, skimmed, or separated milk, and from milk enriched with cream.

Cheeses may be classified in various ways, as for example—

1. Whether hard or soft.
2. According to the amount of fat in the raw material —*i.e.*, whether made from

(*a*) Cream;
(*b*) Cream and milk;
(*c*) Whole milk;
(*d*) Partly-skimmed milk;
(*e*) Skim milk.

The manufacture of the various kinds of cheese is a matter that requires great skill and experience, owing to the fact that so many conditions are necessary for success. For practical details respecting the complicated processes involved in cheese-making we would refer to our work on 'The Analysis of Milk and Milk Products.'

American Cheese.—This is a Cheddar cheese, and usually contains an excellent amount of fat, averaging about 33·0 per cent.

According to W. Fleischmann (translated by Aikman and Wright, 1896), much cheese has lately been made in

America by the American Cheddar process from milk partly deprived of its fat. This may be the case ; but among about sixty samples of American cheese bought during the past two years, we have not found any that has shown much below 30 per cent. of fat.

In the preparation of American Cheddar, the procedure in use in making Cheddar cheese in England is followed with but slight variations.

Camembert.—Camembert is made in France, and imported into this country in considerable quantities. It is usually kept two months before being eaten.

Cheddar.—Cheddar is the typical cheese of English manufacture, and its composition, and the conditions under which the greatest success is attainable, have formed the subject of many researches. Of recent work on this point, we may mention that of F. J. Lloyd, particularly his report for 1895, entitled, 'Observations on Cheddar-Cheese Making,' in the *Bath and West of England Society's Journal.*

Cream Cheese.—Cream cheese may be made either by coagulating the cream with rennet, or by allowing it to sour naturally ; in either case it should contain about 75 per cent. of butter-fat.

Dutch Cheese.—This cheese is prepared from milk that has been partly deprived of its fat.

Gloucester Cheese.—Two varieties of this cheese are made : Single Gloucester and Double Gloucester. The distinction between these is one of size only, and has no reference whatever to quality.

Gorgonzola.—Gorgonzola when ripe is permeated by moulds. The spores of the moulds are introduced by adding, in the process of manufacture, powdered bread crusts on which moulds have been allowed to grow.

Gruyère.—The term Gruyère is applied to two kinds of hard Swiss cheese, which are prepared from cow's milk. Much of the cheese sold in England as Gruyère is known as Emmenthaler in Switzerland.

Parmesan Cheese.—Parmesan cheese is prepared from partly-skimmed goat's milk.

Roquefort Cheese. — Roquefort cheese is prepared from partly-skimmed ewe's milk.

Skim-Milk Cheese.—Skim-milk cheese is prepared either by natural souring or by the use of rennet.

Stilton.—Stilton cheese is prepared in this country, in

America, and on the Continent. It is prepared from a mixture of whole milk and cream, or from whole milk alone.

The following table of analyses of typical cheeses is taken from a paper by the authors (*Analyst*, July, 1894).

Analyses of Various Commercial Cheeses.

	Name of Sample.	Water.	Ash.	Fat.	Reichert cc. $\frac{N}{10}$	Nitrogen.	Casein.
1	Cheddar ...	33·0	4·3	29·5	24·2	4·31	27·4
2	Cheddar ...	35·5	4·2	25·6	28·8	4·39	27·8
3	Cheddar ...	33·8	4·1	30·5	26·4	4·20	26·7
4	Cheddar ...	33·3	3·6	30·6	24·0	4·34	27·6
5	American ...	29·8	3·7	33·9	26·2	4·76	30·3
6	American ...	30·6	3·6	27·7	3·0	4·84	30·8
7	American ...	29·1	3·7	35·3	23·0	4·41	28·1
8	American ...	24·1	3·9	32·0	25·8	—	—
9	American ...	27·0	4·5	30·1	24·8	—	—
10	American ...	25·0	7·9	20·1	30·4	—	—
11	American ...	27·2	4·4	30·9	25·4	—	—
12	American ...	28·1	4·5	33·0	25·6	—	—
13	Gorgonzola ...	40·3	5·3	26·1	22·1	4·36	27·7
14	Gorgonzola ...	33·9	4·6	26·7	23·6	4·06	25·8
15	Dutch	41·8	6·3	10·6	27·0	5·11	32·5
16	Dutch	37·6	6·5	22·5	23·0	4·58	29·1
17	Gruyère ...	28·2	4·7	28·6	30·0	4·93	31·3
18	Gruyère ...	35·7	3·7	31·8	31·1	4·49	28·7
19	Stilton	19·4	2·6	42·2	29·0	4·73	21·1
20	Stilton	21·2	2·9	45·8	32·0	4·14	26·3
21	Cheshire ...	37·8	4·2	31·3	31·6	4·03	25·7
22	Cheshire ...	31·6	4·4	35·3	31·8	4·16	26·5
23	Gloucester ...	33·1	5·0	23·5	31·4	4·99	31·8
24	Gloucester ...	37·4	4·6	28·1	32·3	4·45	28·3
25	Camembert ...	47·9	4·7	41·9	31·0	3·43	21·8
26	Camembert ...	43·4	3·8	22·6	35·0	3·83	24·4
27	Parmesan ...	32·5	6·2	17·1	28·0	6·86	43·6
28	Roquefort ...	29·6	6·7	30·3	36·8	4·45	28·3
29	Double Cream ...	57·6	3·4	39·3	31·2	3·14	19·0
30	Bondon	39·5	0·7	24·4	29·4	1·48	9·4
31	Cream (York) ...	63·1	1·4	6·5	29·0	2·76	17·9

Particular attention is called to No. 6 American cheese.

This is a margarine cheese, and was bought by the vendor as genuine cheese.

The following figures call for notice, viz. : Water in the Camemberts, Nos. 25 and 26, which contain 48 and 43 per cent. respectively; the York cream, 63 per cent. ; the double cream, 57 per cent. Fat is very deficient in the following : York cream, 6·5 ; Parmesan, 17·1 ; and the Dutch No. 1, 10·6. The nitrogen is very low in the case of the Bondon cream, namely, 1·48 ; in the York cream it is 2·6 ; and in the Parmesan it reaches the highest amount of 6·86 per cent.

From the above figures we would conclude that none of the cheeses (excepting the Dutch and so-called cream cheese) have been prepared from milk from which fat had been removed.

In consequence of the variations exhibited in the amount of fat, a further number of samples of Dutch cheese were examined by one of us, which gave the following amounts of fat (February, 1897—C. G. Moor) :

Sample.	Fat, per cent.	Valenta test.	Price per lb.
1	15·5	23·0 °C.	6d.
2	10·7	24·0	6d.
3	15·5	24·0	6d.
4	16·2	26·0	6d.
5	28·4	27·0	6d.
6	26·1	30·0	8d.
7	14·6	30·0	7½d.
8	21·0	30·0	7½d.
9	15·0	30·0	6½d.

The following figures, which may be of interest, were obtained by us on some imitations of foreign cheeses which were made in England under the auspices of the British Dairy Farmers' Association :

	Fat.	Water.	Ash.	Nitrogen.	Proteids. N×6·3.
Port de Salut ...	36·2	31·3	4·6	4·2	26·5
Caerphilly ...	30·4	24·8	3·4	5·9	37·2
Culommier ...	24·1	37·8	4·1	3·9	24·6
Gorgonzola ...	33·2	33·5	3·5	6·0	37·8
Camembert ...	33·2	35·0	2·9	5·5	34·6
Gervais ...	69·3	15·8	0·6	3·0	18·9

We found that in flavour they compared favourably with the foreign product.

Adulteration of Cheese.—The only adulteration of cheese at the present day is the substitution of foreign fats for the true butter-fat, such cheeses being known as margarine, or 'filled' cheeses. They are prepared as follows: Skim milk is churned up with an emulsion of clarified animal fats (margarine), and the mixture is curdled, pressed, and treated in the ordinary way. The sale of this product as cheese, without any qualifying description, is an offence under the Food and Drugs Act, and convictions have been from time to time obtained, so that very little margarine cheese is to be found in this country.

Margarine cheese is prepared at Dunragit, N.B., at Hamburg, in Holland, and in America. Its sale and its preparation are both prohibited in Canada, New York State, Pennsylvania, Massachusetts, and Wisconsin.

The smell of margarine cheese is peculiar, and its detection analytically is so exceedingly easy and certain that there is little chance of its ever being sold in England, except on rare occasions. The methods for its detection are given under 'The Analysis of Cheese.'

In addition to the sample of margarine cheese examined by us (No. 6 in the table), R. Bodmer, in July and September, 1895, examined two samples of cheese purchased under the Food and Drugs Act, which proved to be margarine cheeses. They contained 42·4 and 31·7 per cent. of fat respectively, and the Reichert figure obtained on the fat showed them to be almost entirely devoid of butter-fat. The samples were sold as American cheeses, and in each case the vendors were convicted and fined.

It is said that starch is occasionally added, but we have never found it in any of the samples we have examined.

Standard for Cheese.—There is no standard for cheese in this country, and consequently a skimmed-milk cheese containing only traces of fat may be sold as cheese without any qualification, while we are confronted with the anomaly that, if margarine-fat is added, it is an offence to sell the resulting product as cheese unless its character is declared.

It is therefore evident that a standard for cheese should

be adopted, and the following would be a fair one, namely: not less than 30 per cent. of true butter-fat, while no starch or other extraneous matter should be present. Any cheese that does not come up this standard should be required to be plainly marked and sold as 'prepared from milk from which a portion of the fat has been removed.'

At present we may purchase as 'cream cheese' what is not even a milk cheese, and if cream cheese is really desired it must be asked for as 'double cream,' or under some fancy name.

In 1885, a special New York State brand was adopted for 'pure cream cheese,' which is said to have accomplished much in restricting the sale of the spurious article.

Wynter Blyth ('Foods and their Adulterations') says: 'A cheese which shows under 10 per cent. of fat may with propriety be called skim,' and with this we heartily agree.

The Analysis of Cheese.—Water is estimated by drying 5 grammes of the sample in thin slices at a temperature of 105° C. till constant in weight.

The ash is estimated on the above by igniting at as low a temperature as possible. It will vary very largely (without taking into consideration the added salt) according to the amount of acidity that was permitted in the process of manufacture; the higher the acidity, the more calcium salts will have been dissolved and run off with the whey.

The proteids are obtained by multiplying the nitrogen figure by the factor 6·3.

Fat.—Many methods may be employed to estimate the fat in cheese. After having given some considerable attention to the matter, we prefer to use one of the two following methods:

Ether Extraction Process.—Fifty grammes of the cheese are ground up in a mortar with a fairly large quantity of sand. The powder so obtained is placed in a tall stoppered cylinder, and extracted by means of four successive portions of ether, using in all about 500 cc. The ether-washings are then made up to a definite volume and an aliquot portion taken; the ether is evaporated, and the residual fat weighed in the usual way.

Mechanical Process.—When it is merely necessary to estimate the fat, it can be quickly and accurately determined by means of the following modification of the Leffmann-Beam process for milk :

Two grammes of the cheese are taken and reduced to as fine a state of division as possible ; this is then transferred to a small dish and treated on the water-bath with 30 cc. concentrated hydrochloric acid until it is dissolved and the solution is of a dark purplish colour. The mixture is then poured into a Leffmann-Beam bottle, and the dish rinsed with 3 cc. of the HCl-fusel-oil mixture into the bottle, and, finally, enough hot strong HCl is added to fill the bottle to about the zero mark. It is then centrifugated for one minute.

The Leffmann-Beam bottles are graduated so that ten divisions equal 1·0 per cent. by weight on 15·55 grammes (=15 cc.) of milk.

It follows, therefore, that the factor, in order to make use of the bottle for cheese, will be :

$$\frac{15{\cdot}55}{2} = 7{\cdot}77.$$

With very little practice concordant readings are easily obtained, which agree with the ether extraction process already explained.

To obtain fat on which to do further work, it is generally sufficient to chop up about 50 grammes of the sample, which are hung up in a muslin bag in the water-bath ; the fat will generally run out clear. The fat from the remainder of the ether in the process already given can also be obtained by evaporation.

The fat should be examined by means of the Reichert and Valenta processes, as described under 'Butter,' to prove that it is true milk-fat.

Starch should be tested for by taking a portion of the fat-free residue, boiling with water, filtering, and testing the filtrate for starch, by means of a dilute solution of iodine in potassium iodide. No blue coloration should be obtained.

WHEAT-FLOUR AND STARCHES.

Wheat-flour should not contain more than 20 per cent. of moisture, and should be free from grit and alum. Such adulterations as oat, maize, rice, and potato starches have been found, but they are very rare, on account of their easy detection under the microscope. Wheat-starch has two sizes of granules, one much larger than the other, both being circular.

Flour contains varying amounts of gluten, from about 12 per cent. to 18 per cent.; the gluten is estimated by kneading dough produced from a known quantity of flour with water till all the starch is washed out. The remaining stringy mass is dried and weighed.

In flour from which the husk has been separated there is little or no 'indigestible fibre.'

Jago's doughing test is performed thus: 50 grammes of the sample are placed in a dish, and stirred with a rod, water being run in from a burette till a dough of ordinary consistence is obtained.

This may appear to be a somewhat indefinite point, but in practice it is surprising what concordant results can be obtained. The point at which a dough of 'ordinary consistence' is obtained depends on two things: first, the amount of water present in the flour; and secondly, the amount of gluten in the sample. The main object of the test is to ascertain the number of loaves which can be prepared from a sack of the flour in question.

Old and damaged flour has been ground up with fresh flour, and the resulting mixture is very liable to go bad.

Such mixtures may be detected by their notable amounts of acid, while good flour is neutral, or nearly so.

Microscopical Characters of Starches—1. *Wheat.*—This is characterized by having two sizes of granules, both of which are circular, the one large and the other small, while but few intermediate sizes are to be seen.

2. *Barley.*—The starch of barley is very similar to that of wheat, having two sizes of granules, both of which are circular, but the small ones in barley are smaller than the corresponding ones in wheat.

3. *Rye.*—This starch is similar to the foregoing, but in

addition to the large and small granules, a few intermediate-sized ones are also to be seen.

4. *Maize.*—This starch consists of large irregular faceted granules, all much the same size ; they generally exhibit a hilum in the shape of a star.

5. *Oat.*—The starch granules in the case of oats are also faceted and irregular. They are considerably smaller than those of maize, and are frequently to be seen united in a spherical formation.

6. *Rice.*—This starch consists of small faceted granules, smaller than those of oats, and all much of the same size.

7. *Potato.*—In this starch the granules vary much in size, from very large down to very small ; they generally show concentric marks and a circular hilum. The only starch likely to be confused with a potato starch is the *Canna Indica*, or arrowroot.

8. *Arrowroot* (Bermuda). — Small, irregularly oval-shaped granules, which do not exhibit much marking.

9. *Arrowroot* (*Canna Indica*).—Large, irregular, oyster-shaped granules not unlike those of potato.

10. *Tapioca.* — This starch consists of small circular granules, which generally exhibit a well-marked hilum, and are frequently bell-shaped.

11. *Pea.*—Pea-starch consists of large oval granules, rather irregular in shape ; it can hardly be distinguished under the microscope from the starch of the bean, haricot, or lentil.

12. *Sago.*—Consists of large, irregular-shaped oval granules.

The starches of wheat, maize, and rice are official in the British Pharmacopœia, 1898.

To examine starches under the microscope, the specimen should be mixed with a little cold water ; a drop should then be placed on the slide, and the cover-slip lowered on to it. It should be examined, first with a $\frac{2}{3}$-inch, then with a $\frac{1}{6}$-inch, objective—the first to get a general view of the field, the second to examine individual granules. Fairly permanent preparations of the starches may be made by setting them up in a drop of warm glycerine jelly, and ringing the slide in the ordinary way with Japan black. Such preparations have retained their characteristic features for several years unimpaired. We

give below the average percentage composition of some of the farinaceous materials commonly used as food :

	Water.	Proteids.	Fats.	Carbo-hydrates.	Ash.
Arrowroot ...	15·40	0·80	—	83·5	0·30
Barley meal .	11·30	12·70	2·00	71·00	3·00
Flour ...	16·50	13·00	1·50	68·30	0·70
Maize ...	13·50	10·00	6·70	64·50	1·40
Oatmeal ...	15·00	13·00	6·00	63·00	3·00
Peas	15·60	22·60	2·00	58·00	2·40
Rice	10·00	5·00	0·10	84·40	0·50

BREAD.

Bread is made by kneading wheat-flour with water, the coherence of the dough being due to the moistened gluten. The porosity of bread, which is essential to its easy digestion, is produced by enclosing in the dough minute bubbles of carbonic acid gas. This is accomplished in one of three ways :

1. By the use of yeast, which sets up fermentation of a small portion of the starch, forming alcohol and carbonic acid gas.

2. By the use of baking-powders containing an acid salt and a bicarbonate, which on being moistened give off carbonic acid.

3. By kneading the dough with water charged with carbonic acid gas under pressure (Dauglish's system).

Bread is very rarely adulterated, and any adulteration in the shape of foreign starches would be difficult to detect, as in the baking the starch-granules are ruptured and lose their shape.

We have also been informed on good authority that it is the practice of many bakers at the present time to add a small quantity of boiled potatoes to their dough.

This addition is said to render the bread white, and does not appear to be made with any fraudulent intent, such as making the bread hold more water, and the quantity of potato added (about 1 per cent.) is not sufficient to effect any economy in flour.

In sampling bread for analysis, a loaf should be divided

into three parts as usual, and each portion should be sealed up in a tin after wrapping in oil-paper ; this precaution must be taken, or evaporation will stultify any subsequent work.

Alum is the only mineral adulterant of bread, although its use has now almost entirely ceased. The action of alum in increasing the whiteness and apparent quality of inferior flour is undoubted. Alum can readily be detected in bread and flour by means of the logwood test, which will with care readily detect as little as 7 grains of alum in a 4-lb. loaf.

Logwood Test. — For this test a freshly-prepared alcoholic tincture of logwood (made by digesting 5 grammes of fresh chips or raspings in 100 cc. alcohol), and a saturated solution of ammonium carbonate, are required.

Flour is tested as follows : 10 grammes of the sample are made into a paste with 10 cc. of water ; 1 cc. each of the logwood tincture and the ammonium carbonate solution are then added, and the whole well mixed with a glass rod. If the sample is free from alum, the mixture is of a pinkish colour, gradually fading to a dirty brown ; but in the presence of alum the pink colour is changed to a lavender tint or actual blue colour.

Bread is tested as follows : 5 cc. each of the logwood tincture and ammonium carbonate solution are mixed and made up to 100 cc. with water ; this is then poured without delay over a lump of bread free from crust weighing about 10 grammes. After about five minutes, when the bread has soaked up the liquid evenly, the excess of fluid is drained off, and the bread dried in the dish at 100° C. If alum is present, the sample assumes a light violet or blue tint, which becomes more marked on drying.

With bread free from alum the brownish tint first obtained fades to a buff or light-brown colour.

Sour bread may give the logwood reaction, and therefore the results must be accepted with caution. If alum is indicated by the logwood test, an estimation of alumina and sulphates will be necessary.

The WATER is estimated by drying to constant weight at 102° to 103° C., the same quantity being available for the estimation of the ash.

When determining the amount of water in bread, it must be remembered that the percentage of water in the crust is about half of that in the crumb, and that care must be taken to work on a duly proportioned mixture of crust and crumb, to obtain which a large piece should be chopped up finely.

The Ash of all flours and meals is difficult to obtain on account of the hard cake of carbon that is formed, unless some special means are adopted. A good plan is to moisten the carbonaceous mass with strong nitrate of ammonia solution, then dry and carefully ignite. After about two treatments of this kind, a clean ash is readily obtained.

The dish in which the ash is being obtained may with advantage be covered with a strip of platinum.

The most obstinate substances—oatmeal, for example—are speedily reduced to ash by the application of a gentle stream of oxygen, which is conveniently applied by leading it under the lid of the dish by means of a piece of tobacco-pipe stem.

The ash of flour and of bread should not exceed 1·5 per cent.

Determination of Silica and of Added Alum.—The same process as directed for the estimation of alum in baking-powders (see p. 70) is employed. 100 grammes of the bread or flour is carefully incinerated with the precautions there mentioned. After the fused mass has been dissolved and evaporated to dryness with hydrochloric acid, and again taken up in acid, the residual silica is washed, dried, ignited, and weighed as SiO_2.

The alumina in the total filtrate is then precipitated in the manner described on p. 70, when the amount of aluminium phosphate obtained multiplied by 3·713 will give the ammonia alum, or by 3·873 the amount of potash alum, in the 100 grammes of sample taken.

The amount of alum found by the above process requires a correction equal to the amount of silica obtained. By multiplying the percentage of alum found by 280, the number of grains of alum per 4-lb. loaf will be obtained. The number of milligrammes of $AlPO_4$ per 100 grammes of bread gives without calculation a close approximation to the number of grains of ammonia alum per 4-lb. loaf (A. H. Allen). It is customary to

subtract 16 grains of alum per 4-lb. loaf from the total amount found to allow for the alumina naturally present.

The following is the average composition of bread made from fine wheat-flour :

Moisture	35·8
Carbo-hydrates...	51·5
Nitrogenous matter	7·0
Ash	1·0
Sugar	4·0
Fat	0·4
Woody fibre	0·3

BAKING-POWDER.

According to a recent unfortunate and illogical legal decision (*Analyst*, xix. 48), baking-powder is neither an article of food nor a drug, and consequently may contain alum or anything else. Hence no action can be taken under the Sale of Food and Drugs Act in the case of a baking-powder containing alum ; but if a baker buys and uses this baking-powder he may be convicted, because his bread is an article of food, even if baking-powder is not.

Thus, there is no protection for ignorant persons who may buy such alumed baking-powders for their private use, though the sale of bread prepared with these powders would be illegal.

Most of the baking-powders at present sold may be divided into three classes, according to the nature of the acid constituent they contain :

1. Tartrated powders, in which the acid constituent is tartaric acid in some form.

2. Phosphate powders, in which the acid constituent is phosphoric acid.

3. Alum powders, in which the acid constituent is sulphuric acid contained in some form of alum salt.

Most of the baking-powders at present on the market fall under one of the above headings, although there are several which are mixtures of at least two of the above classes. All the above have fillings composed of flour or some form of starch.

In America baking-powders are sold containing other ingredients, such as ammonium carbonate, superphosphate of calcium, and bisulphate of potassium, but we have not met with any powders of this nature in this country.

The best and most satisfactory baking-powder is a mixture of cream of tartar with a proper proportion of bicarbonate of soda mixed with a filling of about 20 per cent. of pure starch.

Carbonate of ammonium is unobjectionable as a constituent of baking-powder ; so also is superphosphate of calcium if free from calcium sulphate, and practically free from neutral calcium phosphate.

Alum and bisulphate of potash are absolutely objectionable as constituents of baking-powders, and certainly should not be permitted to be sold. Not only are the residual sulphates powerful purgatives which are certain to interfere with the normal digestive processes when habitually taken into the body, but the free alumina liberated by the reaction of the alum powders is liable to render insoluble the phosphates naturally present in the food, thus making them unavailable for nutrition.

In considering the relative values of different kinds of baking-powers, it is necessary to bear in mind some standard by which to judge the others. The ideal baking-powder giving the maximum gas-producing power is indicated by the following formula :

$$\underset{188}{KHC_4H_4O_6} + \underset{84}{NaHCO_3} = \underset{210}{KNaC_4H_4O_6} + \underset{18}{H_2O} + \underset{44}{CO_2}.$$

That is to say, 1 gramme of a powder in the above proportions would yield approximately 81 cc. of gas, at N.T.P., equivalent to 16 per cent. by weight of CO_2.

The Analysis of Baking-powder.—The qualitative examination of a baking-powder presents no particular difficulty. Microscopical examination of the powder direct, or, better, of the insoluble residue in cold water, will determine the nature of the filling used. This is generally a matter of minor importance.

The full quantitative analysis of a baking-powder is a matter of considerable difficulty, and is seldom required in practice. The following determinations are usually all that is required. A determination of the alkalinity or acidity, as the case may be, of the aqueous solution of

the powder is useful as showing if any great excess of acid or alkaline constituents is present.

Available Carbon Dioxide.—This is the most important estimation, as it determines the strength of the powder. It is made by acting upon the powder with water, and measuring the resulting gas given off. The best form of apparatus for exact determinations is that recommended by Fresenius, in which the gas is absorbed with soda-lime, or one of the forms of the Schrötter type may be used, although they are apt to give results below the truth if not carefully used. Excellent comparative results may also be obtained in the apparatus such as is used in determining the amount of urea in urine (*q.v.*).

The efficiency of a baking-powder may also be estimated roughly by weighing out 1 gramme and placing it in the cup of a nitrometer ; this is corked, and the nitrometer being filled with saturated brine, the tap is opened, and the instrument manipulated so as to wet the baking-powder, which gives off its gas, and after the action is over and the nitrometer levelled, the amount of gas may be roughly ascertained.

Estimation of Tartaric Acid.—5 grammes of the sample are weighed out and washed into a 500 cc. flask with about 100 cc. of water, 15 cc. of concentrated hydrochloric acid is added, and the whole diluted with water up to the mark. The starch and other insoluble matter are allowed to settle, and the supernatant liquid filtered. To 50 cc. of the filtrate (=0·5 gramme original) is added 10 cc. of a 30 per cent. solution of carbonate of potash, and the solution boiled for half an hour; it is then filtered into a dish, and the filtrate and washings evaporated to a bulk of about 10 cc. Add gradually with constant stirring 4 cc. glacial acetic acid, and then 100 cc. of 95 per cent. alcohol, stirring the liquid until the precipitate assumes a crystalline appearance. After the liquid has stood long enough for this precipitate to form and settle, best for several hours, decant through a small filter, add alcohol to the precipitate and bring it on to the filter, wash out the dish, and finally the filter, with alcohol until it is entirely free from acetic acid. Transfer precipitate and filter to a beaker, add water, and boil, washing out the dish with boiling water if any precipitate adheres to it. The resulting liquid is titrated with decinormal alkali, using

phenolphthalein as indicator. Each cc. used=0·0188 gramme of potassium bitartrate, or 0·0150 gramme of tartaric acid.

Phosphoric Acid.—This can be readily estimated by Stock's method as described under 'Infants' Foods.'

Estimation of Aluminium.—5 grammes of the sample is incinerated at a moderate heat so as not to fuse the ash. The process is completed by adding pure sodium carbonate and a little nitre, and heating the mixture to fusion. The fused mass is dissolved in water and rinsed into a beaker, carefully acidulated with hydrochloric acid, and evaporated to dryness. The residue is carefully taken up in dilute hydrochloric acid, diluted with water and made up to 500 cc. The solution is then filtered if necessary; 50 cc. of the clear liquid (=1 gramme sample) is taken, and dilute ammonia added until the precipitate barely redissolves on stirring, when a slightly acid solution of ammonium acetate is added and the solution brought to the boil, and then allowed to stand some hours. The precipitate of aluminium and iron phosphates is filtered off, washed, and redissolved in the smallest possible amount of hydrochloric acid. The resulting solution is poured into an excess of an aqueous solution of pure caustic soda, contained in a platinum dish. After heating for a short time the solution is considerably diluted and filtered. The filtrate is acidulated with hydrochloric acid, ammonium acetate and a little of a solution of sodium phosphate added, and then a slight excess of ammonia. The solution is warmed until all smell of ammonia is lost, when it is filtered, and the precipitated aluminium phosphate is washed, ignited, and weighed. The weight of the precipitate multiplied by 3,713 gives the hydrated ammonia alum, or by 3,873 the potash alum present.

Estimation of Sulphuric Acid.—0·5 to 1 gramme of the sample is boiled with hydrochloric acid until all the powder, including the starch, goes into solution, barium chloride is added in slight excess, and the solution allowed to stand some hours. The barium sulphate is then filtered off, washed, dried, ignited, and weighed.

VINEGAR.

Vinegar has been defined as 'the product of the alcoholic and acetous fermentation of a vegetable juice or infusion.' This definition includes all kinds of brewed vinegar, but excludes wood-vinegar. Brewed vinegar of whatever source will naturally be distinguished from wood-vinegar (acetic acid and water), by containing extractive matters which will remain when the sample is evaporated.

In the case of malt-vinegar, by which we understand vinegar brewed either entirely from malted barley or from a mixture of not less than one-third malt and two-thirds barley, we find the extractive matter to range about 2·5 per cent.

The process of vinegar-making is as follows: The malt or malt and barley (the latter finely ground) are 'mashed,' soaked in successive quantities of hot water till all that is soluble is extracted. The clear liquor is then run off into another vessel, and yeast added. Fermentation then takes place, with evolution of carbonic acid. The 'wort,' or 'wash,' is then pumped over piles of birch-twigs placed in high vats, to which a regulated supply of air is supplied. The twigs become coated with *Mycoderma aceti*, 'vinegar plant,' and the alcohol produced by the fermentation is thereby converted into acetic acid.

Small quantities of other bodies, as acetic ether, aldehyde, etc., are formed, which give malt-vinegar its pleasant taste and smell.

In good working all the alcohol is not converted into vinegar, as a little alcohol improves the flavour and assists the 'keeping' of the finished product, which is generally kept for a year in order that the flavour may fully develop.

Malt-vinegar, whether made from malt or a mixture of malt and barley, yields very characteristic figures on analysis, which distinguish it from glucose vinegar or vinegar brewed from substances other than malt.

Vinegar is sold at different strengths, which are denoted by numbers as follows: 24 the strongest, and 16 (or diamond corresponding to 14) being the weakest. The intermediate strengths are made by adding water to the

strongest. In making the weakest, almost an equal bulk of water is added to the strongest vinegar, and this reduces its sharpness so much that many makers add some distilled vinegar to make it more acid. This may be a convenient method, but it exposes the makers to the danger of their vinegar being accused of consisting partly of acetic acid 'derived from sources other than malt,' as distilled vinegar is undistinguishable from acetic acid.

The lowest-strength vinegar that may be sold contains 3·0 per cent. of acetic acid.

Genuine malt-vinegar varies but very little from the following composition:

Specific gravity at 15·5° C.	=1·019
Acetic acid	=5·50 per cent.
Extract	=2·50 ,,
Phosphorus as P_2O_5	=0·08 ,,
Nitrogen	=0·08 ,,
Ash	=0·50 ,,

If the vinegar in question is one of the lower strengths, of course the other constituents should vary in proportion with the acetic acid.

'Distilled' or 'white' vinegar is largely used in the North of England. It is usually prepared by distilling malt-vinegar; the distillate is generally collected in two receivers: the one nearest the still is the strongest, and is sold as distilled vinegar, while the other is used up as indicated above.

'Wood-vinegar,' so called, is prepared by diluting down acetic acid. This is sometimes coloured with caramel, and sold as malt vinegar. The above is often called in the trade pyroligneous acid; this term, however, properly belongs to the crude wood acid which runs from the condenser when wood is destructively distilled.

Dilute acetic acid is also sold under the name 'white wine vinegar.' In both the above cases the sale is fraudulent.

A small quantity of real 'wine-vinegar' and some 'cider-vinegar' are to be found in commerce, but they are not important enough to warrant description here.

Strong acetic acid, termed 'malt acid,' coloured to imitate malt-vinegar, is sold with directions how to dilute it to prepare a fictitious malt-vinegar.

It is not legal to add any sulphuric acid to vinegar, though this was once permitted under the idea that it was necessary for its preservation. Such addition, however, has been illegal for years. The custom has not quite died out, as several convictions were obtained in the Northern counties recently for this offence.

The addition of sulphuric acid causes the total solids to be black instead of brown.

Analysis of Vinegar.—The analysis of vinegar is performed as follows : The **Specific Gravity** is taken with the Westphal balance or specific gravity bottle. In the case of a strong vinegar, *i.e.*, 5 per cent. acetic acid, this should be about 1·019.

Acetic Acid.—10 cc. should be titrated with $\frac{N}{10}$ soda using phenolphthalein as indicator after well diluting with distilled water, when :

No. of cc. of $\frac{N}{10}$ NaHO × ·006 × 10 = per cent. acetic acid.

The **Total Solids** are determined by evaporating 25 cc. in a tared platinum dish ; this, after drying to constant weight, is ignited at a low temperature to obtain the ash. The alkalinity of the ash can be determined, but this figure is not of much value.

The **Nitrogen** is determined by Kjeldhal's process (see under 'Milk') by evaporating 25 cc. of the sample in a Kjeldahl flask, by heating over a low flame, and blowing air into the flash by means of a foot-bellows ; the evaporation then proceeds very quickly, the process is then carried on in the usual way. If the vinegar is one of the lower numbers (weaker strengths), the figures, as before mentioned, should vary in proportion to the amount of acetic acid present. It is often useful for purposes of comparison to calculate the P_2O_5 and nitrogen as parts per hundred on the total solids, rather than on the original sample, as deviations are then more apparent.

The **Phosphoric Acid** can be readily estimated by Stock's method on the ash (see under 'Infants' Foods').

If **Free Sulphuric Acid** is suspected to be present, it may be estimated by the method as devised by O. Hehner. This process depends on the fact that whenever the ash of vinegar has not an alkaline reaction, free mineral acid has undoubtedly been added.

50 cc. of the sample is evaporated to dryness in a

platinum dish with 25 cc. $\frac{N}{10}$ NaHO; this is then ignited at the lowest possible temperature. 25 cc. $\frac{N}{10}$ HCl is then added to the dish, heated to expel CO_2, then filtered. The filter is washed with hot water, and the washings added to the filtrate. The free acid is then estimated with $\frac{N}{10}$ NaHO and phenolphthalein. The number of cc.'s of soda used is multiplied by ·0049, which gives the amount of free sulphuric acid in 50 cc. of the sample.

TEA.

Tea is the prepared leaf of the *Thea sinensis*, and allied species belonging to the genus *Camellia*. It is a shrub-like plant, and is grown in China, Japan, India, Ceylon, Natal, Brazil, etc. Black and green teas are the product of the same plant, and not of different ones, as was formerly supposed, the difference in colour being due to the mode of preparation.

The tea-leaves are picked at various stages of their growth, and the resulting crops are known as 'Pekoe,' 'Souchong,' 'Congou,' and 'Bohea,' the earlier pickings being considered the best in quality. Teas, in addition to being classed according to the age of the leaf and mode of preparation, are also classified according to their place of origin. The chief varieties of black tea are Ceylon, Moning, Oolong, Japan, and Assam; of green tea Gunpowder, Hyson, Twankay, etc. The mode of preparing tea varies very much with the locality, the essential difference between black and green tea being that in the case of the black tea the leaves are allowed to undergo a fermentative process before baking, whereas green tea is baked directly it is picked. Tea, as sold, is nearly always blended by mixing two or more kinds, with the object of securing a standard quality and flavour.

The chemical composition of tea is very complex, and varies greatly with the different varieties. The following are among the chief constituents: Moisture, caffeine, tannin, albuminous matters, ethereal oil, gum, dextrin, fat, wax, chlorophyll, woody fibre, ash, etc.

The principal constituents to which the aroma, flavour, and physiological action are due are (1) the volatile oil, which is present to the extent of about 0·5 per cent.,

and appears to be a product of the fermentative process ; (2) the alkaloid caffeine or theine ; (3) the tannin, which is present to the extent of 13 to 18 per cent.

Tea is now very rarely adulterated, on account of the provision made under the Sale of Food and Drugs Act, whereby tea is examined by the Customs authorities, and any samples found to be adulterated are not allowed to be imported.

Damaged tea used to be destroyed, but is now allowed to be sold for the manufacture of caffeine, after it has been treated with certain chemicals which render it incapable of being sold for human consumption. In September, 1894, a successful prosecution was instituted by the Inland Revenue authorities against some persons in London who had been collecting exhausted tea-leaves from refreshment houses, and drying them to sell for mixing with low-grade teas.

Foreign leaves are practically unknown in this country, but it occasionally happens that samples are met with which contain an undue proportion of stalks and ash. Tea-leaves may be examined by soaking in warm water, and carefully unrolling on a tile ; they can then be compared with genuine leaves.

The addition of foreign leaves to tea, such as willow, elder, sloe, hawthorn, beech, etc., was at one time a common practice, particularly abroad, and excellent illustrations of these leaves are to be found in Allen's 'Commercial Organic Analysis' (vol. iii., part ii.).

It has been proposed by Winter Blyth to utilize as a test the constant presence of manganese. There are, however, traces of manganese in many of the leaves that have been used to adulterate tea. A single tea-leaf reduced to ash and fused with sodium carbonate will give a perceptible green tint.

The facing or blooming of green tea with Prussian blue, indigo, etc., to give a bright appearance, used to be common years ago ; but the custom has now died out. The Japanese Government, in 1884, made it a criminal offence to adulterate tea. Facing of green tea was, however, justified by the plea that otherwise Japan teas would not suit the tastes of American consumers.

Caffeine, $C_8H_{10}N_4O_2$.—This alkaloid, the active principle of tea, was formerly known as **Theine**, while the name

Caffeine was generally given to the active principle of coffee. They were supposed to be distinct bodies, on account of apparent observed differences in their physiological action; now, however, they are known to be identical, and the name 'caffeine' is applied to both equally.

Caffeine has the following characters: Colourless, silky, acicular, odourless crystals, soluble in 80 parts of cold water, the solution having a faintly bitter taste, and being neutral to litmus. Easily soluble in boiling water, alcohol (90 per cent.), or chloroform; sparingly soluble in ether. When crystallized from aqueous solutions, caffeine contains one molecule of water. It dissolves without colour in sulphuric and nitric acid. In an aqueous solution of the alkaloid tannic acid gives a white precipitate soluble in excess of the reagent, but no precipitate is caused by Mayer's reagent or by iodized iodide of potassium; distinction from nearly all other alkaloids except theobromine.

The Analysis of Tea.—A complete analysis of tea is not often necessary; it is generally sufficient to see that there are no leaves of a suspicious character, and that the total ash, soluble ash, and the alkalinity of the soluble ash, calculated as K_2O, are normal.

Moisture.—5 grammes of the sample are dried in a platinum dish to a constant weight. The moisture contained in commercial teas varies from 4 to 11 per cent., the average being about 6 per cent.

Caffeine.—A. H. Allen, after careful study of all the methods proposed for the estimation of caffeine in tea, found the following method the most convenient:

6 grammes of the finely-powdered tea are boiled with 500 cc. of water under a reflux condenser for six hours; the liquid is then filtered through coarse filter-paper, and, together with washings, made up to 600 cc. The liquid is heated to boiling, and 4 grammes of finely-powdered acetate of lead added, and the liquid again boiled for ten minutes. The source of heat is then removed, and if the precipitate shows signs of settling rapidly, no further addition of lead acetate is required; and, after giving the liquid a few minutes to settle, it is poured on to a filter, and 500 cc. of filtrate collected. This is concentrated to 50 cc., and then extracted four or five times with

chloroform. On evaporation of the chloroform in a weighed flask, the caffeine is obtained in an almost pure condition.

The amount of caffeine in tea ranges from 1·8 to 3·5 per cent. With the exception of the case of the re-drying of exhausted tea-leaves already mentioned, we cannot find any record of prosecutions for adulterated tea in this country of late years.

In many of the published figures on tea the caffeine is too low, owing to the older methods for the estimation of this body failing to extract all the caffeine present.

Total Ash.—After the estimation of moisture, the tea is ignited at the lowest possible temperature. The ash should be gray, not green. If it is greenish, it has been ashed at too high a temperature. The total ash varies from about 5 to 7 per cent., the average being about 6·0 per cent.

Ash insoluble in Water.—The total ash, after weighing, is washed on a filter, and thoroughly extracted with boiling water until the washings are no longer alkaline. A considerable amount of boiling water is necessary, never less than about 400 cc.

Soluble Ash.—The filter containing the ash insoluble in water is now returned to the platinum dish, and ignited until the filter-paper is completely ashed. The weight of this **insoluble** ash, deducted from the **total** ash, gives the **soluble** ash.

The **alkalinity** of the **soluble ash** is determined by titrating the washings of the total ash with $\frac{N}{10}$ hydrochloric or sulphuric acid, using methyl orange as indicator. 1 cc. $\frac{N}{10}$ acid = ·0047 K_2O.

Out of 160 samples examined by us under the Food and Drugs Acts, the above constituents varied as under :

	Per cent.		Per cent.
Total ash	4·8	to	7·0
Soluble ash	2·8	„	4·0
Alkalinity as K_2O	1·3	„	2·0

These results are in accordance with those of other observers. C. E. Cribb has recently analysed a sample of 'Lie Tea' (sweepings, tea-dust, etc.), which contained 12 per cent. of ash.

Any addition of exhausted leaves would be detected by

the low percentage of caffeine, soluble ash, and alkalinity as K_2O.

T. Macfarlane, the analyst to the Canadian Government, in 1892 examined fifty-eight samples of tea, out of which he found two that had been adulterated with exhausted leaves.

Dr. J. Bell examined a number of exhausted teas, and found them to give the following average figures on analysis :

	Per cent.
Total ash	4·4
Soluble ash	0·7
Alkalinity as K_2O	0·2

It will thus be seen that the addition of exhausted leaves is one of the adulterations most difficult to detect, especially when present in small proportion.

COFFEE.

Coffee consists of the seeds of the *Coffea arabica*, belonging to the Natural Order *Cinchonaceæ*. Coffee is grown in many parts of the world. The best qualities come from India, Java, and Ceylon, but a large bulk of the supply is grown in Brazil. The coffee-beans vary in colour from light to dark yellow, and differ somewhat in size, the Java variety being the largest. The ripe fruit somewhat resembles a small black cherry, the pulp of which usually contains two berries enclosed in a hard membrane-like pericarp, known as the 'parchment.' The berries are freed from pulp and parchment by drying and passing through rollers, or, as in Java, by undergoing a fermentative process, after which the pulp can be washed away with water.

Coffee contains on the average 1·2 per cent. of caffeine, to which important alkaloid its characteristic properties are due. The properties of this body have already been referred to under 'Tea.'

The coffee-berries before use are roasted at a temperature of about 450° F., the effect of which is to develop the aroma, flavour, and colouring matter. The characteristic flavour and odour of coffee which is developed during the roasting is due to an oily volatile body known as caffeol.

The following analyses of two typical samples of coffee by Dr. Bell shows the general composition of the coffee-berries, and the effect of the roasting process upon the same :

	Mocha.		East Indian.	
	Raw.	Roasted.	Raw.	Roasted.
Caffeine	1·08	·82	1·11	1·05
Saccharine matter...	9·55	·43	8·90	·41
Caffeic acids ...	8·46	4·74	9·58	4·52
Alcohol extract, containing nitrogenous and colouring matters ...	6·90	14·14	4·31	12·67
Fat and oil... ...	12·60	13·59	11·81	13·41
Albumen	9·87	11·23	11·23	13·13
Dextrin	·87	1·24	·84	1·38
Cellulose and insoluble colouring matter	37·95	48·62	38·60	47·42
Ash	3·74	4·56	3·98	4·88
Moisture	8·98	0·63	9·64	1·13
	100·00	100·00	100·00	100·00

In the above analyses the caffeine is in all probability somewhat under-estimated, owing to an obsolete method having been employed for its estimation.

The most frequent adulterant of coffee is roasted chicory, but the following substances have been used : dandelion root, mangold wurzel, turnips, beans, peas, flour, condemned sea biscuit, etc., more or less caramelized by baking and the addition of burnt sugar. The beans may be entirely spurious, since a patent has been issued for the moulding of artificial berries from a composition consisting of chicory and other adulterants. These artificial beans are used on the Continent to adulterate the genuine article, but probably are not to be met with in this country, where, owing to the fact that coffee being

generally purchased ready ground, it lends itself to more profitable forms of adulteration.

Adulteration of coffee with other substances than chicory is now almost unknown in this country, though they are still known abroad.

Various 'extracts' of coffee are now sold, and it may therefore be expected that some attempt will be made to work up the grounds, just as was quite recently done in the case of exhausted tea-leaves; so that it may be wise in future to keep a look-out for exhausted coffee in ground coffee. Its presence would probably be revealed by a low extract gravity and low soluble ash.

We have recently examined a sample of artificial coffee which is sold in France, and has been offered for sale in this country. This sample of fictitious coffee yields an extract similar to that of pure coffee; but its microscopic appearance would at once distinguish it from genuine coffee, and render its detection certain, even if it were not present in a mixture to a greater extent than 5·0 per cent. It shows indications of the presence of a small quantity of exhausted coffee; but the striking characteristics are the clusters of starch cells, resembling the heads of Indian corn, and the flat network, which is totally unlike anything ever seen in coffee; these structures we found to be typical of acorn. On analysis, the sample gave the following figures (*Analyst*, xx. 176):

	Per cent.
Moisture	3·2
Ash	3·4
Soluble	2·7
Insoluble	0·7
Fat	12·3
Caffeine	none
Decoction gravity	1009·5

In Canada, during 1896, Macfarlane, on examining 141 samples, found thirty-six adulterated, the adulterants being chicory, and roasted pea, bean, rye, and wheat flours.

In most of his analyses the fat is about 10·0 per cent. on the pure coffees; but as fat or oil is added during roasting, this figure may vary. The moisture was about

6·0 per cent., and the gravity of a 10·0 per cent. decoction averaged 1009·5 (water 1000).

Chicory.—As already mentioned, chicory is the most important adulterant of coffee. The chicory plant is the well-known weed with the blue flowers, sometimes known as the wild-endive, *Cichorium intybus*, belonging to the Natural Order *Compositæ*. The following figures are the averages obtained by one of us (November, 1898) on nine samples of chicory :

	Per cent.
Moisture	10·4
Total ash	5·4
Phosphates (P_2O_5)	0·4
Insoluble ash (sand)	1·9
Fat	1·8
Nitrogenous matter ($N \times 6{\cdot}33$) ...	8·1
Soluble extract	55·5
Specific gravity of decoction, 10 per cent.	1024·3

The fact should not be overlooked that chicory is sometimes adulterated with some of the substances already enumerated under the adulterations of coffee. E. G. Clayton has recently pointed out that chicory is frequently adulterated by the addition of saccharine matter.

Analysis of Coffee.—A full analysis of coffee is seldom required when a sample is adulterated ; it generally resolves itself into a determination of the relative proportions of coffee and chicory.

Moisture. — The moisture is determined upon 5 grammes, which are dried at 100° C. until constant in weight. The percentage of moisture should not exceed 6·0 per cent.

Caffeine.—Caffeine is determined by the process given under 'Tea.' The extraction is best made by several successive boilings with water to ensure the thorough extraction of the alkaloid. The percentage of caffeine in genuine coffee varies from 1·1 to 1·3 per cent. This determination furnishes the most reliable datum upon which to calculate the percentage of genuine coffee in a mixture.

Total Ash. — The ash is determined by igniting

5 grammes of the sample until a nearly white ash is obtained. The total ash of coffee varies from 3·5 to 5·0 per cent.

Microscopical Examination.—This is conducted as follows: A little of the sample is shaken in fine muslin to separate the finer particles, which are then treated in a test tube with boiling 2 per cent. sulphuric acid for about a minute. The residue is filtered off, washed slightly, and again transferred to a test-tube and boiled for a minute with 2 per cent. solution of caustic potash. The residue is washed with water, and a little mounted in glycerine and water upon a glass slip, covered with a cover-glass, and examined with an inch and a quarter-inch power.

The following procedure is also recommended: The sample is extracted with ether to remove fat, and then extracted with spirit and water to remove colouring matter. On examining the residue mounted as above, starchy and other structures will be readily seen.

Coffee contains a structure of bars not unlike a wooden paling, while chicory (and certain substances with which chicory may be adulterated) shows hollow spiral ducts never seen in coffee.

Descriptions of microscopic characters are unsatisfactory, and are of little value in the case of adulterants of coffee, so we shall not attempt to give any here. If a suspected sample is found to have any abnormal characters, when compared with a genuine sample prepared under the same conditions, it should be compared with slides of all the possible adulterants. This furnishes the only possible means of recognising some of the rarer adulterants of coffee.

Estimation of Chicory.—The most useful qualitative test for chicory in coffee is the behaviour of a pinch of the sample when dropped on to the surface of a glass of water. Coffee remains floating on the top—for some minutes, at least; while chicory sinks almost instantly, colouring the water in its fall. The fragments which have sunk to the bottom, and are presumably chicory, should be taken out separately, and examined first as to their hardness. Coffee is hard; chicory is soft. They are then examined under the microscope.

The most reliable method of determining the relative proportions of chicory and coffee in a mixture is to deter-

mine the specific gravity of a 10 per cent. decoction. This is best carried out as follows: 20 grammes of the sample are treated with 200 cc. of water and just raised to the boil, when the liquid is strained off through muslin. The strained liquid is then filtered through filter-paper, cooled down to 15·5°C., and the specific gravity taken with a bottle or the Westphal balance. Under these conditions, pure coffee gives a decoction having a specific gravity not exceeding 1009·5, while chicory gives a specific gravity of 1024·0 to 1025·0. So roughly we may consider every rise of 1·6 in the specific gravity above 1009·5 as equivalent to an addition of 10 per cent. of chicory.

The following example shows how the exact amount may be calculated:

Specific gravity of coffee decoction = 1009·5.
Specific gravity of chicory decoction = 1024·5.
Specific gravity of suspected mixture = 1016·0.

1016 < 1009·5 difference = 6·5
1024·5 „ = 8·5

15·0

15 : 6·5 :: 100 : 43·3% chicory.
15 : 8·5 :: 100 : 56·6% coffee.

The fact that saccharine matter is frequently added to chicory, is obviously a point of considerable importance, seeing that it is customary to judge of the quantity of chicory in a mixture, by the specific gravity of the decoction, or the extractive matter, for any excess of saccharine matter might cause an over-estimate to be formed of the quantity of chicory present.

Other methods for the estimation of the amount of chicory in a mixture have been used, such as comparing the tints produced by a 10·0 per cent. decoction of the sample with the colour yielded by known mixtures of coffee and chicory; this would hardly be safe to use now, seeing that the chicory may be tinted with caramel.

Starch.—Starchy matters are tested for by the iodine test, which is applied as follows: A 10 per cent. decoction of the sample is boiled with animal charcoal to decolourize it, or it is treated with a solution of permanganate of

potash slightly acidulated with sulphuric acid, until nearly decolourized. The decolourized decoction can then be tested with a drop or two of solution of iodine in potassium iodide, when a blue colour will indicate the presence of starch. The presence of starch would point to the possible presence of breadcrumbs, acorns, rye, wheat, bean, pea, or other form of starch-containing substance.

COCOA.

The cocoa of commerce is the prepared seed of the *Theobroma cacao* and allied species, belonging to the Natural Order of *Byttneriaceæ*. The cocoa-trees are cultivated largely in the West India Islands, Mexico, Brazil, etc., and its cultivation has been introduced into some parts of Asia and Africa.

The seeds of the cocoa plant are contained in a pod, each pod containing from 25 to 50 seeds packed in a pulpy substance. The pods are picked when ripe, and allowed to undergo a fermentative process which renders the separation of the seeds from the pulp easy. The seeds are then dried in the sun or in ovens, after which they are roasted in iron cylinders, much in the same way as coffee. The roasting process, as in the case of coffee, develops the aroma and flavour of the cocoa, which entirely depend upon the skill with which this operation is conducted. After the roasting process, the seeds or beans are passed through a machine which gently cracks them and renders the outside husk or shell easy of separation. The crushed beans are then passed through a kind of winnowing machine which separates the husk from the cocoa 'nibs.'

The following figures obtained by König, the average results obtained upon eight samples of decorticated cocoa-beans and of the husks from the same, show the general composition of raw cocoa :

	Moisture.	Nitrogenous Matters.	Fat.	Starch.	Cellulose.	Ash.
Cocoa-beans free from shell ...	3·2	14·7	49·0	13·3	3·7	3·65
Cocoa husks ...	7·8	14·3	6·4	—	14·7	7·1

The following figures are the average results obtained

upon eight samples of raw cocoa, after removal of the husk, by Eastes and Terry :

Moisture.	Fat.	Theobromine.	Ash.	Phosphoric Acid (H_3PO_4).
4·5	51·5	1·3	3·0	1·1

Theobromine, $C_5H_2(CH_3)_2N_4O_2$. — This alkaloid is a most characteristic and important constituent of cocoa. It is the lower homologue of caffeine, to which it presents a general resemblance. Its physiological action is similar to, but more powerful than, caffeine. The amount of theobromine in genuine cocoa varies from 1·3 to 1·7 per cent.

Starch.—Cocoa contains about 10 per cent. of natural starch. The granules are small and round, and are easily distinguished from any added starch with which cocoa is likely to be adulterated.

Fat.—The fat of cocoa, known as 'cocoa-butter,' is the *Oleum theobromatis* of the British Pharmacopœia. It is a yellowish fat, consisting mainly of stearin, having an agreeable odour and taste of chocolate. Cocoa or cacao butter is largely used in pharmacy for the making of pessaries and suppositories, its value depending upon the fact that its melting-point is slightly below the temperature of the body. According to the British Pharmacopœia, its melting-point ranges from 31° to 34° C.

Care should be taken not to confuse the fat of cocoa with cocoanut oil.

Commercial Cocoa.—The simplest form of commercial cocoa consists of the roasted seeds, free from husks, ground to a paste and moulded into cakes. This form of cocoa is sometimes known as 'rock-cocoa.' Owing to the large proportion of fat, averaging 50 per cent., contained in natural cocoa, it is quite impossible to make it into a powder, which mechanical difficulty is generally overcome by the addition of dilutants, such as starch and sugar, or by the partial removal of the fat by the process of hot pressing. The use of natural cocoa is objectionable in some cases owing to this excessive amount of fat, which renders it difficult of digestion.

The cocoa preparations known as 'cocoa extract' and 'cocoa essence' consist of natural cocoa partly deprived of its fat. The term 'chocolate' is generally given to pre-

parations of cocoa mixed with sugar, starch, added cocoa-fat, etc., flavoured with vanilla.

The term 'cocoa' is frequently misapplied to mixtures of cocoa with some form of starch and sugar. It is much to be desired that the term 'chocolate' be applied to all preparations of this class.

Mixtures of cocoa with starch and sugar are allowed by law to be sold as cocoa if the fact that they are mixtures is disclosed on the label. Some of the leading brands of cocoa are practically free from fat, and their value is certainly enhanced thereby. Some of the cocoas sold as mixtures contain considerably more starch and sugar than they do cocoa.

A further treatment of concentrated cocoa is practised by some manufacturers of cocoa extract, particularly on the Continent. This treatment consists of the addition of alkali to the cocoa, which may be either ammonia, or soda, or potassium carbonates. The presence of this added alkali causes the fat to become emulsified, and any free fatty acid is saponified, with the result that on treating the cocoa with hot water there is less tendency for the fatty globules to separate. In the case of a well-known brand of cocoa, potassium carbonate is the form of alkali added. The cocoa preparations containing added alkali are called 'soluble cocoa.' In view of the practice of adding alkali to cocoa, it is worth quoting the opinion of Dr. Ringer, who states that 'The sustained administration of alkalies and their carbonates renders the blood poorer in solid and in red corpuscles, and impairs the nutrition of the body.' Of ammonia, carbonate of ammonia, he says : 'These preparations have many properties in common with the alkaline potash and soda group. They possess a strong alkaline reaction, are freely soluble in water, have a high diffusion power, and dissolve the animal textures.'

Thus it will be seen that the continued administration of alkaline carbonates may have a detrimental action on the functions of the body.

Analysis of Cocoa.—Cocoa does not appear to be so often adulterated as is stated by some authors. Out of 120 samples examined by us under the Food and Drugs Acts, only six samples were adulterated. Pure cocoa should contain no addition of starch or sugar, and may

with advantage be freed from part or all of its natural fat.

The purity of a cocoa can generally be decided by the following data : Total ash, the cold-water extract, and the soluble ash. The microscope will detect the presence and nature of any added farina or starch.

Total Ash.—This is determined by igniting 5 grammes of the sample. The total ash calculated on the fat-free sample is fairly constant, provided that no treatment with alkali has taken place. The ash of a pure cocoa is about 5·0 per cent. Blyth found 0·9 per cent. of phosphoric acid in genuine cocoa, and proposed to make this a basis for calculating the amount of true cocoa in a mixture. Other observers have, however, found more phosphoric acid than this, so that the method cannot be relied on.

Soluble Extract.—5 grammes of the sample are rubbed up in a mortar with 250 cc. of cold water until a smooth mixture results. It is then shaken at intervals and allowed to stand overnight. The supernatant liquid is poured off and filtered, and 50 cc. (=1 gramme of sample) are evaporated in a dish and dried to constant weight.

The cold-water extract should not exceed 18 per cent. ; any material excess beyond this will probably be due to added sugar.

Soluble Ash.—The residue from the above determination is gently ignited. The resulting ash should not fall below 2·0 per cent.

Fat.—This is estimated by extracting 5 grammes in a Soxhlet extractor with ether or petroleum spirit.

Theobromine.—Theobromine may be estimated by extracting 20 grammes of the sample with petroleum ether. After previously drying at 100° C., the residue is first heated in the water-bath to free it from petroleum ether, and then extracted with alcohol (specific gravity ·850), which dissolves sugar, theobromine, tannin, etc. ; the alcohol is distilled off, the residue is taken up with water and clarified with lead acetate, and the lead is afterwards removed by sulphuretted hydrogen ; from this liquid the theobromine is extracted by repeated shakings with chloroform. On account of its presence in such variable proportions, it cannot be made of use to determine the amount of true cocoa present in a mixture.

Theobromine contains 31·1 per cent. of nitrogen.

Proteids.—These can be approximately estimated by determining the total nitrogen by the Kjeldahl method on 2 to 3 grammes of the sample. After deducting the amount of nitrogen due to theobromine, the remainder multiplied by the factor 6·25 will equal the proteid or nitrogenous matter present.

The total nitrogen of cocoa-nibs varies from 2·1 to 2·5 per cent. the average amount being 2·2 per cent.

Sugar and Starch.—If the sample is found to contain added sugar and starch, it is better to make a direct estimation of them than to trust to any other method for estimating their amount.

Ten grammes of the sample are dried at 100° C., placed in a filter-paper cylinder and extracted with petroleum spirit, in a Soxhlet's extractor. The cylinder is then dried, and extracted with alcohol (specific gravity ·850); this dissolves the sugar and a quantity of other matters, which must be removed. This is effected as detailed above: the purified sugar is boiled for ten minutes with 2·0 per cent. hydrochloric acid neutralized, and the *invert sugar* estimated by the Pavy process. The residue containing the starch must now be boiled for 6 hours with 200 cc. of 2·0 per cent. sulphuric acid at the atmospheric pressure, or for one hour at a temperature of 120° C. (15 lb. per square inch); this can be conveniently effected by using a screw-stoppered bottle placed in an oil-bath, or in boiling saturated solution of sodium nitrate. The residue, consisting of *insoluble fibre* and some nitrogenous matter, may be treated with a 2·0 per cent. solution of caustic soda, filtered and washed on the filter with very weak hydrochloric acid, alcohol, and ether, dried and weighed as *insoluble fibre*. It is not improbable that some of the cellulose may not be converted into sugar. In calculating the amount of starch from the sugar obtained by its conversion, it must not be omitted to subtract the amount of starch natural to cocoa, *i.e.*, 10·0 per cent.

Analyses of Commercial Cocoas.—The following figures were obtained by the analysis, in Mr. Allen's laboratory, of a specimen of the best cocoa-nibs and two of the leading brands of cocoa essence, or soluble cocoa, to which no starch or sugar had been added:

	Cocoa-nibs.	Sample A.	Sample B.
	Per cent.	Per cent.	Per cent.
Ash	2·53	4·93	8·25
Insoluble in water	1·71	3·50	2·09
Soluble in water	0·82	1·43	6·16
Alkalinity (K_2O) of soluble portion ..	0·32	0·49	3·23
Cold-water extract	9·72	11·64	18·66
Alkalinity (K_2O) to methyl-orange ..	0·69	0·71	2·02
Acidity (K_2O) to phenolphthalein ..	0·63	0·76	0·38
Hot-water extract	16·84	20·36	27·16
Containing: Ash	3·34	4·93	7·85
Organic extract	13·50	15·43	19·31

The following results were obtained by E. E. Ewell (*Bulletin U.S. Department of Agriculture*, No. 13) by the analysis of well-known brands of commercial cocoa and its preparations:

Description of Sample.	Fat.	Fibrine.	Cane-sugar.	Reducing-sugar.	Total Ash	Added Starch.
Fry's Cocoa Extract	30·95	3·89	..	..	4·24	None.
Schweitzer's Cocoatina ..	31·13	3·70	..	..	6·33	None.
Van Houten's Cocoa ..	29·81	4·38	..	..	8·64	None.
Blooker's Dutch Cocoa ..	31·48	3·76	..	..	6·06	None.
Rowntree's Extract of Cocoa	27·56	4·42	..	..	8·48	None.
Rowntree's Powdered Chocolate	25·84	1·30	51	none	1·66	Very small amount of arrowroot.
Epps' Prepared Cocoa ..	25·94	1·51	26	none	3·15	Much arrowroot.
Fry's Diamond Sweet Chocolate	18·60	·81	55	some	1·16	Much wheat starch, with some arrowroot.
London Cocoa (unknown maker)	11·13	2·13	32	some	2·82	Very largely diluted with arrowroot.
Chocolate Menier	21·31	1·10	58	none	1·40	None.

According to evidence given in the case of Gibson *v.* Leaper, Epps' Cocoa contains 40 per cent. of cocoa, 16 of starch (West Indian arrowroot), and 44 per cent. of sugar.

PEPPER.

Black pepper is described in the British Pharmacopœia as 'the dried unripe fruit of the *Piper nigrum*. Characters: Roundish, usually about one-fifth of an inch in diameter; pericarp thin, blackish-brown, wrinkled, and containing a hard, smooth, roundish seed of a yellowish-brown or gray colour; odour aromatic; taste pungent and bitterish.'

Both black and white pepper are obtained from the *Piper nigrum;* this is a perennial climbing shrub. The plant is capable of growing to a considerable height, but, for the sake of convenience, is usually kept low, and often trained on poles. When one or two berries at the base of the spike begin to turn red, the whole spike is pinched off; the berries are picked off and dried.

White pepper is prepared from black pepper by removing the dark outer layer of pericarp; this is done by soaking the fruit in water to soften the husk, which is then removed by friction; the 'fibre' in white pepper is therefore much less than in black pepper. The pepper-husks have a legitimate use in the manufacture of sausages and sauces.

Pepper is imported from Penang, Singapore, Tellicherry, Malabar, Sumatra, etc.

The chief constituents of pepper are the acrid resin, to which pepper owes its pungency; a volatile oil, which is present to the extent of about 2 per cent.; a neutral principle termed piperin, which is present to the extent of 2 to 3 per cent.; cellulose or woody fibre; and starch.

Clifford Richardson (*Bulletin U.S. Agricultural Department*, No. 13) gives the following figures as representing the composition of black and white peppers:

	Black.	White.
Water	8 to 11	8 to 11
Ash	2·75 „ 5·0	1 „ 2
Volatile oil	0·5 „ 1·75	0·5 „ 1·75
Piperin and resin ...	7 „ 8	7 „ 8
Starch	32 „ 38	40 „ 44
Fibre	8 „ 11	4·11 „ 8
Albuminoids	7 „ 12	8 „ 10

He describes as adulterants maize, rice, buckwheat, ground nutshells, gypsum, cayenne, etc.

Macfarlane (Canada) in 1890 obtained the following figures :

Average Composition of Six Samples of Genuine Black Peppercorns.

	Per cent.
Moisture, etc., lost at 100° C.	12·03
Ash soluble in hot water	2·41
Ash insoluble in water	2·05
Total ash	4·47
Ash insoluble in HCl	0·36
Sand expressed as a percentage on the total ash	8·0
Alcoholic extract	8·71

Average on Five Samples of Genuine White Peppercorns.

	Per cent.
Moisture, etc., lost at 100° C.	12·34
Ash soluble in hot water	0·54
Ash insoluble in water	2·46
Total ash	3·0
Insoluble in HCl	0·55
Sand expressed as a percentage on the total ash	2·10
Alcoholic extract	7·73

After grinding, both black and white pepper are graded by bolting through sieves, which tends to cause all the mineral impurities to collect in the lower grades.

Black peppercorns, which are much corrugated, are dried upon earthen floors, and in this process take up a considerable amount of dirt. They are frequently imported into the United Kingdom in this dirty condition, and are ground without having been cleansed. The pepper prepared from such peppercorns will be found to contain a large proportion of dirt, consisting mainly of sand, and a desire has been expressed that a limit should be fixed to the proportion of mineral matter which pepper may legitimately contain. A consignment of pepper stated to have been imported from the Straits Settlement contained an admixture of imitation peppercorns made of clay.

Other forms of adulteration consist of the admixture of ground olive-stones, or 'poivrette;' and white pepper is sometimes mixed with ground rice, or with 'long pepper.'

In 1884 W. Leng showed that the extracts yielded by true pepper, and by mixtures of true pepper with various adulterants, to ether, alcohol, and water, afford no means of judging of the purity of the sample, while he was in favour of the estimation of the starch. Heisch at the same date published a number of analyses of genuine peppers showing that the piperin and starch vary greatly in genuine samples. In 1887 Bostock Hill examined a sample of pepper adulterated with clay to the extent of 24 per cent., while Dr. Bernard Dyer examined some white pepper which consisted of undecorticated peppercorns that had been faced with a white clayey material. Campbell Brown, in 1887, examined several samples of 'poivrette,' and proved their identity with ground olive-stones.

The following are some of the figures obtained by him (*Analyst*, xii. 67) on three samples of long pepper (*Chavica Roxburgii*):

	Ash.	Cellulose.	Total Nitrogen.	Ether Extract.
1	8·91	15·7	2·1	5·5
2	8·98	10·5	2·0	4·9
3	9·61	10·7	2·3	8·6

In vol. xiii. of the *Analyst* will be found drawings by F. W. Rimmington showing the microscopical appearance of true pepper and of long pepper, the starch in long pepper being larger and more irregular in shape than that of true pepper.

Stoddart, in 1889, drew attention to a mixture composed of rice-starch, barytes, chalk and lead chromate, which was sold for the purpose of improving the colour of pepper. He also examined a sample of pepper which contained 10 per cent. of steatite. In vol. xiv. of the *Analyst* W. Johnstone has published some very complete analyses of thirteen samples of pepper of known origin, showing that an estimation of the fibre is of more value than an estimation of the starch. He finds the fibre to vary from 10 to 15 per cent., and considers that no

genuine pepper can contain over 20 per cent. of fibre. In 1890 Macfarlane examined seventy-three samples of commercial pepper sold in Canada, with the result that two-thirds of them were regarded as adulterated. The following substances were used as adulterants: Rice, maize, leguminous starches, wheat-flour, ground cocoa-nut shells, mineral matters, mustard cake, and certain other matters which could not be identified.

The Analysis of Pepper.—It will be apparent from the foregoing that, in spite of the extended researches made by many observers, our power of recognising adulterations is at present very limited, and that we are largely dependent on the microscope, owing to the wide natural variations in the chemical constitution of pepper. The microscopic appearance of genuine pepper and of possible adulterants should therefore be carefully studied. In order to obtain satisfactory results, it is essential to observe the same mode of preparation of the specimen to be examined under the microscope, and for this purpose it should be prepared by boiling some of the sample in 5 per cent. sulphuric acid for five minutes, and then washing by decantation ; it is then boiled for five minutes in a 5 per cent. solution of caustic potash, and finally washed with hot water. In the case of black pepper when treated thus, we shall see portions of the husk which are characteristic, while in white pepper, from which most of the husk is removed, the starch peculiar to pepper will be more prominent. The chief adulterations to be expected in this country are poivrette and long pepper. Rice and ground (spent) ginger have been found, but are so readily detected by the microscope that they are not likely to be often used.

The following figures were obtained by Campbell Brown (January, 1887) on two samples of poivrette and a sample of ground olive-stones :

	Ash.	Matter soluble by boiling in dilute acid.	Albuminous and other matter soluble in alkali.	Woody fibre insoluble in acid and alkali.
White pepperette	1·33	38·32	14·08	48·48
Black „	2·47	34·55	17·66	47·69
Olive-stones ...	1·61	39·08	15·04	45·38

In the same paper the author points out that the reservation in section 8 of the Sale of Food and Drugs Act, permitting the sale of mixtures provided the fact is disclosed on the label, would not apply to the case of poivrette, as the admixture is manifestly made to increase the weight and bulk.

Estimations of the fibre in pepper and its possible adulterants have been made by several observers : Stokes (*Analyst*, xii. 147), Rabourdin (*Journal de Pharmacie*, April, 1884), Heisch (*Analyst*, xiii. 149), Clifford Richardson (*Bulletin U.S. Agricultural Department*, No. 13), and Stock (*Analyst*, xvi. 224).

Determinations of Fibre in Samples of Genuine Pepper and Possible Adulterants.

	Stock.	Stokes.	Rabourdin.	Clifford Richardson.	C. Heisch.
White pepper :					
Maximum ...	9·6	13·8	—	8·0	6·7
Average ...	6·5	13·3	17·5	—	5·3
Minimum ...	3·5	12·7	—	4·1	3·4
Black pepper :					
Maximum ...	18·0	26·3	35·0	11·0	27·8
Average ...	14·0	24·4	—	—	16·7
Minimum ...	10·0	21·0	30·0	8·0	11·5
Long pepper :					
Maximum ...	—	22·3	—	—	12·9
Average ...	—	21·0	—	—	12·2
Minimum ...	—	20·0	—	—	11·4
Poivrette :					
Average ...	—	62·2	74·5	—	65·0

From an inspection of the above results, it will be seen that the differences in the methods employed have produced greater differences than can be accounted for by the fact that the observers quoted were working on different samples and at different dates. The latter consideration should always be borne in mind in comparing analyses, as new methods of cleaning or grading may have come into use, or, what is possible in the present

case, the cheapening of the article may have removed the temptation to add pepper-dust or husks.

Examining the table of figures given above, it will be seen that the results obtained by Stock and C. Heisch agree fairly if we take the averages, while Stokes' figures are more in accordance with those of Rabourdin. This is due to the fact that the two latter observers rely on sulphuric acid alone as a means of removing starch, etc., from the insoluble matter weighed as 'fibre,' while Stock (and probably Heisch) used the ordinary method of treatment, first with acid, and then with alkali. Stokes prefers to use acid only for fear of the cellulose being dissolved, but this treatment does not completely remove resin, etc. Whichever method is used, the results are only comparable with figures obtained in the same way. The limits given at the end of this article are for fibre estimated by the *acid and alkali treatment*.

Neither poivretten or long pepper is easily recognised by simple examination under the microscope, but both are at once distinguished with ease and certainty by the method published by A. W. Stokes (*Analyst*, xiv. 82). He first warms a little of the sample with ammonia, and after washing with water examines it in a drop of glycerine with an inch power and polarized light. On rotating the prisms it will be found possible to obtain an entirely dark field when pure pepper is present, but no position will give a dark field when either poivrette, long pepper or rice is present. The fragments of these latter bodies appear as faintly illuminated ghost-like fragments, the poivrette showing a distinctly reddish light, while the colour emitted by long pepper is perceptibly blue.

Mr. Stokes kindly demonstrated this method to one of us, and everyone who tries it will recognise its value. For the success of this method the sample must be treated as described, and merely flattened out on the slide with the blade of a penknife, not ground to a fine dust.

W. Busse (*Analyst*, 1895, p. 180) describes a method for ascertaining the amount of pepper-husk added to pepper, depending on the colouring matters that are present in the husk alone.

D. Martelli (*Analyst*, xx. 181), after reviewing the various published methods for the detection of poivrette,

recommends the following test: Digest for two or three days 1 gramme of phloroglucol in 50 or 60 cc. of hydrochloric acid (specific gravity 1·1), and decant the clear solution. To about 0·5 gramme of the sample of pepper add enough of the reagent to cover it, and heat cautiously till fumes of hydrochloric acid begin to come off. Poivrette and like substances give a very intense and cherry-red colour. Poivrette has the following microscopical characters, as seen with a half or quarter inch objective: pale dense ligneous cells, some entire and marked with linear air-spaces, some torn and indistinct.

The ash of genuine black pepper should never exceed 7 per cent., while it is very rarely that it exceeds 4 to 5 per cent., and when it does it is due to dirt either deliberately added, or to earthy matter which may have adhered to the berries when soft, *i.e.*, before drying. There is excuse for some extraneous matter being present for this reason in the case of black peppers, though 7 per cent. is a liberal allowance. In the case of white peppers, which are made from decorticated corns, there is no excuse for the presence of any mineral matter, beyond that due to the inorganic constituents natural to pepper.

Stock gives 3·0 per cent. as the maximum limit of ash that should be permitted in white pepper, which is confirmed by Macfarlane's analyses given above, in which the average ash of five samples of genuine decorticated corns was 3 per cent.

Stock has drawn attention to the addition of small quantities of chalk to white pepper by the grinders, and he considers that no white pepper should be passed as genuine in which the proportion of lime (in terms of calcium carbonate) to the ash exceeds 60 per cent.

We may therefore regard those samples as genuine which fulfil the following conditions:

	Total Ash.	Fibre.
Black pepper, not exceeding ...	7	10 to 18
White pepper, „ „ ...	3	3·5 „ 9·5

provided, of course, that the microscopic appearance is normal.

The proportion of phosphoric acid to the total ash in pepper does not seem to have been recently determined, and it would be interesting to know whether it is constant.

CAYENNE PEPPER.

Cayenne pepper consists of the ripe dried fruit of the *Capsicum fastigiatum* and *Capsicum annuum*, belonging to the Natural Order *Solanaceæ*. Cayenne pepper is also known in commerce as pod pepper and Guinea pepper. The plant is a native of tropical Africa and America.

The pods of *Capsicum fastigiatum* vary from about half to three-quarters of an inch in length, and are about a quarter of an inch in diameter, somewhat shrivelled, and composed of a smooth shining pericarp of a dull orange-red colour, enclosing several small, white, roundish seeds. The taste and smell are both pungent and burning. The pods of *Capsicum annuum* are smaller than the above, and the powdered product is brighter in colour.

Cayenne pepper contains a red colouring matter, fat, resin, cellulose, and a body known as capsaicin ($C_9H_{14}O_2$), which is a colourless, highly acrid body, soluble in ether, benzene, alcohol, acetic acid, etc. The total ash of cayenne pepper is generally said to vary from 4 to 6 per cent., but we have examined a sample which contained 9·4 per cent. Various adulterations are mentioned; in fact, almost anything red is said to have been used. Since the year 1879, when A. H. Allen found 6·0 per cent. of red lead in a sample taken in the neighbourhood of Sheffield, we cannot find any recorded adulterations. It would be a difficult matter to introduce any substance which would not be easily detected microscopically or in the ash.

The microscopic appearance is that of a cellular structure enclosing occasional drops of oil, but no starch. As in all cases of this description, the sample in question should be compared with one of known origin. The pungency for which cayenne is valued resides entirely in the matter extractable by ether (oil and resin), the residue left after extraction being tasteless. W. Blyth gives the following figures obtained by him as the mean of his analyses of several samples of genuine cayenne.

	Per cent.
Aqueous extract of dried cayenne ...	32·1
Alcoholic extract	25·79
Benzole extract	20·00
Ethereal extract	10·43
Ash	5·69
Total nitrogen	2·04

The following figures were obtained by one of us on three samples of genuine red pepper :

	1.	2.	3.
Moisture ...	8·6	9·3	9·0
Ash	6·1	9·4	—
P_2O_5	0·70	0·73	0·71
K_2O	2·6	4·6	2·3

GINGER.

Ginger is defined in the British Pharmacopœia as 'the scraped and dried rhizome of *Zingiber officinale*. Characters : Flattish irregularly branched pieces varying in length, but commonly from about 3 to 4 inches, each branch marked at its summit by a depressed scar; externally pale buff and somewhat striated and fibrous, breaking readily with a mealy, short, but rather fibrous fracture. Odour agreeable ; taste strong, pungent.'

Ginger has been known and valued as a condiment from very early times. The ginger plant is a native of India, but has been acclimatized in various other countries. At present in this country we import ginger from Jamaica, Cochin China, Bengal, Africa, and Japan.

Jamaica ginger is most valued, while African is the lowest quality. To prepare the root for commerce it is either scraped and washed and dried in the sun, or it may be dried with the epidermis left on, when it is known as 'coated ginger.' There appears also to be a practice of splitting the rhizome in the case of some Japan gingers (Bernard Dyer, *Analyst*), besides which gingers are sometimes dipped into boiling water to soften the outer skin. In addition to this, whole ginger is sometimes whitewashed to preserve it from the attacks of insects, or treated with chloride of lime to bleach it, or whitewashed

with carbonate or sulphate of calcium. Both carbonate and sulphate of calcium were found in ginger by Garside (*Pharm. Journ.*, April 18, 1874).

The composition of ginger varies very greatly, for not only do differences occur in the products of different countries, but the same races of plants present considerable variation *inter se*. Ginger contains a volatile oil possessing the odour of ginger, but not its pungent taste. Fluckiger and Hanbury obtained 4·5 oz. of this oil from 112 lbs. of Jamaica ginger, or, roughly, 0·25 per cent. Its specific gravity was ·878, and a column 50 mm. long deviated the ray of polarized light 21·6°. If this oil were a constant constituent of genuine ginger, its estimation might be of value.

Dyer and Gilbard (*Analyst*, xviii. 199) point out that, while the volatile oil of ginger is too variable in amount to be of much service, the alcoholic extract after complete removal of the ethereal extract is of value, as in the gingers they examined it ranged from 2·1 to 3·8 per cent. with an average of 2·8, while in the exhausted gingers it ranged from ·8 to 1·4 per cent. with an average of 1·2 per cent. The ash soluble in water varied from 1·9 to 3·0 per cent. in the genuine, with an average of 2·7, while in the exhausted gingers it varied from 0·2 to 0·5, averaging 0·35 per cent. The following figures were obtained by one of us (T. H. Pearmain) on five samples of genuine ginger, and appeared in a paper by A. H. Allen (*Analyst*, xix. 125) :

	1.	2.	3.	4.	5.
Total ash, less sand ...	3·1	3·9	3·7	5·0	4·5
Ash soluble in hot water ...	2·2	2·7	2·4	1·8	2·0
Fixed ether extract ...	3·2	3·0	2·5	5·0	4·2
Alcoholic extract after treatment with ether ...	2·7	3·1	3·4	2·9	3·0

These results practically substantiate those previously published by Dyer and Gilbard. In the same paper appear a number of figures obtained by A. H. Allen on samples purchased under the Act, some of which were eventually admitted to contain exhausted ginger. It appeared that the cold-water extract was likely to prove of value, but this was again shown to be unreliable in the following series of figures obtained by A. H. Allen and

one of us, on samples of known origin supplied to us by W. Chattaway, Apothecaries' Hall :

	Jamaica Gingers.				
	1.	2.	3.	4.	5.
Moisture	11·2	10·98	13·95	12·76	13·96
Ash	—	—	3·90	3·29	3·45
Soluble ash	1·70	1·41	3·05	1·75	1·71
Cold-water extract ...	15·65	13·25	14·40	12·25	11·85

	Cochin Gingers.			African Gingers.	
	1.	2.	3.	1.	2.
Moisture	10·64	13·50	13·23	15·97	13·70
Ash	—	3·81	3·62	3·66	3·90
Soluble ash	1·71	2·03	2·04	2·28	2·41
Cold-water extract ...	13·00	8·65	11·65	10·80	10·10

The average of the above is :

Moisture	13·00
Ash	3·66
Soluble ash	2·01
Cold-water extract	12·12

The microscopical appearance of ginger is so distinctive that it is (in this country) rarely or never adulterated with anything but exhausted ginger.

Exhausted ginger possesses exactly the same appearance under the microscope as genuine ginger, as the starch cells are not altered in shape by the extraction practised. Budden (*Analyst*, xviii. 201) mentions a case in his experience where ginger which had been exhausted by strong spirit, so as to leave merely a mass of cellulose fibre and starchy matter remaining, was used to adulterate ground ginger.

Ginger is exhausted either after grinding or after being roughly crushed. In both cases very weak spirit is generally employed, containing at most 25 per cent. of proof

spirit. This extracts much of the oil of ginger, but does not remove the resin. It is desired that the extract shall not become milky on mixing with water, as it would if strong spirit were used, which would take up resin.

Allen (*Analyst*, xix. 217) has collected the determinations of ash by a number of observers extending over several years, and found the grand average of 104 samples to be 4·46 of ash. As these samples include a number of commercial specimens which may have contained varying amounts of sand and matters added to improve the colour or to protect the roots from insects, the figure is higher than that which is obtained by averaging results from genuine samples only.

The ash of scraped gingers—that is, of all gingers except African ginger—never exceeds 4·5 per cent., unless it is raised by the addition of unnecessary dirt, or excessive amounts of substances used for whitewashing the unground rhizomes. It may be regarded as legitimate to whitewash the unground ginger to preserve it from the attacks of insects, though many importers contrive to bring it in good condition without such means. This practice must not, however, be made the excuse for additions of mineral substances to ground ginger, and any sand or matter insoluble in hydrochloric acid above 1·0 per cent. should be regarded as an adulterant.

The composition of the ash of ginger does not seem to have been exhaustively studied, but W. F. K. Stock determines the potash, which, in a number of genuine samples examined by him, varied between 1·8 and 0·8 per cent.

The starch of ginger consists of irregularly spherical granules, some similar to arrowroot starch, which are not altered in shape by alcoholic exhaustion as usually practised (Allen and Stock), but might be swelled or burst by immersing the roots into boiling water.

The external layer of unscraped ginger is about 1 millimetre thick ; within this is the mealy portion, composed of starch granules, masses of the resins peculiar to ginger, drops of ginger oil, and fibrous matter.

In grinding ginger roots for experimental purposes, it will be noticed that the fibrous matter very readily tends to separate from the more friable portion, and it might therefore be expected that the operations of grinding and 'grading' would cause different grades to yield analytical

results exhibiting wide differences. This, however, has been shown not to be the case by Stock (*Analyst*, xix. 312), who passed a sample of ground Jamaica ginger through a fine sieve, so as to cause as much separation of the fibrous matter as possible, and then examined each portion separately, as well as some of the same sample unsifted. The result showed that this treatment had very little effect on the ash (total, insoluble, and soluble), or on the potash.

A sample of ground ginger may be regarded as genuine when it possesses the following characters :

Total ash not exceeding 5 per cent.
Soluble ash not less than 1·5 per cent.
Cold-water extract* not less than 10 per cent.

And when in addition the microscope shows its appearance to be normal.

MUSTARD.

Mustard is defined by the British Pharmacopœia as 'black mustard seeds and white mustard seeds mixed.' It has the following characters : A greenish - yellow powder, of an acrid, bitterish, oily taste, scentless when dry, but exhaling when moist a pungent, penetrating, peculiar odour, very irritating to the nostrils and eyes. A decoction cooled is not made blue by tincture of iodine. The proportion of black and white seeds to be used is not prescribed. The black mustard plant is found wild all over Europe except in the extreme north, and in many other countries. White mustard is more confined to the South of Europe, and differs from the foregoing in having the pods bristly and spreading (Fluckiger and Hanbury). The seeds are about $\frac{1}{12}$ inch in diameter, weighing about $\frac{1}{10}$ grain. There are four to six in each pod. The seeds of the black mustard are much smaller and only one fifth the weight of white mustard seeds, and are dark reddish-brown in colour when of good quality. If rain occurs while they are ripening they become grayish and lose much of their

* 2 grammes of sample to 100 cc. of cold distilled water.

pungency. Both species are cultivated in England. Mustard seed is also imported from Holland and Syria.

Mustard seed when ripe contains no starch. It contains about 35 per cent. of fixed oil, which is almost devoid of taste and smell; varying amounts of volatile oil, on which its pungent properties depend. This volatile oil (*Oleum sinapis*) is an official preparation of the British Pharmacopœia, which defines it thus: 'The oil distilled with water from black mustard seeds after the expression of the fixed oil. Characters: Colourless or pale yellow. Specific gravity 1·018 to 1·030; distils between 147° and 152° C. Dissolves readily in alcohol and ether, and to a slight extent in water. Has an intensely penetrating odour and a very acrid burning taste. Applied to the skin it causes almost instant vesication.'

Besides the above-mentioned constituents, mustard contains a high proportion of nitrogenous matter, namely, from 25 to over 30 per cent. of albuminoids calculated by multiplying the figure for nitrogen by the 6·3 factor.

Mucilage is present, particularly in the husk, in varying proportions.

The ash of mustard is about 4·5 to 5 per cent., never falling below 4·0 per cent. Mustard contains about 1 per cent. of sulphur, but its presence is not sufficiently constant to be of value in determining its purity.

The following figures were obtained by Clifford Richardson (U.S. Bulletin, xiii. 182):

	Per cent.
Water	3 to 7·0
Ash	4 „ 6·0
Volatile oil	·5 „ 2·0
Fixed oil	31 „ 37
Starch	none
Fibre	5 to 18
Albuminoids	25 „ 32

At the present time mustard is very little adulterated in this country. The addition of turmeric is not generally regarded as an adulteration. If turmeric is present, an orange-red colour is produced on the addition of ammonia. Turmeric may also be tested for by digesting 2 or 3 grammes of the sample in strong spirit and filtering. The filtrate

is evaporated to dryness; the residue is taken up with a few drops of hydrochloric acid, and is then tested with a saturated solution of boric acid. If turmeric is present, an orange-red colour is produced, turning blue or green on the addition of caustic soda.

Any addition of starch, such as is made for convenience in grinding, and for the better subsequent keeping of the finished product, is an offence under the Food and Drugs Act, unless the mustard is sold as a mixture. In the evidence taken before the Select Committee on Food Products Adulteration, it was stated by a prominent manufacturer that both pure and mixed mustards were prepared by his firm and were sold at the same price, the mixture being made, not for the purpose of increasing the profit, but to suit the public taste. It was also stated that no deleterious article was known to the witness to be added to mustard by English manufacturers, but that large quantities of coloured earth are sold from Germany to America for mixing with mustard.

In Canada, Macfarlane examined 95 samples in the year 1890. Of these he concludes that only seven can be regarded as pure, while nine he calls 'compound,' and the remaining 79 are returned as adulterated. The adulterations found are as follows: Flour, clay, gypsum, terra alba, cayenne, buckwheat, rice, and mustard husks. Some of the samples contain very little true mustard, and many of them consisted largely of 'mustard cake,' from which a large percentage of oil had been expressed.

He mentions mixed starches, buckwheat, and cayenne as being found in mustard of English manufacture, and recommends that the lowest quantity of oil (fixed oil) that should be permitted in commercial mustard should be 30 per cent. in mustard sold as pure, and 22 per cent. in mixtures. Macfarlane obtained the following figures working on genuine mustard seed, mustard husks, mustard farina, and mustard cake:

	Ash.	Moisture.	Fixed Oil.	Extracted by Alcohol.	Sulphur.	Nitrogen.
Mustard seed, white	4·98	6·58	27·01	15·64	1·09	4·07
Mustard husks „	4·42	8·20	23·66	13·74	·88	4·42
Mustard farina ...	4·27	6·90	30·50	18·46	1 14	5·16
Mustard cake ...	7·35	7·98	14·30	15·58	1·03	5·00

It would have added very greatly to the value of these analyses if estimations of the volatile oil and cellulose had been included. Charles P. Worcester (Massachusetts) in 1892 found 30 per cent. of the samples of mustard he examined to be adulterated, in 1893 27 per cent., and in 1894 31 per cent. The adulterations consisted of wheat, maize, gypsum, mustard husks, and cayenne.

Analysis of Mustard.—It is generally sufficient to examine microscopically for added starches and cayenne pepper. The amount of oil is determined by extracting with ether in a Soxhlet extractor. Supposing that mustard normally contains 30 per cent. of oil, the percentage of true mustard in a mixture is found as follows:

$$\frac{\text{Per cent. of oil} \times 100}{30} = \text{per cent of pure mustard present.}$$

If starch is seen to be present, it can be estimated by conversion into sugar ; but this must be preceded by treatment with ether and proof spirit (Allen), or the result will be invalidated owing to the production of sugar from some of the substances natural to mustard. There do not appear to be any data for judging the amount of mustard husk that should be allowed in commercial mustard. Mustards containing undue proportions of husk are said to find a ready sale in Germany, but not to be much used in this country. The price is half that of mustard of good quality. Some analyses, showing the amount of husk normally present in ground mustard seed, both white and black, would be of value, as would analyses of known mixtures of ground mustard seed with added husk.

Probably the estimation of cellulose would be the best for this purpose, as the ash, oil, nitrogen, and sulphur appear to be present in almost the same quantity as in genuine ground seeds.

Aniline Dyes are stated to be in use in America for colouring mustard which has been adulterated by the addition of wheaten flour.

The New York State Board of Health in 1883 gave legal sanction to the addition of flour or starch to mustard, provided the fact of such addition is marked legibly on the label.

SPICES.

Spices are liable to many gross forms of adulteration, particularly in the powdered condition. The adulteration takes many forms. Not only are they exhausted of their essential oil, as in the case of cloves and caraway seeds (the resulting 'drawn' or 'spent' samples being used to adulterate genuine samples), but the substitution of a cheaper spice for one of higher value is frequently practised, as in the case of pimento for cloves, cassia for cinnamon, etc. In addition to these forms of adulteration, powdered spices are adulterated with refuse of all kinds. The following substances are among those that have been found to be used for this purpose: Sand, gypsum, ground walnut and cocoa-nut shells, ground olive stones, exhausted ginger, pepper refuse, mustard husks, biscuit dust, various forms of farina, etc.

The following are the characters of the more important spices :

Cardamoms.—Cardamoms consist of the dried ripe seeds of many plants belonging to the Ginger Order. Common cardamoms are the produce of *Elettaria cardamomum*, a reed-like perennial common in the moist mountain forests of Malabar. 'Grains of Paradise' are the fruits of *Amomum Melegueta*, an allied plant of West Africa ; they have been used to give pungency to spirits, etc.

Three varieties are known to commerce : (1) 'Shorts' (official), about $\frac{1}{6}$ inch long ; (2) 'Short-longs,' about $\frac{7}{10}$ to $\frac{9}{10}$ inch long ; (3) 'Long-longs,' from $\frac{9}{10}$ to a little more than an inch in length.

Cardamoms contain about 10 per cent. of fixed oil, about 4·5 per cent. of volatile oil, an acrid resin, and about 4·0 per cent. of ash.

Caraway Seeds.—These are derived from the dried fruit of the *Carum carui*, belonging to the Natural Order *Umbelliferæ*. The chief supply comes from Holland, and occurs in commerce in the form of separated mericarps.

Caraways contain volatile oil to the extent of 4·7 to 5·4 per cent., and the ash yielded by average samples is about 8·0 per cent.

Dyer and Gilbard (*Analyst*, 1896, p. 207) have recently

called attention to the adulteration of caraways with drawn or exhausted 'seeds.'

Cinnamon and Cassia.—These spices are the barks of several species of the genus *Cinnamomum*, the true cinnamon being a native of Ceylon, where it is largely cultivated, and the cassias being derived from several other species growing in China, India, and the East Indies. Cinnamon consists of the true bark, or *liber*, of *Cinnamomum zeylanicum;* it was known in very ancient times as a spice. The crop is gathered about May and November, the two-year-old shoots being stripped and slightly fermented.

Cinnamon as it reaches the market is very thin, the outer and inner coats of the bark having been removed. Cassia, on the other hand, is thick, as it consists of the entire bark, and can be distinguished by its retaining its natural outer surface. Cinnamon is far more valuable than cassia, as there is a smaller supply, and intrinsically it contains a much larger proportion of volatile oil, and that of greater and more delicate aroma. In consequence, cassia is largely substituted for cinnamon.

Dyer and Gilbard (*Analyst*, 1895, p. 129) have called attention to the fact that powdered cinnamon is extensively adulterated with ground walnut shells. W. F. Keating Stock (*Analyst*, 1897, p. 253), has found several samples of cassia adulterated with exhausted ginger, also very abnormal amounts of mineral matter in commercial samples of ground cassia.

Cinnamon contains from 0·5 to 1·0 per cent. of essential oil, the total ash averaging 5·5 per cent.

Cloves.—Cloves are the dried calyx and flower-buds of *Eugenia caryophyllata*, an evergreen tree belonging to the Myrtle Order. Our supplies come from Zanzibar and the West Indies. Cloves are used in flavouring cordials and apple tarts and puddings.

The flower-buds of the clove-tree, carefully picked and dried, constitute the spice known by that name. Their valuable properties are due to the volatile oil which they contain, the best having as much as 16 per cent.

Cloves should, if possible, always be purchased whole, as they deteriorate less readily in that form.

The removal of the oil is so very easy that it is the commonest method of deception to do this before grinding the spice, and then to dispose of it as pure. We have

ready means of determining the loss chemically, but the microscope gives no indications. The addition of the cheaper clove-stems is also practised. The microscope reveals their presence by certain cells which they contain, which are absent in the bud.

Pimento is sometimes substituted in part or entirely, as it has a clove-like flavour, but only 4 or 5 per cent. of volatile oil. It is worth less than one-fifth the price of cloves. Its chemical composition and its structure, that of a berry, reveals its presence. The addition of the coarser adulterants, mineral matter, cocoanut shells, flour, peas, and the like, have only been observed in two instances, but no doubt frequently occur, as has been found in Canada.

Nutmeg and Mace.—These spices are different portions of the fruit of a tree known as the nutmeg-tree, *Myristica fragrans*, which grows principally on the Banda Islands, the nutmeg being the inner kernel, and the mace one of the outer coats, or arillus. They can always be obtained in their original condition, and should be so purchased. When ground they are mixed with diluents of various descriptions, principally cereals and their refuse, which are easily detected. Owing to the infrequency of the sale of the powdered nutmeg and mace, their adulteration has attracted but little attention.

The long nutmeg is the produce of *M. fatua*. The nutmeg contains about 6 per cent. of an aromatic and pungent essential oil. Mace contains about 4·5 per cent. of essential oil.

Pimento or Allspice.—Allspice or pimento is a small dry berry, the fruit of *Pimenta officinalis*, an evergreen tree of the Myrtle Order common in the West Indies. Pimento contains about 4 per cent. of an aromatic pungent oil much like that of cloves. Our supplies come wholly from Jamaica.

HONEY.

Honey consists of the saccharine substance collected by the bees (*apis mellifica*) from the nectaries of flowers, and deposited by them in the cells of the honeycomb.

The composition of honey is complex, but the essential constituent is a mixture of dextrose and lævulose, and a solution possesses the physical property of turning the plane of polarized light to the left. This property furnishes an easy and accurate method for the detection of the adulterated article, and while we have never met with a pure honey which was not lævo-rotatory, yet there are statements on record which claim that honey has been met with which was dextro-rotatory.

Honey is very largely adulterated; the substances generally used are glucose and cane-sugar. The former, on account of its low price, is the most common, and, mixed with enough of the genuine article to give it a flavour, is extensively sold as 'pure extracted honey.' One will also find a small piece of genuine comb-honey in a jar, which is filled with glucose syrup. The honey in the comb gradually diffuses itself through the mass, giving the required flavour.

Genuine honey on microscopical examination will always show the presence of pollen grains.

Examination in the Saccharimeter. — There are many forms of this instrument in use. The Soleil-Scheibler is a convenient type of instrument for the examination of honey. The 'normal weight' of this instrument is 26·048 grammes—that is to say, 26·048 grammes of pure cane-sugar (sucrose) dissolved with 100 cc. water, and a tube 200 mm. in length filled with the solution will indicate 100 on the scale. *Cane-sugar* and *glucose* will therefore indicate *plus*, and *lævulose*, or *honey*, will mark *minus* the zero. The same weight of glucose will turn the plane so far to the right (or *plus*) that it will exceed 100. The commercial glucose, when the normal weight is used, will indicate from 155 to 170, according to the greater or lesser amount of dextrine present. Pure honey will indicate from – 4 to – 15. Seldom, however, as low as – 15. It will therefore readily be seen that owing to the high dextro-rotatory

power of glucose, a comparatively small amount will neutralize the lævo-rotatory power of the honey if added. The same, of course, is true if cane-sugar syrup is added; but in this case the indication will not exceed 100, as will be the case if a sufficient amount of glucose is present.

The mode of procedure is as follows: 26·048 grammes of the honey are taken, dissolved in a flask of 100 cc., and the solution filtered through a small quantity of bone black in order to clarify the solution. A tube of 200 mm. is then filled with the solution and placed in the instrument and the instrument adjusted, the indication of the scale being noted. If *minus*, we may assume that the sample is genuine. If the indication of the scale is *plus*, however, that will indicate that either cane-sugar or glucose has been used; and if the scale indicates more than 100, the presence of glucose is conclusive, but if not we must proceed to learn which. This is accomplished as follows: A solution is prepared as stated, or 50 cc. of the original solution is taken and treated with one-tenth volume of hydrochloric acid, heated at a temperature of 80° C. for a few minutes, cooled and re-polarized. If, now, the scale still reads to the right, the presence of glucose is assured; while if to the left cane-sugar is shown to have been the cause of the original reading being to the right.

The action of the acid is to *invert* the cane-sugar—that is, to change it to a substance which no longer is dextro-, but is lævo-rotatory, and which is termed *invert* sugar, and acts in the same manner as honey. While cane-sugar can be added to a honey which will not indicate *plus*, yet practically the amount used is so great that such is not likely to be the case.

Temperature has more or less effect on the rotatory power of invert sugar; consequently all the readings of the solutions should be at a uniform temperature in order for a proper comparison.

The **Water** in genuine honey varies from 15 to 25 per cent., and the ash from 0·1 to 0·9 per cent. O. Hehner (*Analyst*, x. 217) states that the ash of genuine honey is always alkaline, whereas that of artificial glucose is invariably neutral. The addition of commercial glucose may often be detected by the turbidity produced by the addition of ammonium oxalate to a filtered solution of

the sample, due to the presence of calcium sulphate, a common impurity in commercial glucose. Artificial comb consisting at least partially of paraffin-wax is now coming extensively into use. *The detection of paraffin-wax* in honeycomb is very easy. Genuine beeswax has a melting-point of about 64° C., whereas paraffin-wax is always lower. Paraffin-wax is not affected by treatment with boiling strong sulphuric acid. Beeswax, on the contrary, undergoes carbonization.

The comb, after dissolving out the honey with water and drying, can be examined by the Koettstorfer process (see under oils). Beeswax requires from 9·2 to 9·7 per cent. of KHO for saponification. Paraffin-wax, on the other hand, is an unsaponifiable body.

LIME AND LEMON JUICE.

Lime and lemon juice contain, in addition to free citric acid, citrates, traces of organic acids other than citric, albuminoid and mucilaginous bodies, sugar, etc. The British Pharmacopœia gives the characters of lemon-juice as follows : A slightly turbid yellowish liquid, with a sharp acid taste ; specific gravity, 1·030 to 1·040 ; citric acid, 30 to 40 grains to the fluid ounce (= 7 to 9 per cent.). Should not yield more than 3·0 per cent. of ash. Raw lime-juice contains from 3·5 to 8·0 per cent. of free citric acid.

Lemon-juice is stated to contain, according to R. Warington, from 6·5 to 8·4 per cent. free citric acid. T. A. Ellwood, who has recently examined a large number of lemon-juices, found the specific gravity to be 1·038 and citric acid 8·6 per cent. ; the highest result being 9·8 per cent., the lowest 7·4 per cent. These results tend to show the Pharmacopœial standard to be too high.

The **Free Citric Acid** in lime and lemon juice is estimated by titrating about 20 cc. of the sample with $\frac{N}{2}$ NaHO, using phenol-pthalein as indicator. Each cc. of the semi-normal soda solution used will equal 0·035 of the hydrous citric acid ($H_3C_6H_5O_7H_2O$).

The **Citric and other Combined Acids** are determined as follows: The neutralized juice from the above is

evaporated to dryness ; the residue is ignited at a low as possible temperature. The ignited mass is thoroughly extracted with water, and a known volume of standard H_2SO_4 added ; the liquid is then boiled and filtered. The filtrate is then titrated with standard NaHO, using methyl-orange as indicator. The amount of sulphuric acid neutralized by the ash is equivalent to the total organic acid of the sample, as on ignition all the salts of the organic acids were converted into the corresponding carbonates ; 49 parts of H_2SO_4 neutralized=40 of NaHO=70 of $H_3C_6H_5O_7H_2O$. The result gives the *total organic acid* in the sample calculated as *citric acid*. By subtracting the free citric acid found from the total will give the *combined acid* as *citric*.

Lime and lemon-juice has been found to be extensively adulterated by the addition of sulphuric and hydrochloric acids. Sulphites, salicylic acid, and alcohol are frequent additions made for preserving purposes.

Free Sulphuric and Hydrochloric Acids may be estimated by Hehner's process as given under 'Vinegar.' Genuine lime and lemon juice contain but small traces of sulphates and chlorides.

Alcohol is estimated by distillation as given under 'Beer.'

Sulphites are best determined by oxidation to sulphates, and determining the sulphates so formed by barium chloride.

Salicylic Acid.—This should be tested for by Fe_2Cl_6. If present the sample is treated with acetate of lead to precipitate albuminous matters, filtered, and the filtrate exhausted by several extractions with ether. The ethereal solution is then evaporated, and the residue proved to be salicylic acid by the usual methods.

SUGAR.

Commercial sugar is obtained from the sugar-cane (*Saccharum officinarum*), and from the white beet (*Beta maritima*). The sugar-cane yields from 10 to 20 per cent., and the beet from 7 to 11 per cent. sugar. Sugar, as derived from these two sources, consists of sucrose ($C_{12}H_{22}O_{11}$), together with a small quantity of glucose, or

'invert sugar.' Sucrose forms large monoclinic prisms, and exercises a powerful dexto-rotatory action on a ray of polarized light. A 10 per cent. solution at the normal temperature deviates the ray 66·5° for the D line. When sucrose is heated above 160° C., it is converted into an amber-coloured body known as 'barley sugar,' which consists of a mixture of dextrose and lævulose. At a higher temperature, a reddish-brown substance is formed, known as caramel. Sucrose is soluble in half its weight of cold water, and in all proportions in boiling water.

Sugar is met with in commerce in all conditions of purity, from the brown or 'moist' sugars to the almost absolutely pure sucrose sold as 'loaf sugar,' 'white crystals,' etc. Sugar is examined by taking the moisture, ash, glucose, and tested for foreign colouring matter.

Moisture.—5 grammes is dried at 105° C. until the weight is constant. Raw sugars contain from 0·5 to 6·0 per cent. ; refined sugars below 0·5 per cent.

Total Ash.—5 grammes of the sample is ignited. The incineration of sugar is a somewhat difficult matter. It is a good plan to moisten the sugar with pure concentrated sulphuric acid before ignition. The ash of sugar varies from a trace to about 2·0 per cent. ; brown sugar sometimes contains an undue proportion of sand.

The ash of sugar consists of silica, alumina, lime, salts of potash and soda.

Glucose.—Invert sugar is estimated by Pavy's method, as described under 'Milk.' A 10 per cent., or even stronger solution is made, and this run into the boiling 'Pavy' solution until the blue colour is discharged. Raw sugar should not contain more than 2 per cent., and refined sugar not more than 0·1 per cent. of glucose.

Foreign Colouring Matter.—Aniline dyes are coming into use for colouring sugar. C. E. Cassal (*Analyst*, xv. 141) first called attention to the colouring of sugar with tropœolins and other aniline colours. Beet sugar crystals are coloured with these dyes, and sold as 'Demerara' sugar.

Since the market price of beet crystals is about 12s. per cwt., and genuine 'Demerara' 18s. per cwt., it will be seen that the substitution of artificially coloured crystals for genuine 'Demerara' is a manifest fraud.

Dyed samples will generally turn pink on the addition of hydrochloric acid to the sample. The suspected sample should be extracted with absolute alcohol; this will, in the case of dyed sugars, extract the foreign colouring matter, leaving the sugar crystals colourless. The natural colouring is not extracted by this treatment. A skein of wool, preferably slightly mordanted with aluminum acetate, is dipped into the solution, and warmed for some time in the water-bath, well washed, and dried. The skein will be dyed of a more or less marked yellow colour if artificial dyes are present. In the case of dyed sugar, the crystals, on examining with a lens, will generally be found to be very unequally coloured.

We have met with several samples of these dyed crystals in London recently. Sugar found artificially coloured should be returned as adulterated. A natural product having been tampered with by admixture with an artificial dye 'is not of the nature, substance, and quality required by the purchaser.'

INFANTS' FOODS.

The last few years have witnessed a large increase in the number of preparations made and sold for the use of infants and invalids. It is possible that the great increase of these articles is due to the fact that many of them, the constituents of which cost little, are sold at large profit.

The improvement in processes of manufacture, and the great increase of knowledge regarding the physiology of nutrition, and the relative food-values of alimentary substances, have contributed to the number and variety of foods for infants and invalids.

The materials that enter into the composition of these proprietary foods are very numerous. The most generally used are: (1) various starchy substances, as wheaten, corn, lentil, rice, and maize flours, which may be partly cooked by baking, etc.; (2) milk-powders and condensed milk, prepared from whole or skim milk; (3) sugar; (4) albumin; (5) malted preparations of starch, milk, etc.

An ideal infant's food should, when mixed with water, produce an emulsion simulating the characteristics and containing the component parts of human milk as far as possible.

For very young children no starchy foods should be used at all. Many children thrive well on sweetened condensed milk diluted with water. The unsweetened brands would probably be better, so diluted as to contain about 3·0 per cent. of fat. There are also milk-powders which possess the proper proportion of needful constituents. The 'humanized' milk preparations which are now largely coming into use have their enthusiastic supporters.

A few of the patent infants' foods contain the right proportion of nutritious material, but by far the larger quantity are composed mainly of starch, which is indigestible in the case of young children, and is the cause of the puffy and bloated appearance often mistaken for robust health.

The composition of infants' foods is so varied and complex in character that it is impossible to give any scheme of analysis that would apply to every case. Almost every preparation requires special treatment for its analysis, but much information will be obtained by ascertaining the following particulars: The amount of water, matter soluble in cold water, fat, fibre, ash, phosphoric acid, albuminoids, nitrogen.

Moisture is estimated by drying a weighed quantity at 100° C. until constant in weight.

Soluble Matter.—Five grammes is extracted by agitating with 500 cc. cold water, and allowing to stand over-night. The supernatant liquid is then siphoned off and filtered. An aliquot part is then evaporated and dried to constant weight. This residue will give the amount of soluble constituents, such as milk-sugar, maltose cane-sugar, soluble albumin, salts, etc.

The solution can then be precipitated with a slight excess of lead acetate to remove interfering bodies, such as albumin. The excess of lead acetate is removed by sulphuretted hydrogen, and the reducing power of the solution on Pavy's solution estimated. This will give the amount of milk-sugar and maltose. A portion of the solution is then 'inverted' with very dilute hydrochloric acid to convert the cane-sugar into 'invert sugar.' The reducing power of the solution is again estimated by Pavy's method and the increased reducing power calculated to cane-sugar. (See under 'Urine.')

Starch.—Starch, if present, can be estimated by drying the residue insoluble in cold water at a low temperature.

About 0·5 gramme of the residue is 'inverted' under pressure, as described under 'Cocoa'; the glucose so formed is estimated by Pavy's method and calculated to starch by dividing the amount of glucose found by the factor 0·9.

Fat.—The fat is estimated by the ether extraction process as given under 'Cheese.'

Fibre.—Wood-fibre is estimated as given under 'Pepper.' Woody fibre should be entirely absent from food preparations intended for infants.

Ash.—The total ash is estimated by igniting 5 grammes in a platinum dish at as low a temperature as possible.

Phosphoric Acid.—Phosphoric acid is estimated by the following modification of the molybdate of ammonia process. The following method was devised by W. F. K. Stock, and after an extended use of it we have found it most accurate. By its use it is possible to obtain exact estimations of the phosphoric acid in foods in a much shorter time than by the old methods.

The ash of the sample is treated with nitric acid, the solution diluted and filtered. About 20 cc. of strong ammonia is then added, then nitric acid, till the precipitate first formed is quite dissolved. Dilute ammonia is now added till a faint permanent opalescence is formed. The volume of the solution at this stage should not much exceed 70 cc. 2·5 cc. of fuming nitric acid is added, and the solution is warmed to 70° C., and 20 cc. of a 10 per cent. solution of ammonium molybdate run in with constant stirring, which is continued for some minutes. If these directions are carefully carried out, the precipitate is entirely yellow and free from the white molybdic acid. The solution is set aside to cool, when the precipitate is transferred to a small filter, washed with 25 cc. hot water, then with a like quantity of alcohol, and lastly with ether. The filter is placed in the bath to dry. The precipitate is then brushed off the paper into a watch-glass and weighed. The weight thus obtained is multiplied by the factor ·0373, which will give the amount of phosphorus as P_2O_5.

Nitrogen.—This is estimated by Kjeldahl's process on from 1 to 3 grammes, as described under 'Milk.'

Albuminoids are found by multiplying the amount of nitrogen found by the factor 6·33. This will give a very close approximation to the total amount of albuminoids or proteids present, as they contain on the average 15·8 per cent. of nitrogen.

Analyses of some Infants' Foods.

	Milk Food.	Milk Food	Lactated Food.	Cream Emulsion.	Malted Milk.	Raw Flour	Cooked Flour
Water	4·0	5·7	7·7	24·3	2·2	9·5	10·5
Fat	1·9	6·8	1·6	15·3	5·3	0·8	—
Albuminoids ...	11·0	10·5	16·0	8·2	16·9	11·3	10·5
Milk-sugar ...	9·7	5·8	27·0	43·1	50·4	—	—
Malt-sugar ...	—	—	—			—	—
Cane-sugar ...	34·6	36·0	—	—	—	—	—
Starch	33·0	30·8	33·8	—	5·6	71·9	72·3
Ash	1·4	1·2	2·6	2·6	3·1	0·5	0·4
Phosphates (P_2O_5)	—	—	0·3	—	—	—	—

BEER.

Beer is a fermented liquid prepared from malt, flavoured with an infusion of hops. This definition must be extended as follows to embrace beers as brewed at present—a fermented saccharine infusion to which has been added a wholesome bitter. The chief constituents of beer are :

(*a*) Water.

(*b*) Alcohol.

(*c*) Malt extractive, containing maltose, dextrine, albuminoid constituents, salts, etc.

(*d*) Bitter principle.

(*e*) Saline constituents from water, etc.

Strictly speaking, beer should consist wholely of the products of malt and hops ; but malt substitutes, such as malted Indian corn and rice, also glucose, technically known as 'saccharin,' prepared by the inversion of various starchy materials, are now largely used.

It is at present legal to use any saccharine or starchy materials in the brewing of beer, and as the duty is levied on the quantity of soluble saccharine matter made into beer, as estimated by the gravity of the infusion or 'wort,' the exact nature and origin of the fermentable materials employed is disregarded by the Excise authorities. The substitution of bitters other than that of the hop is not an offence under the Food and Drugs Act unless injury to health can be proved by their use.

Beers differ largely in their composition, according to the nature and quantity of material used in their manufacture, and the manner in which the fermentation has been conducted. Two methods of fermentation are in use, the 'High' and 'Low.'

The 'high' or surface fermentation is used in England. It takes place at a higher temperature, and is of shorter duration than the 'low' or 'sedimentary' process.

The 'low' or 'bottom' fermentation is largely employed in Germany. The yeast is a different variety to that used in English breweries. The beer is fermented at a low temperature, the yeast remaining at the bottom of the vat. Beers prepared by this process are highly charged with gas, and contain a much larger quantity of extractive matter and less alcohol than those prepared by the 'high' or English process. These beers are very liable to undergo a secondary fermentation unless kept at a very low temperature.

The best-known varieties of malt liquors are :

1. Pale and bitter ales, made from the finest pale malt, and bitter derived from hops in excess.
2. Mild ales, rather sweet, and containing less bitter and more alcohol than the pale ales.
3. Porter, a rather weak liquor coloured and flavoured with roasted malt, which gives it its black colour.
4. Stout, a stronger variety of porter.
5. Lager or German beers, prepared by the 'low' fermentation process, and, as before stated, containing more extractive and less alcohol than English brewed beers.

Analysis of Beer.

Alcohol.—Alcohol is estimated by direct distillation, or by the indirect method.

Direct Distillation.—About 100 cc. of the sample is rendered just alkaline with caustic soda, a little tannin added (to prevent frothing), and the liquid distilled until about 90 cc. of distillate have collected. A small piece of pumice-stone or platinum-wire placed in the distillation flask will prevent bumping. The distillate is cooled to 15·5° C., made up to the 100 cc. mark with distilled water, mixed, and the specific gravity taken with a bottle or

Westphal balance. The amount of alcohol is then found by means of the alcohol tables.

Indirect Method.—This process was devised by Tabarie. The specific gravity of the original liquid is first accurately determined. A measured quantity, say 100 cc., is then boiled until all the alcohol and other volatile matters are evaporated off. This will generally be the case if the liquid is boiled down to one-third of its original bulk. The residue is then cooled and made up to its original bulk with water, and the specific gravity taken at 15·5° C., when

$$\frac{\text{Sp. gr. of the original liquid}}{\text{Sp. gr. of the 'extract'}} = \text{Sp. gr. of alcohol evaporated.}$$

The percentage of alcohol is found by reference to the alcohol tables.

The Extract.—5 cc. of the sample is evaporated in a flat-bottomed platinum dish, and dried at 100° C. until constant in weight. The extract may also be estimated with fair accuracy from the de-alcoholized liquid obtained by Tabarie's method of determining the alcohol. The specific gravity of the 'extract' at 15·5° C. is taken, and the excess above 1,000 divided by 3·86, when the dividend is the number of grammes of dry 'extract' contained in 100 cc. of the sample.

Ash.—The ash is obtained by igniting the extract at a low temperature.

Sodium Chloride.—Common salt is determined by exhausting the ash with water, and estimating the chlorides with $\frac{N}{10}$ $AgNO_3$, using neutral potassium chromate as indicator. Each cc. of $\frac{N}{10}$ $AgNO_3$ = ·00585 NaCl or ·00355 chlorine. Salt is now but rarely added to beer in excessive amount. It is often stated that 50 grains per gallon is permitted by the Inland Revenue. This is an error, the origin of which was that the Board of Inland Revenue instructed their officers that in cases in which the chlorides in beer did not exceed 50 grains per gallon as sodium chloride, it was unnecessary to inquire into the origin of the same.

Acidity.—The acidity of beer is partly 'fixed' and partly 'volatile.' The former consists principally of lactic acid, with a little succinic acid. The volatile acid consists mainly of acetic, with traces of other acids.

Fixed Acid.—About 20 cc. of the beer is diluted to 100 cc., and evaporated down to about 50 cc. This is

again diluted, titrated with $\frac{N}{10}$ sodium hydrate, using litmus as indicator. The number of cc. of $\frac{N}{10}$ NaHO used multiplied by ·009 = fixed acid as lactic.

Volatile Acid.—20 cc. of the original sample is well diluted with water, and titrated with $\frac{N}{10}$ NaHO, using litmus as before. The number of cc. of alkali used are then noted. From this is deducted the number of cc. of NaHO required for the fixed acid. The remainder multiplied by ·006 will give the amount of volatile acid as acetic.

Determination of the Bitter Substance.—The bitter principles used to flavour beer are derived from hops, quassia, gentian, calumba, chiretta, etc. These bitters are mostly of the nature of glucosides. Noxious bitters, such as those of *Cocculus indicus* (picrotoxine), nux vomica (strychnine), and picric acid, have been stated to have been used in the past, but their occurrence now is exceedingly unlikely.

1,000 cc. of the sample is concentrated by evaporation to one-half and precipitated boiling with neutral lead acetate ; the liquid is boiled for fifteen minutes, and filtered hot. The filtrate is treated with a slight excess of sulphuric acid. The lead sulphate is filtered off, and the clear acid filtrate gently evaporated to about 100 cc. The excess of acid is removed with chalk, and the liquid filtered. The filtrate is then tasted. If it is free from bitterness, hops have been used to bitter the beer. On the contrary, if the residue is bitter, quassia or some other hop substitute has been used.

The liquid is acidified with dilute sulphuric acid and well extracted with chloroform, which is separated and evaporated. The residue, if bitter, may be due to *gentian*, *quassia*, or *calumba*.

The aqueous liquid, after extraction with chloroform, is shaken with ether. The ether will extract the bitter or *gentian*, *calumba*, and *chiretta*.

The extract left on the evaporation of the ether is dissolved in a little alcohol, hot water added, and the hot solution treated with ammoniacal lead acetate. The liquid is filtered, and the residue and filtrate treated as follows :

(*a*) The precipitate is heated with water, and decomposed with sulphuretted hydrogen. The filtered liquid is bitter in the case of *gentian*.

(*b*) The filtrate is heated with an excess of dilute sul-

phuric acid, filtered, and tasted. Bitter taste indicates presence of *calumba* or *chiretta*.

The aqueous liquid, if still bitter after extraction with chloroform and ether, is rendered alkaline with ammonia, and shaken with ether-chloroform. A bitter extract may be due to *berberine* (*calumba*) or *strychnine*. Portions of this extract may be tested as follows :

On treatment with concentrated sulphuric acid, the residue will turn olive-green in the case of *berberine*, whereas no effect is produced if *strychnine* be present.

(*a*) A portion of the residue is dissolved in HCl, on the addition of chlorine-water ; a red colour will be produced if *berberine* is present.

(*b*) On the addition of concentrated sulphuric acid, together with a trace of powdered potassium bichromate, the residue will turn blue, passing quickly to violet, and red if *strychnine* be present.

Preservatives.—Preservatives are frequently added to beer in the form of salicylic and boric acids.

Salicylic Acid may be detected as follows : About 200 cc. of the beer is concentrated by evaporation to 50 cc. at a gentle heat, cooling and extracting with ether. The ethereal solution is evaporated, and the residue dissolved in warm water. On the addition of Fe_2Cl_6, a violet coloration will be produced if *salicylic acid* be present.

Boric Acid can be tested for in the ash of the beer as described under 'Milk.' A beer 'preservative,' in somewhat extensive use, is a mixture of boric and salicylic acids prepared by dissolving salicylic acid in a solution of boric acid and borax.

Saccharine (*Benzoyl Sulphonic-imide*).—The use of this sweetening agent has been prohibited by the Commissioners of the Customs and Inland Revenue (May, 1888).

For the detection of saccharine in beer and wine, C. Schmitt recommends that 100 cc. should be acidulated with sulphuric acid, and shaken with 50 cc. of a mixture of equal measures of ether and petroleum spirit. After separating the upper layer, and agitating the aqueous liquid with another quantity of the ethereal mixture, the ether-petroleum is evaporated with a little caustic-soda solution, the residue is then carefully heated to 250° C. for a short time ; the residue is then taken up in water, and the solution tested for salicylic acid. If this is found, saccharine was present in the liquid. Of course the

absence of salicylic acid in the original liquid must be first ascertained.

Determination of the Original Gravity of the Beer Wort.—The 'original gravity' of the wort is sometimes required, as a 'rebate' or 'drawback' is allowed when beer is exported. The duty on beer is calculated from the strength of the 'wort' as indicated by its specific gravity. By the process of fermentation the specific gravity of the wort is diminished to an extent dependent upon the amount of alcohol formed. The weight of alcohol being approximately half of the saccharine matters destroyed by the fermentation, it is evident that a determination of the alcohol in the fermented liquid would give the means of ascertaining the quantity of sugar destroyed, and hence of making the necessary correction for the reduction in the density of the wort caused by the fermentation.

The alcohol is first carefully estimated by the direct distillation process. The specific gravity of the distillate is deducted from 1000, the difference is the *spirit indication*. Now find from the following table the number of degrees of 'gravity lost'; thus, suppose the spirit indication is 11·5, take 11 in the first column, and under ·5 in the seventh column we find 51·7 equal to 'gravity lost.'

Degrees of Spirit Indication.	·0	·1	·2	·3	·4	·5	·6	·7	·8	·9
0	—	·3	·6	·9	1·2	1·5	1·8	2·1	2·4	2·7
1	3·0	3·3	3·7	4·1	4·4	4·8	5·1	5·5	5·9	6·2
2	6·6	7·0	7·4	7·8	8·2	8·6	9·0	9·4	9·8	10·2
3	10·7	11·1	11·5	12·0	12·4	12·9	13·3	13·8	14·2	14·7
4	15·1	15·5	16·0	16·4	16·8	17·3	17·7	18·2	18·6	19·1
5	19·5	19·9	20·4	20·9	21·3	21·8	22·2	22·7	23·1	23·6
6	24·1	24·6	25·0	25·5	26·0	26·4	26·9	27·4	27·8	28·3
7	28·8	29·2	29·7	30·2	30·7	31·2	31·7	32·2	32·7	33·2
8	33·7	34·3	34·8	35·4	35·9	36·5	37·0	37·5	38·0	38·6
9	39·1	39·7	40·2	40·7	41·2	41·7	42·2	42·7	43·2	43·7
10	44·2	44·7	45·1	45·6	46·0	46·5	47·0	47·5	48·0	48·5
11	49·0	49·6	50·1	50·6	51·2	51·7	52·2	52·7	53·3	53·8
12	54·3	54·9	55·4	55·9	56·4	56·9	57·4	57·9	58·4	58·9
13	59·4	60·0	60·5	61·1	61·6	62·2	62·7	63·3	63·8	64·3
14	64·8	65·4	65·9	66·5	67·1	67·6	68·2	68·7	69·3	69·9
15	70·5	71·1	71·7	72·3	72·9	73·5	74·1	74·4	75·3	75·9

This figure is added to the specific gravity of the de-alcoholized beer obtained as described under Tabarie's method of determining alcohol. The result is the 'original gravity' of the wort.

Example—	1000·0
Specific gravity of alcoholic distillate	993·6
Spirit indication=	6·4
= Gravity lost (see table)	26·0
Specific gravity of de-alcoholized beer	1014·2
Original gravity of wort=	1040·2

Analyses of some Typical Beers by Various Authorities.

	Sp. Gr. at 15·5° C.	Alcohol (% by Weight).	Extract.	Ash.	Acidity as Acetic.	Albu-minoids.
Bitter Ale	—	5·4	5·4	—	0·1	0·16
Burton Mild ...	—	5·4	5·1	—	0·1	0·21
Burton Mild ...	—	6·8	6·7	—	0·2	0·26
Burton Pale ...	1·0106	5·3	5·1	—	0·2	—
Scotch Pale... ...	—	8·5	10·9	0·3	0·2	0·74
Bass's Pale	1·0138	6·2	7·0	—	—	—
Alsopp's Pale ...	1·0144	6·4	4·4	—	0·3	—
Stout, Guinness X.	—	6·6	7·2	—	0·2	—
Stout, Guinness XX.	1·0244	5·0	5·5	—	0·2	—
Porter	1·0207	5·4	6·0	—	—	—
Porter, Barclay ...	—	5·4	6·0	0·4	0·2	—
Lager, Pilsen ...	1·0130	3·5	5·1	0·2	—	—
Lager, American ...	1·0162	2·8	6·0	0·3	0·3	0·45
Lager, Munich ...	1·0110	5·1	5·0	0·2	—	0·83

WINE.

Wine is the fermented juice of the grape. In the preparation of wine the grapes are generally separated from the stalks, and then placed in a press and the juice expressed. The juice or 'must' is then placed in vats, in which the fermentation takes place spontaneously. The addition of yeast is unnecessary, as the requisite amount exists naturally on the skins of the grape. The juice before fermentation contains about 20 per cent. of solid matter, of which from 12 to 18 per cent. consists of glucose, the remainder containing albuminous matters, potassium-hydrogen tartarate, calcium tartarate, traces of gum, colouring matter, mineral matter, etc. During the fermentation the glucose is converted into alcohol and carbon dioxide. After the fermentation has ceased, the wine is separated from the 'lees' or residue, which consists largely of yeast-cells and potassium-hydrogen tartarate, and run off into casks, in which an after-fermentation takes place, resulting in a further deposit of tartarates, etc. The wine is then drawn off into fresh casks, in which it is kept for various periods to 'age' or 'mature.'

Wines may be divided into various classes as follows, according to the country of their production: French—clarets, burgundy, champagne; German—Rhine, Moselle; Spanish—port, sherry, madeira; Italian, Hungarian, Greek, Cape, Californian, and Australian, etc.

Wines may be classed broadly into red and white, also by their general characteristics 'dry' and 'sweet.' The 'dry' wines, such as Burgundy, Rhine, Moselle, and Gironde, etc., contain no sugar and a comparatively large amount of acid. The 'sweet' wines, as port and madeira, contain a considerable amount of undecomposed sugar.

The nature of wine is largely influenced by the place of growth, climate, temperature, in addition to the amount of glucose and acids contained in the original grape-juice.

The proportion of sugar and acid best adapted for the production of wine is 40 to 1. If these conditions do not obtain in the natural juice, additions are made so as to satisfy these requirements. If the acid is in excess, the 'must' is diluted and the necessary amount of sugar added

in the form of glucose or cane-sugar. This process is largely used in the champagne country.

The sweet wines are frequently 'fortified' by the addition of alcohol, generally in the form of brandy ; this is to prevent the unfermented sugar from undergoing subsequent fermentation. The proportion of alcohol in 'fortified' wines is sometimes as high as 22 per cent., but in natural wines it never exceeds 13 per cent.

By the English Customs regulations 10 per cent. of brandy is allowed to be added to wines in bond, while in France the sophistication is permitted in wines intended for export, provided the total amount of alcohol does not exceed 21 per cent.

The total solid matter of wines ranges from 1·8 to 3·4 per cent. ; the mineral matter from 0·35 to 0·15 ; the acidity, as tartaric acid, from 1·0 to 0·5 ; glycerine from 1·3 to 0·6 ; phosphoric acid from 0·06 to 0·02 per cent.

The 'plastering' of wines consists in adding calcium sulphate (plaster of Paris) to the 'must' or the wine. This addition is claimed to add to the keeping properties of the wine by removing any excessive acidity. This addition is very detrimental, as it gives rise to the formation of free sulphuric acid and acid sulphates, as well as calcium tartarate and potassium sulphate. The calcium salt, being insoluble, is deposited with the 'lees' ; the potassium sulphate remains in solution, and as it exerts a decidedly purgative action on the body, its presence cannot fail to be detrimental. In France, the sale of wine containing over 0·2 per cent. of potassium sulphate is prohibited. The plastering of wines is chiefly carried on in Spain, Portugal, and the South of France. The ash of pure wine does not exceed 0·35 per cent., but in samples of sherry often met with it reaches 0·5 per cent., and is composed almost entirely of sulphates.

Many of the Continental wines are mixed or blended, especially for export purposes ; this probably constitutes the most frequent form of adulteration. Natural wines of the same manufacture vary to some extent from year to year in colour, flavour, and other characteristic properties, so mixing is resorted to in order to supply the trade with a product always possessing the same qualities. In many cases, the flavour of the wine is improved by blending, and also the intoxicating effect increased, both results being due to some extent to the compound ethers formed.

From the chemical point of view, the most important constituents of wine are the primary constituents of the fermentation, such as the alcohol, glycerine, acids, etc.; but the commercial value is far more dependent on the flavour and bouquet, which are due to the compound ethers, or 'esters,' of acetic, caproic, caprylic, tartaric, and other organic acids. The total amount of ethers is very small, 0·3 per cent. at the maximum. 'Pasteuration,' as largely used in France, is a process to effect the artificial ageing, and to promote the keeping qualities of the wine. This is accomplished by subjecting the bottles of wine to a temperature of from 50° to 100° C. for several hours. Wines which exhibit ropiness and other diseases are restored by this treatment, which causes the destruction of the disease organisms.

Pure cultures of the various micro-organisms procured from the high-class vintages are now largely coming into use to improve the lower grade wines, and also to give to fictitious wines some of the flavour and bouquet of the genuine article.

It is stated that over 100,000,000 gallons of fictitious wine was manufactured in France during last year (1894). The greater part of this is made from dried raisins imported from Spain and the Levant. F. Schaffer (*Zeits. Anal. Chem.*, xxiv., p. 559) has published the following analysis of these artificial wines:

	A.	B.	C.
	Per cent.	Per cent.	Per cent.
Alcohol	8·05	9·55	7·02
Extract	2·39	1·96	1·80
Sugar	0·33	0·41	0·32
Ash	0·21	0·13	0·16
Acidity (as tartaric) ...	0·74	0·50	0·77
Cream of tartar ...	0·26	0·23	0·47
Phosphates (as P_2O_5) ...	0·02	0·01	0·02

So-called 'unfermented wines' are now extensively sold in the temperance interest and for sacramental purposes. They generally consist of clarified fruit juices preserved with salicylic and boric acids, often with the addition of saccharine.

The Analysis of Wines.

Specific Gravity.—This is taken at 15·5° with a bottle or the Westphal balance.

Alcohol.—Alcohol is estimated by direct distillation as given under 'Beer.' It is better to take 50 cc. and dilute to 100 cc. with water, and multiplying the indicated amount of alcohol as given by the tables by 2 to obtain the percentage of alcohol. The indirect method of Tabarie can also be used. (See under 'Beer.')

Extract.—This is estimated by drying 5 grammes as described under 'Beer.' A very close approximation can be obtained as follows: From the specific gravity of the de-alcoholized liquid is subtracted 1000; the remainder divided by 4·6 will give the number of grammes of extract per 100 cc. Another method of calculating the total solids, which is preferable in the case of very sweet wines, is to divide the difference between the specific gravity and 1000 by 3·86 instead of 4·6, and subtract the percentage of ash from the figure so obtained. This method is based on the assumption that the organic solids of wine have the same solution density as extract of malt, and that the mineral matters have twice that density.

Sugar.—The sugar of natural wine consists wholly of glucose. It is estimated by boiling off the alcohol and removing the colouring and other reducing bodies by a slight excess of basic lead acetate, filtering, removing the excess of lead by suitable means, and the reducing power of the liquid on Pavy's solution estimated. 100 cc. of light wines, or 50 cc. of sweet wine, treated as above and diluted to 200 cc., will give solutions of suitable strength for the above test.

Ash.—The ash is obtained by igniting the dried residue at a low temperature.

Phosphoric Acid.—This is estimated on the ash by Stock's process as described under 'Infants' Foods.'

Acidity.—The acidity of wine exists both as fixed and volatile. The former consists principally of tartaric, the latter of acetic acid.

Fixed Acid.—This is determined by diluting 20 cc. of the sample with water, boiling down to a low bulk, again diluting, and boiling down as before. The residue is taken up in water, and titrated with $\frac{N}{10}$ NaHO, using phenol-

phthalein as indicator. The number of cc. of soda used multiplied by ·0075=*fixed acid as tartaric.*

Volatile Acid.—20 cc. of the original wine is well diluted with water, and titrated with $\frac{N}{10}$ NaHO, using phenol-phthalein as before. The number of cc. of alkali used in the case of the fixed acid is deducted from the result. The remainder multiplied by ·006=*volatile acid as acetic.*

Free Tartaric Acid and Potassium Bitartrate.—In the presence of a small amount of free acids—*i.e.*, acids other than tartaric—the detection of a considerable amount of free tartaric acid may fairly be considered as strong evidence that the wine is artificial. Nessler recommends the following qualitative test: 20 cc. of the sample is repeatedly shaken with a little freshly and finely ground cream of tartar. After standing one hour, the solution is filtered, 3 or 4 drops of a 20 per cent. solution of potassium acetate is added, and the mixture is allowed to remain at rest for twelve hours, when, in presence of free tartaric acid, a precipitation will take place. The quantitative estimation of free tartaric acid and potassium bitartrate is made by Berthelot's method as follows: Separate portions of the wine (20 cc. each) are introduced into two flasks, a few drops of 20 per cent. solution of potassium acetate being added to the second flask. 200 cc. of a mixture of equal parts of alcohol and ether are then added to both flasks; their contents are repeatedly shaken, and finally set aside for eighteen hours at a temperature between 0° to 10° C. The separated precipitates are now washed with the ether-alcohol mixture, and then titrated with $\frac{N}{10}$ NaHO solution. That formed in the first flask corresponds to the *potassium bitartrate* originally contained in the wine. The second represents the *total tartaric acid* present. The addition of a small quantity of clean sand will assist in the separation of the precipitates.

Detection of Foreign Colouring Matter.—Many processes and tests have been published from time to time to detect foreign colouring in wine, but very few of them are of any practical use. A very simple method has been devised by Dr. Dupré to determine the presence or otherwise of foreign colouring in wine. A 10 per cent. solution of the best transparent gelatine is prepared, which is then run into a mould to set. The hard jelly is then cut up into

small cubes about $\frac{3}{4}$ inch square. Two or three of these are placed in the suspected sample of wine. After standing in the wine for twenty-four hours the cubes are removed, washed with a little cold water, then cut in half. On examining the section, in the case of genuine wines, the colouring matter will not have penetrated more than about $\frac{1}{16}$ inch. The majority of the foreign colourings added to wine, such as *fuchsine*, *cochineal*, *logwood*, *litmus*, *beetroot*, *Brazil-wood*, *indigo*, etc., penetrate the gelatine to the centre of the cubes. Dilute ammonia will dissolve from the stained cake the colouring matter of *logwood* and *cochineal*. *Alkanet* is the only colouring matter in general use which resembles the natural colouring of wine, in the slow rate in which it diffuses into the jelly, hence if no coloration of the interior of the jelly be observed, *alkanet* is the only foreign colouring matter likely to be present. Ammonia changes the red colouring matter of *alkanet* root to a beautiful blue.

A test in use in the Paris Municipal Laboratory is as follows: A few drops of the wine are dropped on to a piece of recently calcined lime; the following colours are stated to be produced: *Natural red wine* gives a yellowish-brown coloration; wine coloured with *fuchsine* or *Brazil-wood* gives a rose colour; wine coloured with *logwood* gives a reddish-violet colour.

If ammonium hydroxide be added to the suspected sample of wine till a distinct alkaline reaction is obtained, then a little ammonium sulphide, and the liquid filtered, the filtrate from genuine wine will possess a green tint, whereas that obtained from artificially coloured wine will exhibit other colours, such as red, violet, brown, etc.

Logwood may be detected as follows: 20 cc. of the sample is well shaken with about 2 grammes of finely-powdered manganese dioxide, and treating the filtered liquid with zinc and hydrochloric acid, which destroys the brown coloration of the oxidized logwood. The colourless and neutralized liquid, if logwood be present, gives a red-violet colour with lime water, and a violet with ammonium molybdate in a solution slightly acid with nitric acid.

Rosaniline salts (*fuchsine* and *magenta*) may be detected according to A. H. Allen by rendering 50 cc. of the wine slightly alkaline with ammonia, and boiling the

liquid with a little white wool till all the alcohol and ammonia are expelled. The wool is then removed, washed, and at once heated with a few drops of solution of soda till dissolved. After cooling, about 5 cc. of water and the same measure of alcohol are added, and the liquid is shaken with 10 cc. of ether. On separating the ethereal layer and adding to it a drop of acetic acid, a red or pink colour will be developed if a mere trace of rosaniline be present. For the detection of a somewhat larger quantity, it is sufficient to render the wine alkaline with ammonia, agitate with ether, and shake the separated ethereal solution with dilute acetic acid, when a red coloration will be produced.

For the detection of *aniline colours* in wines and fruit-juices, etc., C. O. Curtman (*Zeitschr. f. Anal. Chem.*, H. 4) has published the following test, dependent upon Hofmann's isonitril reaction: To 4 cc. of the wine are added 4 cc. of potash solution and 2 drops of chloroform. After first gently warming and subsequent boiling, the characteristic smell of isonitril is plainly perceptible. The *sulpho-compounds of rosaniline* give the reaction only after some time. The test may be rendered more delicate by finally adding a little sulphuric acid. The small quantities of compound ethers present in the wine do not interfere with the delicacy of the test. The test is also successful with *aniline blue*, *purple*, *violet*, *magenta*, *red*, and many *yellow* and *green aniline colours*.

Preservatives.—Both salicylic and boric acids are frequently added to wine to prevent after-fermentation.

Salicylic Acid.—This is detected as given under 'Beer,' or by the following test devised by Curtman (*Jour. Pharm.*, xiv. 523): To 4 cc. of the wine (or beer) is added 2 cc. of methyl alcohol and 2 cc. of sulphuric acid. Shake the mixture, heat gently for two minutes, then allow to cool. Next heat to boiling, when, if salicylic acid is present, the odour of oil of wintergreen (methyl salicylate) will be perceptible.

Boric Acid.—Test the ash of the wine by the flame coloration test as directed under 'Milk.' Boric acid is stated by many anthorities to be a normal constituent of wine. Recently Professor Rising, of San Francisco, found it to exist in traces in Californian wines, whilst Ripper found it in 1,000 samples of German and other wines.

G. Baument (*Berichte*, December 10, 1888) found traces of boric acid to be present in a large number of genuine French, German, and Spanish wines.

Saccharine (*Benzoyl Sulphonic-imide*).—Extensive use of this sweetening agent is made on the Continent. It can be detected by Schmitt's process as given under 'Beer.'

The following are some of the conclusions arrived at by a commission appointed by the German Government to inquire into uniform methods for wine analysis, and to establish standards of purity :*

(*a*) After deducting the non-volatile acids, the extract in natural wine should amount to at least 1·1 grammes per 100 cc. ; after deducting the free acids, to at least 1 gramme per 100 cc.

(*b*) Most natural wines contain 1 part of ash to 10 parts of extract.

(*c*) The free tartaric acid should not exceed one-sixth of the total non-volatile acids.

(*d*) Genuine wines seldom contain less than 0·14 gramme of ash, nor more than 0·05 gramme of sodium chloride per 100 cc.

At the Paris Municipal Laboratory the following standards are adopted : The amount of added water in all wines, not sold as of a special or abnormal character, is calculated on a basis of 12 per cent. of alcohol (by volume), and 2·4 per cent. of extract.

The proportion of potassium sulphate must not exceed in unplastered wines 0·06 per cent. The use of preservatives is prohibited.

R. Borgman gives the following average relations of the various constituents of pure wine :

Alcohol	: Glycerine	=	100 : 10·5
Extract	: Acidity	=	1000 : 16·6
Acidity	: Ash	=	10 : 3·4
Ash	: Extractives	=	1 : 11·2
Phosphoric acid	: Ash	=	1 : 6·8

From the investigations of Dr. Dupré, it would appear that in genuine unfortified wines the amount of alcohol varies from 6 to 12 per cent. Fortified wines, in which

* *Reichsanzeiger*, 1884, No. 154.

fermentation has been checked by the addition of alcohol, often contain 5 per cent. of sugar ; champagnes usually show from 4 to 10 per cent. of sugar.

Analyses of Typical Wines obtained by Various Authorities.

	Specific Gravity at 15·5° C.	Alcohol Percentage by Weight.	Extract.	Sugar.	Ash.	Phosphoric Acid as P_2O_5.	Fixed Acid as Tartaric.	Volatile Acid as Acetic.	Real Tartaric Acid.
Red French ...	—	8·5	2·4	—	0·25	0·30	—	—	—
Red French ...	·9950	12·0	2·4	0·2	0·22	0·05	0·42	0·17	0·18
White French	—	9·4	2·5	—	0·26	0·30	—	—	—
White French	·9920	10·8	1·3	0·9	0·20	0·03	0·43	0·17	0·10
Vin Ordinaire	—	7·0	5·0	0·1	0·45	—	0·61	0·11	—
St. Julien ...	—	9·8	2·7	0·3	0·40	0·08	0·51	0·14	—
Champagne ...	—	7·9	12·4	10·6	0·30	0·05	—	—	—
Rhenish ...	·9934	9·2	1·9	0·1	0·20	0·03	0·42	0·11	0·25
Moselle ...	—	8·0	2·1	—	0·22	0·05	—	—	—
Hock	—	8·8	2·3	—	0·20	0·04	—	—	—
Sherry ...	·9979	17·2	5·3	3·0	0·50	0·20	0·52	—	—
Sherry ...	9940	17·2	4·2	2·5	0·40	0·02	0·27	0·15	0·18
Port	·9974	17·5	5·4	2·3	0·30	0·03	0·50	—	—
Port	·9869	18·3	3·1	1·0	0·20	0·03	0·40	—	—
Port	1·1004	18·5	7·5	4·3	0·30	0·05	0·31	0 08	0·22
Madeira ...	·9939	16·7	5·0	2·1	0·40	0·04	0·54	—	—
Marsala ...	·9966	17·5	5·4	3·2	0·20	0·02	0·32	—	—
Greek... ...	·9931	13·9	2·5	0·4	0·40	0·04	0·23	0·18	0·30
Hungarian ...	·9921	8·5	1·8	0·0	0·20	0·02	0·53	0·15	0·07
Californian ...	—	10·4	2·1	0·0	0·20	0·02	0·48	0·08	—
Californian ...	—	9·8	2·1	0·1	0·20	0·02	0·41	0·10	—

SPIRITS.

Spirits are liquids prepared by distilling alcohol containing liquors. Various grains, such as wheat, Indian corn, barley, fruit-juices, etc., may be made to yield alcoholic

liquids by fermentation. When the alcoholic liquid so obtained is distilled, the distillate constitutes a 'spirit.'

Brandy.—Cognac, or French brandy, is prepared by the distillation of wine. An inferior variety is made from the 'marc' of grapes, consisting of the skins and stalks. The characteristic taste and bouquet of the original wine are to a considerable extent communicated to the resulting brandy, and on these qualities its value depends. 'British' brandy is manufactured from grain spirit, flavoured with ethyl acetate, nitrite, etc., oils of cassia, cloves, tincture of allspice, capsicum, etc.

When brandy is first distilled it is colourless, its amber tint being due to the casks in which it is stored. The constituents of brandy are water, alcohol, traces of various ethers, aldehydes, and acids, chiefly acetic. The specific gravity is usually about ·930. Total solids about 1·0 per cent.

Whisky. — Whisky is prepared from grain, mostly barley, both malted and raw. The distillation of this spirit is largely carried on in Scotland and Ireland. The smoky flavour of whisky is due to the fact that the malt used has been dried in kilns in which peat has been used for fuel. Whisky usually contains a trace of free acid, but this rarely exceeds 0·1 per cent. as acetic acid. Whisky when first distilled is colourless, but by storing in sherry casks, the usual method employed to give flavour, it acquires colour, and takes up traces of sugar, tannin, etc. The total solids rarely exceed 0·15 per cent.

Rum.—Rum is obtained by the distillation of the fermented juice of the sugar-cane or molasses. The distillation is carried on in the West Indies, North America, etc., but a great deal of this article is made from grain spirit, flavoured with various ethers and essential oils. The characteristic odour of rum is due to ethel butyrate. The specific gravity ranges from ·874 to ·926 ; alcohol from 50 to 70 per cent. ; total solids from 0·7 to 1·5 per cent.

Gin is a spirit originally prepared from grain, flavoured with juniper-berries, oil of juniper, coriander-seeds, turpentine, capsicum, etc., with or without the addition of cane-sugar. Hollands and Schnapps are varieties of gin.

Analysis of Spirits.

The most frequent form of adulteration of spirits is the addition of water. The alcoholic strength and total solids

should be determined. Methylic alcohol should be tested for, also fusel oil, to which may be traced the detrimental physiological effects of unmatured spirits. The keeping of spirits in wood greatly improves them, owing to the conversion of the fusel oil into other bodies. Caramel (burnt sugar) is added to spirits for colouring purposes. It may be detected by its bitter taste, and the reducing power it exerts on Fehling's solution.

Alcohol.—This is determined by direct distillation, as directed under 'Beer.' It is best to dilute the sample with an equal bulk of water before distilling, to guard against loss of alcohol caused by distilling too concentrated a liquid. By the Sale of Food and Drugs Amendment Act of 1879, the minimum limit of strength for brandy, whisky and rum was fixed at 25° under proof (= 75 per cent. of proof spirit). For gin the limit was fixed at 35° under proof (= 65 per cent. of proof spirit).

Methylic Alcohol.—This is detected by the Riche and Bardy test, which is performed as follows : Mix together in a flask 10 cc. of the sample, 15 grammes of iodine, and 2 grammes of amorphous phosphorus, and distil off the methyl and ethyl iodides formed into 30 cc. of water. Separate the heavy oily drops from the water, and mix with 5 cc. of aniline in a flask kept cool ; after an hour add some water, and an excess of soda solution, and boil. An oily layer rises to the top, and 1 cc. of this is mixed with 10 grammes of a mixture of 100 parts of clean sand, 2 of common salt, and 3 of cupric nitrate ; place the mixture in a glass tube, and heat for eight hours at a temperature of 90° C.; then exhaust with warm alcohol, filter, and make up to 100 cc. with alcohol. If the sample is pure, the alcoholic liquid is red ; but if as little as 1 per cent. of methyl alcohol is present, the liquid has a distinct violet colour (due to methyl aniline violet), which is deeper according as the percentage of methyl increases. The presence of the aniline violet is corroborated by diluting a portion of the liquid with 2,000 times its volume of water, and immersing some white wool in it for half an hour. If the sample contained methylic alcohol, the wool takes on the violet colour, the depth of tint giving a fair approximate indication of the proportion of methylic alcohol present. In the case of methylic alcohol being found to be present,

the skein of wool is compared with a standard set of skeins made from mixtures containing known percentages of methylic alcohol.

The above process, although reliable, is very tedious.

Fusel Oil (*Amyl Alcohol*) is best detected as follows : About 100 cc. of the spirit is very slowly distilled at a low as possible temperature to distil off the greater part of the alcohol. The residue in the flask is cooled and extracted with ether. If the separation does not take place spontaneously add an equal bulk of water. The ethereal solution is then allowed to evaporate at the ordinary temperature. To portions of the residue are applied the following tests : (*a*) Heat with sulphuric acid and a little potassium bichromate. The odour of valerianic acid is evolved if fusel oil is present. (*b*) Warmed with about double its volume of concentrated sulphuric acid : a violet-red colour is produced, and amyl-sulphuric acid is formed. (*c*) Heated with sulphuric acid and acetate of soda, the odour of jargonelle pear (*acetate of amyl*) is evolved.

The quantitative determination of fusel oil is by no means an easy matter. A very ingenious process has been devised by Marquardt.* The method is based on the extraction of the fusel oil by chloroform, the oxidation of the amylic alcohol to valeric acid, the conversion of the latter to barium valerate, and the estimation of the barium thus combined.

A. H. Allen, who has investigated this process, recommends the following procedure :

150 cc. of the sample is diluted with water to a specific gravity of about ·980, and agitated with 50 cc. of pure chloroform for a quarter of an hour. The aqueous layer is separated and shaken with another 50 cc. of chloroform, and subsequently treated a third time. The 150 cc. of chloroform, containing in solution the amylic alcohol of the spirit, is treated in a strong flask or bottle with 2 grammes of sulphuric acid and a solution of 5 grammes of potassium bichromate in 30 cc. of water. The flask is then closed, and kept at a temperature of 85° C., with frequent agitation, for six hours. The liquid is then distilled till all but 20 cc. have passed over, when 80 cc. of water is added to the residue, and the distillation continued till only 5 cc. remain in the flask. The distillates

* *Berichte*, 1882, pp. 1370, 1661.

are digested for half an hour with barium carbonate, in a flask fitted with an inverted condenser, after which the chloroform is distilled off and the aqueous liquid evaporated to a volume of 5 cc. The solution is then filtered from the excess of barium carbonate, and the filtrate evaporated to dryness at 100° C. The residue ('A') is weighed, dissolved in water, and the solution diluted to 100 cc.; 50 cc. of the solution is acidulated with nitric acid and precipitated by silver nitrate, the resultant silver chloride being collected, weighed, and calculated into its equivalent of chlorine (143·5 of AgCl=35·5 of Cl). The remaining 50 cc. is precipitated with dilute sulphuric acid, the barium sulphate being collected, washed, and weighed. The weight found is calculated into its equivalent of barium (233 of $BaSO_4$=137 of Ba). The sum of the weights of the barium and chlorine found, subtracted from that of the residue ('A'), gives the weight of the valeric radicle contained therein, and this multiplied by the factor 0·871 gives the weight of amylic alcohol in the 150 cc. of spirit employed for the operation. The errors produced by the presence of substances in the fusel oil other than amylic alcohol tend to compensate each other, and hence the results are very fairly accurate. The chloroform for this process is best prepared from chloral, as the ordinary kind, though it may not colour sulphuric acid, is apt to contain impurities, which yield valeric acid and other volatile fatty acids by oxidation. It is always best to do a 'blank' experiment upon pure alcohol.

Spirits should never contain more than 0·1 per cent. amylic alcohol.

POISONOUS METALS IN FOODS.

The poisonous metals which have occasionally to be sought for in foods are lead, tin, copper, and zinc.

Lead may be sometimes found in canned articles of food, in beverages, and in drinking water.

Tin has been found in varying quantities by different observers in canned articles, particularly in the case of acid fruits such as cherries and currants.

Copper may be found in preserved peas, French beans, pickles, and the mixed vegetables known as 'macédoines.'

Zinc is frequently found in dried apples, having been derived from the galvanized wire on which the apples are dried. Zinc salts are also sometimes added in the manufacture of cheese (A. H. Allen and Hudson Cox, *Analyst*, 1897, 187), though it is probable that the practice is now nearly extinct. Zinc has also been found in drinking water, having been taken up by the water in passing through galvanized iron pipes.

It will seldom happen that mixtures of these metals will be present, but if it is considered necessary to search for all of them, the scheme given by A. H. Allen ('Commercial Organic Analysis,' vol. iv., p.295) provides for the recognition of all or any of them. He lays stress on the fact that the oxide of tin is by no means readily soluble in acids, and advises that fusion with an alkali should be always resorted to.

Sampling.—After opening the sample, the *whole contents* should be pulped or mixed, and the estimation should be done on a portion of this mixture. This procedure was discussed (*Analyst*, xxii. 145), as some analysts are in favour of separating the liquid and solid portions of the sample before making estimations.

In the case, however, of canned cherries, currants, etc., it is probable that any heavy metals would be mainly in the liquid, and an additional reason for examining the syrup would be that many people would use it for food together with the fruit.

In the case of nearly all canned articles, it is possible by using a very moderate amount of violence, to detach small beads of solder from the inside of the tin. Such fragments should be searched for and removed, as it would be obviously unfair to dissolve and estimate them along with any metal actually in chemical combination, and a small fragment of solder in, say, 10 or 20 grammes of sample would show a very much higher figure than is ever likely to be obtained from metal really in a state of combination with the sample.

As an illustration, if two No. 5 shot were contained in a snipe, and were ingested together with the flesh of that bird, the lead would bear the proportion to the flesh of about 20 grains to the pound—a quantity that would rightly be condemned if in combination, but which in the metallic state is probably without action.

Attention was first drawn to the fact that tin was often present in canned foods by E. A. Menke in 1878 (*Chem. News*, xxxviii. 971). In 1880 Otto Hehner (*Analyst*, v. 218) found tin in a considerable number of samples of canned fruit, vegetables, and meats. He also found tin in mineral water supplied in siphons.

In 1883 W. Blyth found tin in twenty-one samples of canned fruit, in amounts varying from $1\frac{1}{2}$ grain to 11 grains per lb., with a mean of 4·5 grains. He considered the action mainly due to the acid juices of the fruit.

Dr. F. L. Teed (*Analyst*, xvii. 142) read a paper on the detection and estimation of small quantities of lead in presence of copper and iron. These three metals often occur together in aerated beverages. Dr. Teed takes advantage of the fact that sulphide of copper is soluble in potassium cyanide, while sulphide of lead is not. The liquid to be tested is placed in a cylinder, and a few cc. of ammonia and a little potassium cyanide are added, and then a small quantity of ammonium sulphide. Any coloration produced is due to lead, copper and iron not interfering. This method is of especial value when the ordinary method of precipitating lead as chromate cannot be employed, as in liquids containing tartaric and other vegetable acids or organic matter, which have a reducing action of potassium bichromate.

Aerated water *not* supplied in siphons may contain lead, and different bottles often contain varying amounts, so that when samples are taken, if three bottles are purchased they should be opened, mixed in a jug, poured back into the bottles and sealed up.

In testing *drinking waters* for lead, the most convenient plan is to place 100 cc. of the sample in a Nessler glass and pass sulphuretted hydrogen till the liquid smells strongly of it. If a dark colour is produced which does not disappear with a few drops of hydrochloric acid, it is due to lead (or copper), and can be confirmed by the potassium chromate method.

The tint produced by sulphuretted hydrogen may be compared against standard solutions of lead acetate.

The delicacy of both these methods is very considerable, and in each case it is possible to detect $\frac{1}{50}$ grain of lead per gallon, provided that in the sulphide method the

liquid is placed in a tall cylinder, and in the bichromate method is allowed to stand twelve hours till the precipitate has had time to settle.

Estimation of Copper in Peas, Pickles, etc.—When copper alone is present, its amount may be estimated most conveniently by electrolysis.

In examining peas, beans, or pickles for copper, plenty of H_2SO_4 should be employed during the charring, and if a platinum dish is used this is specially necessary, as otherwise the copper salts may be reduced to metallic copper, which will readily alloy with and penetrate a platinum dish, while it is, of course, well known that a fragment of lead or tin has the same effect.

A large quantity of the sample is ashed, and the ash treated with sulphuric acid, diluted, filtered, and made up to 40 cc., and transferred to a platinum crucible capable of holding 70 cc. The crucible stands on a piece of platinum foil connected to the zinc of a single Grove's cell, while a platinum spatula is clamped so as to be in the centre of the liquid in the crucible, but not to touch its sides ; the spatula is connected to the platinum of the battery. When charged and connected, the battery should be left to run all night.

It can be seen whether the current is passing by the minute bubbles of oxygen which rise from the platinum spatula.

In the morning the action should be complete, and if the liquid is free from copper (a single drop spotted on a tile failing to give a brown or black colour with sulphuretted hydrogen), the contents of the crucible are washed out with recently boiled distilled water, and then with spirit and ether, and dried a few minutes and weighed. The film should be coherent and brilliant in colour. This method is more exact than an estimation as sulphide of copper.

Limits of Metals to be Allowed in Foods.—Neither lead, tin, nor zinc are *natural* constituents of any kinds of food, but copper has been shown to occur in varying quantities in many articles — *e.g.*, oysters, cocoa, peas, wheat, etc. No doubt the quantities of the metal will vary with the locality, and the possible variations are hardly yet definitely ascertained.

The quantity that has been found in oysters is con-

siderably greater than in any other articles of food, while the amount in wheat and cocoa is little more than traces.

The amount to be allowed in preserved peas and vegetables is a matter on which there are differences of opinion, but we should suggest that no action should be taken in cases where the quantity present, reckoned as metallic copper, does not exceed 1 grain per lb. of the mixed contents of the vessel containing the sample.

We are not prepared at the present moment to suggest what limit of zinc should be permitted in dried apples, but as regards cheese, the addition of zinc salts seems wholly indefensible.

With regard to tin in articles of food such as tinned fruits, it seems uncertain whether it occurs in such quantities or in such states of combination as to be seriously injurious to health, and there appears to be no definite information as to what amounts would prove injurious.

The case of lead in drinking water or in aerated waters is, however, different, as there is plenty of evidence to show that lead is a cumulative poison, and that even traces (such as $\frac{1}{50}$ of a grain per gallon) are objectionable.

COMMERCIAL DISINFECTANTS.

Assays as regards the value of commercial disinfectants can only be made by chemical means in so far as the determination of the percentage they contain of bodies on which their action depends, such as carbolic acid, metallic salts, etc.

Their actual efficiency—*i.e.*, power of killing disease-germs—can only be determined by bacteriological tests.

There are a great number of so-called disinfectants, concerning many of which very exaggerated claims are put forth ; some even contain amounts of caustic alkali, which would be harmful if applied to the skin according to the directions given.

It would be a great benefit to the general public if disinfectants were included under the Sale of Food and Drugs Acts, and such a course would prevent the sale of many useless and fraudulent preparations.

Disinfectants may be roughly divided into the following classes :

(*a*) Powders : carbolic and sulphite powders.

(*b*) Liquid disinfectants, depending on the action of carbolic or cresylic acids.

(*c*) Solutions of metallic salts.

Attention was drawn by Dr. Muter as far back as 1887 (*Analyst*, xii. 191), and again in 1890 (*Analyst*, xv. 63), to the fact that several of the so-called carbolic powders of commerce are practically devoid of carbolic acid, or contain it in a condition in which it cannot be reckoned as available.

Some powders contain sulphite of lime, as well as tar-products, and Dr. Muter considers that in estimating the amount 'reversion' should be allowed for.

The local authorities and their advisers are entirely to blame for the loss of life and waste of money occasioned by the supply of rubbish under the name of 'disinfectants,' and the condition of affairs can only be rectified by the adoption of a concise form of specification, and by the periodical sampling and analysis of the deliveries.

The following form of specification is a satisfactory one.

'*Tender for Supply of Carbolic Powder.*—The powder to contain not less than 15 per cent. of tar acids, *i.e.*, crude carbolic acid, of which 62½ per cent. crystallizes at 15° to 20° C., when examined by C. Lowe's test,' the base to contain no lime or chalk.

Assay of Carbolic Powders.—One or other of the two following methods may be adopted. The first is useful in showing how much of the carbolic acid present can be considered available, while the second provides for its complete extraction.

1. The powder is well mixed, and 50 grammes are extracted with spirit ; this extracts all the tar acids not in combination with lime. The extract is mixed with 50 cc. of 10 per cent. solution of caustic potash or soda, and the spirit distilled off and the liquid evaporated to about 30 cc. If any tar oils separate out, they are filtered off. The liquid is then run into a burette, and 50 per cent. sulphuric acid added a little at a time till the soda is completely neutralized. The tar acids are thus 'thrown up,' and will form a separate layer, the volume of which may be read off. On multiplying the number of cc. thus

obtained by 2, we shall get the percentage of phenols and cresols present.

2. This method is applicable to powders which give an alkaline reaction to litmus, showing that they contain free lime. The method of procedure is as follows :

50 grammes of the carefully-sampled powder is placed in a large mortar, and a cold mixture of equal parts—sulphuric acid and water—added drop by drop, the powder being stirred thoroughly meantime till all the lime has been converted into sulphate, as is indicated by a fragment of the powder no longer producing a blue spot with a drop of water on red litmus paper.

The operation should be extended over one hour, and the powder must not be allowed to grow hot, or there will be loss of carbolic acid through volatilization. If the operation has been well conducted, the powder will when neutralized be dry, and free from lumps. If it seems moist, anhydrous calcium sulphate should be added.

The powder is now extracted by four successive treatments with ether, as much ether as possible being poured off each time through a small filter into a flask containing 50 cc. of 10 per cent. sodium hydrate. The ethereal solution is then well agitated with the alkaline liquid, after which the flask is attached to a condenser, and the greater part of the ether distilled off.

The contents of the flask are now poured into a separator, and the flask washed out with small successive quantities of ether and water, which are, of course, poured into the separator. After well mixing by a rotary movement, the separator is set aside, and when the layers have separated well, the lower is run out into a basin, and the upper washed once with water in the separator, the washing being also run into the dish. The ether layer contains any neutral tar-oils, which may be estimated if desired by distilling off the ether in a weighed flask.

The phenate and cresylates of soda in the dish are evaporated to 30 cc., transferred to a burette, and treated as described above.

If it is desired to ascertain how much *crystallizable carbolic acid* is present, we must extract a large enough quantity to yield at least 50 cc. of tar acids, which are then treated as described below :

Assay of Crude Carbolic Acid.—The object of this

test is to ascertain the amount of crystallizable phenols contained in crude carbolic acid, and the temperature at which such crystallization occurs. The higher the temperature at which the selected portion of the distillate crystallizes, the better the sample.

Lowe's Test: 100 cc. of the sample are placed in a small retort, and heated by means of a small flame; no condenser is needed. The *rate* of distillation should be such that the drops follow each other rapidly, but do not fall in a continuous stream.

Two graduated cylinders are required, the first portion of the distillate—that is to say, all the water and 10 per cent. of the oils—being received into the first; this is then exchanged for the other, and 62·5 cc. collected. This is poured into a flask, provided with a cork through which a thermometer passes; and a fragment of phenol having been added, the liquid is gently stirred with the thermometer, and cooled. At the point of crystallization the thermometer rises slightly, and then remains constant. It is then read, and the temperature compared with those of mixtures of known composition.

Liquid Carbolic Preparations.—There are several liquid preparations, whose efficacy depends chiefly on the amount of carbolic and cresylic acid they contain. Some of these are of the same type as the preparations sold as sheep-dips, and form very perfect emulsions with water.

They are prepared by heating rosin with caustic soda, and then stirring in tar oils while the mixture is kept hot. They may be assayed by throwing up the tar acids with 50 per cent. sulphuric acid, and then distilling the tar acids as in the assay of crude carbolic acid.

Solutions of Metallic Salts.—Solutions of the following are used as 'disinfectants': *Zinc chloride,* sometimes known as *Burnett's fluid; permanganates of soda and potash,* 'ozonigen' and Condy's fluid; *iron salts,* especially the sulphate; *mercuric chloride and iodide.*

The Local Government Board, in one of their memoranda, advise the use of a solution prepared with—½ oz. mercuric chloride, 1 oz. hydrochloric acid, 3 gallons of water, 5 grains aniline blue. This gives a solution of 1 in 980.

Bleaching-powder, in solution of 6 oz. to 1 gallon, is a very powerful and economical disinfectant. Of the above-mentioned preparations, mercuric chloride, permanganates

of the alkali metals, and bleaching-powder, are chiefly to be commended as having a true germicidal action. Iron and zinc salts are properly antiseptics and deodorants only.

Bacteriological Testing of Disinfectants.—A bacteriological examination is the only means whereby a true estimate of the value of a disinfectant—*i.e.*, its power to kill micro-organisms—can be obtained. For the methods employed in the bacteriological examination of disinfectants, see the authors' work on 'Applied Bacteriology.'

THE EXAMINATION OF OILS AND FATS.

Animal and vegetable oils and fats are salts of glycerol with organic acids, or more correctly are 'mixtures of ethereal salts formed from glycerol and the fatty acids of the acetic and oleic series.'

The analysis of oils and fats requires great manipulative skill, and the interpretation of the results of analysis can only be successfully undertaken by those who have undergone a special training in this branch of work. There are great difficulties to contend with in the analysis of oils, many of which, of very different market values, are exceedingly similar in chemical constitution; many samples of the same oil may vary amongst themselves. The fatty acids on which the main differences depend cannot be readily separated or caused to enter into definite weighable compounds. Oils vary greatly in value, and hence there exists a constant inducement to adulterate the more valuable ones with those of an inferior kind. It must be borne in mind that, like other natural productions, oils vary very much according to the place of production, method of manufacture, purification, etc. It cannot be too strongly insisted upon that a given sample should not be pronounced as adulterated without comparing it with samples of known purity and origin.

In this small work we will only deal with the analytical constants of a few of the oils and fats in common use.

In the examination of an oil to ascertain its purity and

freedom from adulteration, it is necessary to examine it by the following tests :

The Specific Gravity is taken at 15·5° C. by the specific gravity bottle or the Westphal balance. In the case of oils or fats which are solid at the ordinary temperature, the specific gravity is taken at 99° C. by the Sprengel tube or Westphal balance. (See under 'Butter.')

The Saponification Equivalent (Koettstorfer's Method).—The saponification of oils by alkalies is a definite reaction which may be represented by the following general equation :

$$C_3H_5(O\bar{F})_3+3KHO=C_3H_5(OH)_3+3KO\bar{F}.$$

Therefore if we know exactly the amount of alkali necessary to saponify the oil under examination, we can to some extent determine the nature of the glycerides present. The saponification value varies with the composition of the fatty acids ; for instance, the lower the molecular weight of the fatty acids, the higher will be the amount of potash or soda necessary for the saponification. The saponification value of an oil may be stated in terms of alkali absorbed per cent., or the number of grammes of the oil which would be saponified by one litre of normal solution of alkali, which is usually known as the 'saponification equivalent.'

The following are the saponification values of some of the chief glycerides :

Glyceride.	Formula.	Molecular Weight.	Percentage of KHO required for Saponification.	Saponification Equivalent.
Butyrin	$C_3H_5(O{\cdot}C_4H_7O)_3$	302	55·7	=100·67
Laurin	$C_3H_5(O{\cdot}C_{12}H_{23}O)_3$	638	26·4	=212·67
Palmitin	$C_3H_5(O{\cdot}C_{16}H_{31}O)_3$	806	20·9	=268·67
Stearin	$C_3H_5(O{\cdot}C_{18}H_{35}O)_3$	890	18·9	=296·67
Olein	$C_3H_5(O{\cdot}C_{18}H_{33}O)_3$	884	19·0	=294·67

From the above table it is seen that the amount of alkali required for the saponification of the various glycerides differs largely. Since oils are mixtures of different glycerides, the ratio of which within certain limits is usually constant for the same class of oil, it will be seen that this process becomes very valuable in the examination of oils and fats. The process is carried out as follows : About 2 grammes of the sample is accurately

weighed out in a flask of about 200 cc. capacity. This is then treated with 25 cc. of alcoholic solution of caustic potash of approximately semi-normal strength.* A like amount of the alkali is run into an empty flask for a blank experiment. It is not necessary that exactly 25 cc. be taken, but precisely the same quantity must be taken in each case. A good plan is to let the pipette empty, and then allow three or four drops to fall; this will ensure the same amount of solution being taken in each case. The flasks are fitted with corks carrying vertical tubes about 4 feet long, to act as condensers. Both the flasks are then heated on a water-bath for not less than thirty minutes, with frequent agitation. One or two drops of phenolphthalein are added to the flasks, the contents of which are titrated with exactly $\frac{N}{2}$ solution of hydrochloric acid. Each cc. of $\frac{N}{2}$ HCl used = ·02805 of KHO, therefore the difference between the two titrations multiplied by this factor will give the amount of potash taken up by the oil, and from this is calculated the percentage of alkali required for saponification.

The saponification equivalent is found from the percentage of KHO absorbed by dividing into 5610. It can also be obtained by dividing the weight of oil taken in milligrammes by the number of cc. of normal alkali required for its saponification.

Example—

Amount of oil taken 2·001 grammes.

$\frac{N}{2}$ HCl for titrating back	12·2 cc.
$\frac{N}{2}$ HCl required for 'blank'	26·0 cc.
Difference =	13·8 cc.

$$13{\cdot}8 \times {\cdot}02805 = \frac{{\cdot}38709 \times 100}{2{\cdot}001} = 19{\cdot}3\ \%\ \text{KHO} = \frac{5610}{19{\cdot}3}$$

$$= 290{\cdot}7 \text{ saponification equivalent.}$$

Determination of the Unsaponifiable Matter.—5 grammes of the sample is saponified with 50 cc. of alcoholic potash of approximately semi-normal strength, by boiling under a reflux condenser for about thirty minutes, with frequent agitation. The solution is then evaporated

* This is best made by taking about 60 grammes of 50 per cent. solution of caustic potash and diluting to a litre with 'methylated spirit' which has been redistilled from ignited potassium carbonate.

down to dryness in a dish. The resulting soap is dissolved in about 200 cc. of hot water; when dissolved, the solution is transferred to a separator, which is immersed in cold water to allow the contents to cool. The aqueous solution of the soap is then treated with about half its volume of ether, the stopper inserted, and the whole thoroughly shaken and allowed to rest some time. It sometimes happens that the ether will not separate from the soap solution, a middle layer of gelatinous consistency being formed. In this case separation may be induced to take place by well cooling the separator under a stream of water, or, if this fails, by adding a few cc. of 10 per cent. potash solution, and a little more ether; separation is assisted by giving a rotary motion to the separator. The ethereal layer is separated, and the soap solution again extracted with ether, repeating the treatment again if necessary. The ethereal solutions are mixed, and well shaken up with water to wash out any soap which may have been taken up into the ether. The ether solution is separated, transferred to a flask, the ether distilled off, and the residue dried at 105° C. to constant weight, which multiplied by 20 will give the percentage of unsaponifiable matter present in the oil.

Nature of the Unsaponifiable Matter.—This may consist of cholesterol, phytosterol, mineral oil (hydro-carbons), rosin oil, etc. If the amount does not exceed about 1 per cent., it probably consists of cholesterol or phytosterol. These are higher alcohols of crystalline character and high melting-point which occur in small quantity in animal and vegetable oils respectively. Hydro-carbons, such as heavy mineral oil, may often be detected by the fluorescence which is imparted to the ethereal solution. Rosin oil, if present, may be detected by the bromide of tin test. (See under 'Colour Tests.')

Hubl's Iodine Absorption Equivalent.—This method, devised by Hubl, is based on the fact that all oils and fats are composed of the glycerine ethers of the members of two groups of fatty acids—the acetic and oleic series. The relative proportion of these acids in any variety of oil or fat is constant within certain limits, and differs only in different kinds of oil, but the members of the two groups of acids exhibit a very different behaviour towards chlorine, bromine, and iodine. While under ordinary circumstances the acids of the acetic series are indifferent, those of the

oleic series readily unite with definite quantities of the halogens. If, therefore, it is possible to make a fat or oil unite with a halogen, so that the amount of the latter which enters into the compound may be accurately determined, the number thus obtained would be a constant, and would be dependent upon the amount of unsaturated acids in the oil. Some chemists estimate the bromine absorption, but Hubl's iodine process is more convenient, and is the one we will describe here.

The Hubl solution is made as follows: 25 grammes of iodine is dissolved in 500 cc. of 95 per cent. alcohol, and 30 grammes of mercuric chloride in the same amount of alcohol. These two solutions are then mixed together, and allowed to stand at least twelve hours before using.

The determination is made as follows: About 0·2 to 0·35 gramme of the sample is accurately weighed by difference into a stoppered bottle of about 250 cc. capacity. The oil is then dissolved in 10 cc. of chloroform; when this has taken place, 25 cc. of the Hubl reagent is added from a pipette. A blank experiment is also started, using the same quantity of chloroform and iodine solution. The bottles are then allowed to remain in the dark for not less than four hours. To the contents of the two bottles is then added 20 cc. of a 10 per cent. solution of potassium iodide and about 150 cc. of water. The uncombined iodine is then titrated with $\frac{N}{10}$ sodium thiosulphate solution, the bottles being violently agitated during the titration until the free iodine has nearly disappeared, when a little fresh starch paste is added, and the thiosulphate added drop by drop, until the blue colour is just discharged. The number of cc. of thiosulphate used is deducted from the amount required for the blank experiment; the difference is multiplied by ·0127 (if the thiosulphate is exactly decinormal). This gives the amount of iodine taken up by the oil, and from this is calculated the percentage of iodine absorbed.

Example—

Oil taken 0·205.

Blank experiment required 30·0 cc. $\frac{N}{10}$ $Na_2S_2O_3$.

Sample required ... ... 18·0 cc. ,,

Difference = 12·0 cc. × ·0127 =

$\frac{\cdot 1524 \times 100}{\cdot 205} = 74 \cdot 34$ per cent. *iodine absorbed.*

The thiosulphate solution must always be standardized before using against pure iodine to ascertain its exact strength.

Free Fatty Acid.—Animal oils, when first prepared, and the first runnings in the case of oils of vegetable origin, contain only traces of free fatty acids. On exposure to air, however, the free fatty acid increases rapidly, the result being that the oil becomes rancid. The lower grades of vegetable oils generally contain a large percentage of free fatty acid, often amounting to 30 or more per cent. The determination is made as follows: 5 or 10 grammes of the oil is well shaken with about 100 cc. of boiling neutral alcohol. A drop or two of phenol-phthalein solution is added, and decinormal solution of NaHO run in with constant agitation, until a permanent pink colour is obtained. Each cc. of $\frac{N}{10}$ NaHO = ·0282 oleic acid. Oils intended for dietetic purposes should not have a greater rancidity than 4 or 5 per cent. of free fatty acid calculated as oleic.

Maumené's Test.—This test depends on the rise of temperature which takes place on mixing oils with concentrated sulphuric acid. Much work has been done on this process by various chemists, who have arrived at very opposite opinions regarding its value as a quantitative test. Many of the discrepancies which are found between the results of many of these observers are in great measure due to the different methods of working, and also to the variation in strength of the sulphuric acid employed. Allen and Archbutt, who have carefully investigated this test, find that if the strength of the acid is much below 97 per cent. the temperature attained is not only less, but the reaction is much slower. Other workers have advised the use of 93 and 95 per cent. acid respectively, but these strengths are not to be recommended. The acid which gives the best results is 97 per cent. The strength of the acid must be determined by careful titration, as the specific gravity is quite useless to determine the strength of sulphuric acid, as Lunge has shown that acid of 95 to 100 per cent. may have almost exactly the same specific gravity. The following is the best procedure for the carrying out of this test:

50 grammes of the sample is weighed out into a tall beaker of about 250 cc. capacity; this is wrapped in cotton-wool, which is encased in a suitable box or larger beaker.

The temperature of the oil, which should be at about 18° to 20° C. is carefully noted; 10 cc. of the 97 per cent. H_2SO_4 is then run from a pipette into the beaker containing the oil, the mixture being constantly stirred with the thermometer; the rate of flow of the acid should be so arranged that the delivery of the 10 cc. takes about sixty seconds. The stirring is continued until the temperature ceases to rise; this point is readily observed, as the temperature generally remains constant for some short time before falling. The temperature of the acid should be about the same as the oil, but this is not of very great importance, since E. J. Bevan has found that 5° difference of temperature between that of the oil and acid does not influence the result.

The initial temperature of the oil is deducted from the highest reached after the addition of the acid, and the difference noted as the 'temperature reaction' of the oil.

Messrs. Ballantyne and Thomson have suggested a modified form of the Maumené test, wherein the temperature is compared with that of water under the same conditions. The rise of temperature observed with the oil is divided by that given by water; the figure so obtained he calls the 'specific heating power' of the oil.

The Maumené test should be done in a draught cupboard or in the open air, as in some cases a large volume of sulphur dioxide is given off.

Valenta Acetic Acid Test.—This test has given in our hands such satisfactory results in the examination of oils that we are surprised that it has not held a more important position than it appears to have done. The test, as we have before mentioned, depends on the intermiscibility of the glycerides with acetic acid at various temperatures. (For method of performing test see under 'Butter.')

A somewhat improved method of working this test is, instead of using an ordinary test-tube, to use a short and somewhat thick tube into which a well-fitting stopper has been ground. Into this tube is weighed 2·75 grammes of the fat; 3 cc. of the acetic acid is then run in from a burette or other suitable arrangement. The tube is then stoppered and placed in a beaker of warm water, increasing the heat until, after well shaking the tube, the contents become quite clear. The source of heat is then removed, and the test-tube so placed that it is in the centre of the beaker of heated water, and by means of a thermometer

attached to the tube by a rubber band the whole is allowed to rest until the change from brilliancy to turbidity. The change is very definite, and can be repeated over and over again with a maximum error of about 0·25° C.

The Oleorefractometer.—This instrument, devised by Messrs. Jean and Amagat, has recently come into use for testing oils and fats. The instrument is so arranged that a ray of light from a lamp is passed through a chamber containing lard oil, which is the 'type' or standard oil. In the centre of this chamber is placed a hollow prism, which is filled with the oil or fat under examination. The light, in passing through the prism, is more or less deflected, casting a sharp shadow on a scale which is placed in the focus of the eye-piece of a telescope attached to the body of the oil-chamber. A collimator is placed in front of the telescope, and the oil-chamber is surrounded by a case to contain warm water, so that observations may be made at any required temperature. The scale is divided into divisions, both right and left of the zero mark. Vegetable oils deflect light to the right and animal oils to the left of the zero. These deflections are noted as + or − ; that is, right or left of the zero mark, as the case may be. The liquid oils are examined at 22° C. Butter, margarine, lard, etc., that are not liquefied at this temperature are examined at 45° C.

It should not be overlooked that the figures given by the oleorefractometer are not absolute ones, the instruments frequently differing among themselves ; therefore each instrument should be set against samples of known purity.

Colour Tests.—Many colour tests have been suggested from time to time, dependent on the fact that many oils yield colour reactions when treated with various reagents, such as sulphuric acid, nitric acid, mercurous nitrate, stannic chloride, etc. Many of these tests are not dependent on the oils themselves, but on impurities contained therein, such as albuminous and resinous substances. As these matters are now largely removed by the improved processes of purification and refining used, it follows that very considerable variation must be observed in the behaviour of different samples of the same oil to these reagents. These tests are therefore of little value, so no description of them will be given here.

Analytical Constants of Oils and Fats.

	Oil or Fat.	Sp. Gr. at 15·5° C.	Percentage KHO for Saponification	Iodine Absorption.	Valenta Test.	Oleorefractometer Figure.	Maumené Test.
	VEGETABLE OILS.						
Non-drying Oils.	Olive	·914 to ·917	19·0 to 19·6	77·0 to 86·0	83° to 91°	+ 3·5 to 1·0	40° to 45°
	Almond	·916 to ·920	19·5 to 19·7	94·0 to 99·0	72° to 87°	+10·5 to 8·0	51° to 54°
	Peach-kernel ...	·918 to ·921	18·9 to 19·2	95·0 to 99·5	80° to 83°	+11·5 to 7·5	40° to 46°
	Arachis	·917 to ·921	19·0 to 19·6	95·0 to 102·0	72° to 74°	+ 7·0 to 5·0	45° to 60°
Semi-drying Oils.	Cottonseed ...	·922 to ·930	19·1 to 19·7	101·0 to 116·0	71° to 89°	+23·0 to 16·0	74° to 80°
	Sesame	·923 to .924	18·8 to 19·1	106·0 to 108·0	90° to 97°	+17·0 to 13·0	64° to 68°
	Rape	·914 to ·917	17·5 to 17·8	99·5 to 105·5	77° to 83°	+20·0 to 16·0	52° to 59°
	Castor	·960 to ·966	17·8 to 18·0	83·6 to 83·9	Miscible.	+42·0 to 39·0	45° to 47°
	Mustard ...	·916 to ·920	17·2 to 17·5	92·0 to 100·0	—	—	44° to 49°
Drying Oils.	Linseed	·932 to ·937	18·7 to 19·5	173·5 to 187·7	46° to 48°	+54·0 to 48·0	104° to 120°
	Hempseed ...	·925 to ·931	19·0 to 19·3	144·0 to 150·0	—	+37·4 to 34·0	95° to 98°
	Nigerseed ...	·927 to ·930	18·9 to 19·1	132·0 to 135·0	50° to 53°	+30·0 to 26·0	81° to 83°
	Sunflower ...	·924 to ·926	19·3 to 19·4	122·0 to 133·0	59° to 63°	+35	67° to 75°

Analytical Constants of Oils and Fats.—*Continued.*

Oil or Fat.	Sp. Gr. at 15·5° C.	Percentage KHO for Saponification	Iodine Absorption.	Valenta Test.	Oleorefractometer Figure.	Maumené Test.
ANIMAL OILS.						
Neat's foot ...	·914 to ·916	19·4 to 19·6	69·3 to 70·4	72° to 75°	− 3·0 to 1·0	47° to 49°
Lard oil ...	·914 to ·916	19·1 to 19·6	77·0 to 80·0	74° to 76°	Zero.	65° to 67°
FISH OILS.						
Cod-liver ...	·923 to ·930	18·2 to 18·7	141·0 to 144·0	70° to 76°	+46·0 to 40·0	113° to 116°
Whale	·919 to ·931	18·8 to 22·4	81·0 to 110·0	68° to 71°	+48·0 to 42·0	86° to 90°
Seal	·924 to ·929	18·9 to 19·3	142·0 to 153·5	70° to 75°	+36·0 to 30·0	90° to 95°
VEGETABLE FATS.	Sp. Gr. at 99°C. Water=1.					
Palm oil ...	858·5	19·6 to 20·2	51·0 to 52·5	—	—	—
Cotton stearin ...	864·8	19·5 to 19·6	89·0 to 92·0	—	—	—
Cacao butter ...	858·0	19·2 to 20·1	34·0 to 37·0	—	—	—
ANIMAL FATS.						
Butter fat ...	·865 to ·867	22·1 to 23·2	23·0 to 38·0	29° to 39°	−34·0 to 25·0	—
Margarine ...	·856 to ·860	19·3 to 19·7	50·0 to 57·0	94° to 97°	−18·0 to 13·0	—
Lard	·860 to ·862	19·5 to 19·7	50·0 to 63·0	97° to 99°	−14·0 to 8·0	—
Tallow	860·8	19·3 to 19·8	35·0 to 42·0	96° to 99°	−18·0 to 15·0	—

A colour reaction, to be of value, must depend for its reaction on the oil itself, or on a substance contained in and natural to the oil. To this class belong the Baudouin and Tocher tests for sesame-oil, the silver nitrate test for cotton-seed-oil, the bromide of tin test for rosin oil, and the Hager test for cholesterol.

Hager's Test for Cholesterol.—A small fragment of cholesterol is dissolved in about 2 cc. of chloroform, and an equal volume of strong sulphuric acid added. The chloroformic solution immediately becomes blood-red, afterwards purple, which remains permanent for some days, while the acid layer shows a strong green fluorescence. Phytosterol gives the above reaction, but the chloroformic solution becomes bluish-red after a day or two.

The Bromide of Tin Test for Rosin Oil.—Stannic bromide, containing a little free bromine, is dissolved in carbon disulphide. When a drop or two of this reagent is added to rosin oil, a beautiful violet colour appears.

The stannic bromide is made by allowing dry bromine to drop on granulated tin contained in a flask until a red coloration of the product indicates that bromine is in excess.

This test may be applied to the unsaponifiable matter to determine the presence or absence of rosin oil.

Olive Oil.

Olive oil is obtained by expression or extraction from the fruit of the olive tree (*Oleo Europæa sativa*). The colour of olive oil varies from almost colourless to deep yellow ; some varieties are green, due to dissolved chlorophyl.

The free fatty acid varies very much in different samples. In 'salad' oil it should not exceed 4 or 5 per cent., but in commercial samples it may be as high as 25 per cent. Olive oil gives the lowest mean rise of temperature in the Maumené test—40° to 45°. The unsaponifiable matter in olive oil averages about 1·0 per cent., and consists of cholesterol, whereas in other vegetable oils the unsaponifiable matter consists of phytosterol. Olive oil, on account of its high value, is adulterated to a very large extent. Cottonseed, arachis, poppy, rape, and sesame oils are among those used for its adulteration.

Many of the bottled oils on the market sold as 'Finest Lucca Olive Oil,' and under other fancy names, consist entirely of cottonseed or arachis, or mixtures of these with a little olive oil.

The oleorefractometer is very useful in the examination of olive oil ; 120 samples examined gave the following results : Lowest deviation, +1·0 ; highest deviation, +3·5 ; average, +2·0.

The various oils used to adulterate olive oil would be detected as follows : Cottonseed oil by the high figures obtained with the iodine absorption, oleorefractometer, Maumené test and specific gravity, also by the silver test (see under 'Lard') ; arachis oil by the isolation of arachidic acid (see under 'Arachis' oil) ; sesame oil by the high iodine absorption, specific gravity, and oleorefractometer figures ; rape oil by the oleorefractometer, iodine absorption, Maumené and saponification tests.

Almond Oil.

Almond oil is expressed from the seed of the sweet and bitter almond-tree (*Prunus amydalus dulcis* and *Prunus amydalus amara*). Almond oil has a very pale yellow colour and bland taste. Almond oil is adulterated with peach-kernel, olive, sesame, cottonseed, poppy, and arachis oils. Nearly the whole of the so-called 'foreign' almond oil consists of peach-kernel oil, the characters of which are, unfortunately, so similar to those of genuine almond oil that it is quite impossible to differentiate them by the quantitative tests. They can, however, be distinguished by their behaviour to nitric acid of specific gravity 1·40. Three parts of the oil is shaken with one part of the acid, when—Almond oil gives a light yellow colour, turning brown. Peach-kernel oil gives a bright red colour.

Cottonseed oil would be detected by the silver test, sesame oil by the Baudouin and Tocher tests.

Arachis Oil.

Arachis oil (pea-nut, earth-nut oil) is produced from the seeds of the leguminous plant *Arachis hypogœa*. The

higher qualities, which are 'cold drawn,' have a pleasant taste, recalling kidney beans. Arachis oil has very similar analytical constants to olive oil, from which it cannot be detected except by the isolation of arachidic acid (see below). In chemical composition arachis oil is peculiar, as the glycerides olein and palmitin are replaced by the glyceride of arachidic acid. The isolation of this acid furnishes a very valuable method for determining the presence of arachis oil when used as an adulterant of other oils.

Determination of Arachidic Acid.—This is best done by the original method of Renard, which, though tedious, is very reliable. The process is carried out as follows: Saponify 10 grammes of the oil, separate the fatty acids from the soap solution by hydrochloric acid, dissolve these in 90 per cent. alcohol, and add a solution of lead acetate.* Filter off the precipitated lead salts, extract them with ether, thus separating the lead salts of the unsaturated acids from the lead palmitate and arachidate. Treat these latter salts with hydrochloric acid, separate the fatty acids when solidified, after cooling, from the lead chloride, and dissolve them in 50 cc. of hot 90 per cent. alcohol. If arachis oil is present in the sample, a crop of crystals consisting of arachidic acid will be obtained on cooling the alcoholic solution. Filter the crystals off, and wash them on the filter, first with a measured quantity of 90 per cent. alcohol, then with 70 per cent. alcohol, which dissolves but small quantities thereof, and finally dissolve them by pouring boiling absolute alcohol on the filter, receiving the filtrate in a tared dish or flask. Evaporate to dryness, and weigh the residue, consisting of crude arachidic acid. Add to the weight thus obtained the quantity dissolved by the 90 per cent. used for the washing, 100 cc. of which dissolve 0·022 gramme at 15·5°C., or 0·045 gramme at 20° C. Finally, determine the melting-point of the crude acid, which should be from 71° to 72° C. Renard has isolated from 4·5 to 5·0 per cent., A. H. Allen 5·5 per cent. of arachidic acid from oil of arachis. Hence the amount of acid found will represent approximately $\frac{1}{20}$ of the arachis oil present, and the latter may

* Lewkowitsch shortens the process by neutralizing the excess of alkali with acetic acid, and precipitating with a lead salt without isolating the fatty acid.

therefore be found by multiplying the weight of the acid by 20.

Sesame Oil.

Sesame oil (*Gingili oil, Teel oil*) is obtained from the seeds of the *Sesamum orientale* and *indicum*. The oil has a very pleasant taste, but little odour, and is generally of a light yellow colour. It belongs to the class of semi-drying oils. Sesame oil contains a very small quantity of a body upon the presence of which depend the two following characteristic colour reactions, known as the Baudouin and Tocher tests :

Baudouin's Test.—Dissolve 0·1 gramme of cane-sugar in 10 cc. of hydrochloric acid of specific gravity 1·2. To this is added 20 cc. of the oil to be tested ; shake thoroughly, and allow to stand. In the presence of even 2 per cent. of sesame oil the aqueous liquid will become of a crimson colour.

Tocher's Test.—1 gramme of pyrogallol is dissolved in 15 cc. of concentrated hydrochloric acid. This solution is well shaken in a separating funnel with 15 cc. of the oil, and allowed to stand. After separation has taken place, the aqueous liquid is drawn off and boiled for a few minutes. If sesame oil is present, the solution becomes coloured, appearing red by transmitted, and blue by reflected, light.

Rape Oil.

Rape or colza oil is obtained by expression from the seeds of the *Brassica campestris*, and several allied plants belonging to the natural order Cruciferæ. In this country these oils are known indiscriminately as rape or colza oil. On the Continent, however, a distinction is drawn : the oil from the seeds of the *B. campestris* is known as colza ; the oil from the *B. campestris* var. *napus* is known as rape. Rape oil has a somewhat yellow colour and harsh, unpleasant taste. The oil belongs to the semi-drying class. The oil is extensively adulterated with linseed, hemp, cottonseed, fish, and mineral oils.

Castor Oil.

Castor oil is obtained from the seeds of the *Ricinus communis*. It is a colourless or greenish-tinged oil. It is very viscous, and thickens on exposure to air. It is entirely soluble in 1 volume of absolute alcohol, and in 4 volumes of rectified spirit. It is also miscible with glacial acetic acid in all proportions. Castor oil has the highest specific gravity of any natural fatty oil.

Linseed Oil.

Linseed oil is obtained by expression and extraction from the flax plant (*Linum usitatissimum*). The taste and odour are characteristic ; the oil obtained by hot pressure is sometimes very acrid and nauseous. Linseed is principally imported from the Baltic and Black Sea coast, also India. The Baltic seed yields the best oil. The colour of the cold-expressed oil is a bright golden-brown ; the hot expressed oil is very dark brown. On exposure to air linseed dries hard, absorbing oxygen, and forming a body insoluble in ether, known as linoxyn. Linseed has the highest iodine absorption and Maumené figure of all the fatty oils. On account of its cheapness, linseed is but seldom adulterated with other seed-oils, but rosin, mineral and fish oils are not unfrequently used as adulterants.

In the absence of any appreciable amount of unsaponifiable matter, any lowering of the iodine or Maumené figures would point to adulteration with fish-oils, as would also a low specific gravity.

Lard.

Lard is the internal fat of the abdomen of the pig. There are enormous quantities of lard rendered from the fat of the whole animal. According to Wiley, lards may be divided into the following classes :

(*a*) *Neutral Lard.*—This consists of the fat from the 'leaf' of the animal, rendered quite fresh at a temperature of from 40° to 45°. This lard is used in the manufacture of margarine.

(*b*) *Leaf or Bladder Lard.*—The fat of the 'leaf' rendered by steam heat under pressure.

(*c*) *Choice Lard, Kettle Lard, etc.*—This lard is generally rendered from the fat of the whole animal. According to the Chicago Board of Trade, choice lard is defined as lard made from the 'leaf' and trimmings, rendered by steam heat.

Freshly-rendered lard is quite free from free fatty acid. Lard possesses a pure white colour, a granular texture, and agreeable taste. Lard is extensively adulterated with cottonseed oil, cottonseed and beef stearin. Many of the so-called 'compound' lards do not contain any lard at all, being mixtures of cottonseed oil and beef stearin. Arachis and sesame oils have been stated to have been used to adulterate lard.

Analytical Constants of Lard.

Melting-point ... ... (B.P., 1898 = 37·8° C.)	36° to 45° C.	
Iodine absorption ...	50 to 65	per cent.
Oleorefractometer ...	−14·0 „	− 8·0
Saponification value ...	19·5 „	19·7
Specific gravity at 15·5° C.	=931 „	932
„ „ 99° C.	=860 „	862

The iodine absorption of pure lard will not be above 65 or below 50 per cent. If the iodine absorption falls out of this range, the sample must be considered as adulterated ; but the fact of a normal iodine figure being obtained does not prove the sample to be genuine, as a judicious mixture of cottonseed or arachis oil with cottonseed or beef stearin would give apparently normal figures when examined by the test.

The Silver Test for Cottonseed Oil.—This valuable test has received a great deal of attention at the hands of various chemists. There are many modifications of the test in use. The following method will, in careful hands, detect as little as 1 per cent. of cottonseed oil. The reagent is made as follows : 1 gramme of finely-powdered

nitrate of silver is dissolved in 100 cc. of 95 per cent. alcohol ; when dissolved, 20 cc. of ether and 1 drop of nitric acid are added ; 2 cc. of this reagent is well shaken with 10 cc. of the oil to be examined, and placed in boiling water for ten minutes. Any blackening due to reduced silver proves the presence of cottonseed oil.

Beef Stearin.—W. F. K. Stock has devised a method to determine the amount of beef stearin in lard. He compares the crystals obtained from an ethereal solution with those from two standard sets of mixtures, the first consisting of pure lard melting at 34° to 35° with 5, 10, 15, and 20 per cent. of beef stearin melting at 56° C. ; the second of pure lard, of melting-point 39° to 40°, with 5, 10, 15, and 20 per cent. of beef stearin melting at 50° C. The process is as follows : The melting-point of the sample is determined by the capillary-tube method. Suppose the melting-point be found at 34° C., 3 cc. of the melted fat are run into a graduated cylinder of about 25 cc. capacity ; 21 cc. of ether are added, and the fat dissolved at 20° to 25° C. ; 3 cc. of each of the first set of mixtures are treated in exactly the same way. The five cylinders are cooled down to 13° C., and allowed to remain at that temperature for twenty-four hours. An approximate estimate as to the amount of the adulterant is arrived at by reading off the apparent volume of the deposited crystals. The ether is then poured off as far as possible, and 10 cc. of fresh ether at 13° C. is added in each case. The cylinders are again shaken, cooled as before, and the proportion of crystals read off as before. Finally, the contents of the cylinders are emptied into weighed shallow beakers, the ether drained off carefully, the mass allowed to dry for fifteen minutes at 10° C., and weighed. The weight obtained for the sample under examination is compared with the weight of the crystals obtained from whichever of the standards comes nearest to it. The second set of mixtures is used for samples of higher melting-point. The actual presence of beef fat must be proved by microscopical examination, when the characteristic tufts are seen if beef fat is present. No sample of pure lard melting below 39° C. yielded more than 0·011 gramme of crystals under the above conditions. A sample of the melting-point 45·8° C. gave, however, 0·146 gramme of crystals.

The following are some iodine absorption figures given

by W. H. Wiley, United States Department of Agriculture (Bull. 13, p. 4):

Pure leaf lard (Squire and Co.)	...	56·9 per cent.
Pig's feet lard (Wesson and Co.)	...	77·3 „
Bladder lard (mean of 20 samples)	...	61·2 „
Steam lard (mean of 12 samples)	...	62·0 „
'Compound' lard (mean of 13 samples, Armour and Co.)		64·6 „
'Compound' lard (mean of 17 samples, Fairbank and Co.)		85·3 „
Cottonseed oil (mean of 17 samples)	...	109·0 „
'Oleo' stearin (mean)		17·4 „
Lard stearin (mean)		44·2 „

Water.—Lard used to be very frequently adulterated by the introduction of water, but this sophistication is not now very frequent. Lard must be absolutely free from water and ash. The fat on melting should be clear, and free from suspended matter, such as particles of membrane, etc.

THE EXAMINATION OF SOAP.

The various kinds of soap in every-day use, for household or manufacturing purposes, are not as yet substances which it is the duty of the public analyst to examine in his official capacity, not being included under any of the designations food, drink, or drugs. It is our opinion that considerable advantage to the public at large would result were the scope of the Food and Drugs Acts considerably augmented, so that the public analyst should officially examine many substances in every-day use other than merely edible or potable matters.

Soaps generally may be classified into four groups, viz.:

(1) Scouring soaps and analogous substances used by manufacturers.

(2) Household or laundry soaps.

(3) Medicated soaps.

(4) 'Fancy' soaps.

Of the first group, nothing need be said in reference to the necessity of analysis; those consumers who require such quantities of materials as to make their orders of a wholesale nature have it well within their power to pro-

tect themselves against possible fraud by simply contracting for goods of specified quality.

Soaps of the second class exhibit great divergencies between the intrinsic qualities of soaps sold for household purposes, and these differences are by no means always taken into account in the price. Of course, it cannot be expected that a soap sold at 3d. per lb. should be identical in quality with one sold at 4d. per lb.; but it is evident that the cheaper article ought to contain at any rate three-quarters of the amount of actual soap. It would seem that no injury would result to the trade if certain standards of quality as regards percentage of actual soap were adopted, so that any article sold as soap of such and such a kind should of necessity contain not less than such and such a percentage of actual soap—*e.g.*, a curd soap of first quality might be required to contain not less than 70 per cent. of actual soap, and so on for other qualities. In the same way, soaps treated with silicate of soda for the purpose of increasing detergence and diminishing cost of production, through incorporation of large amounts of water, should be sold as 'silicated soaps,' and not under names calculated to give the impression that they are true soaps devoid of admixture.

As regards medicated soaps, it is an open question as to whether such articles should or should not be regarded as patent medicines subject to stamp duty; but it is obvious that if an article is sold on the understanding that it contains a certain medicinal agent incorporated therewith, and such agent is not there, or is contained to a smaller extent than is represented, the purchaser is prejudiced.

A standard might conveniently be fixed for carbolic acid soaps by insisting on a given minimum of carbolic acid being present in an article sold under such a name. It is by no means infrequent to find soaps sold as containing sulphur, glycerine, honey, milk, cream, etc., and represented as being for that reason highly beneficial, when in point of fact no trace of any such constituent is actually present.

The fourth class of soap above referred to is almost invariably sold in tablet form for convenience of use. Although the title 'toilet soap' or 'fancy soap' does not

actually imply that the products, or the materials from which they are made, have been specially refined and purified so as to render the soap innocuous to tender and sensitive skins, yet there is a general impression that such is the case. The actual state of the case, however, is very far from being in accordance with this fact.

The soaps of commerce may be divided broadly into 'hard' and 'soft' soaps. The hard soaps are made of the various animal and vegetable oils and fats saponified with caustic soda; the soft soaps are manufactured from the fish and other low-grade oils, potash being used in the saponification.

100 parts of neutral glyceride produce about 150 parts of finished soda soap.

Resin is a legitimate substitute for fatty matter in the common soap, as used for household and manufacturing purposes, as the resinates possess powerful detergent properties.

Soap-powders, washing-powders, dry soaps, etc., are generally mixtures of carbonate of soda with dried and powdered soap, sometimes with the addition of soda sulphate and other inert materials.

The Analysis of Soap.

Water.—10 grammes of a representative sample is weighed out after being reduced to the state of thin slices or shavings. It is dried at 50° C., which is afterwards raised to 105° C., and continued at this temperature till no further loss of weight is noted. The object of drying at the lower temperature is to prevent the soap melting, and thus making it very difficult to dry to constant weight.

In the best curd soaps the water varies from 12 to 20 per cent., whereas in some common soaps, such as those made from palm oil, the water may reach 75 per cent.

Uncombined Fat.—The residue, after drying, is exhausted with petroleum ether. This will dissolve out any unsaponified fatty matter—hydro-carbon oils (unsaponifiable matter), phenols, cresols, etc., that may be present in the soap. The petroleum ether is separated, distilled off, and the residue weighed.

Total Alkali. — The residue after treatment with petroleum ether constitutes the soap proper, and any mineral additions that may be present. This is treated with about 200 cc. of hot alcohol until all that will is dissolved. The alcoholic solution is then filtered, and the filter washed with alcohol. The filtrate is then made up to a definite volume, and divided into two parts—*a* and *b*.

(*a*) To this solution (= 5 grammes of the original sample) are added one or two drops of solution of phenol-phthalein, and the liquid titrated with $\frac{N}{10}$ HCl until the pink colour is just discharged. The alkalinity found is calculated to NaHO as *free alkali.*

(*b*) The second portion of alcoholic solution (= 5 grammes original soap) is diluted with water, two or three drops of solution of methyl-orange added, and the solution titrated with $\frac{N}{2}$ HCl. This will equal *total alkali*, which is calculated to K_2O or Na_2O as the case may be.

The residue, if any, left on the filter after treatment with alcohol may consist of carbonate, silicate, or borate of the alkalies ; other substances, such as starch, sand, clay, etc., added as 'fillers ;' pigments, as ultramarine, umber, ochre.

The residue is treated with water, and filtered. Starch, clay, sand, etc., will remain undissolved. This residue is further examined if necessary. This solution, after making up to a definite volume, is tested for carbonates, borates, and silicates. Half the solution should be evaporated in a platinum dish with hydrochloric acid twice to complete dryness, taken up in water, and the residual silica filtered off, washed, and weighed. The silica so found is calculated to silicate of sodium.

Carbonates or borates, if present, may be titrated with standard acid.

Fatty and Resin Acids.—5 grammes of the soap is dissolved in hot water (cooled and exhausted with ether if free glycerides or unsaponifiable matter is present), and then decomposed with a slight excess of dilute sulphuric acid. The precipitated acids are then taken up in ether, the ethereal solution washed free from acid with water ; the ether distilled off, and the residue dried to constant weight.

The residue will be fatty acids, and, if present, resin acids.

Resin.—The resin is estimated in the fatty acids by the method devised by Gladding. About 0·5 gramme of the mixture of the fatty acids and resin is dissolved in 20 cc. of strong alcohol, and with phenol-phthalein as indicator $\frac{N}{10}$ NaHO is run in until there is a slight excess. The alcoholic solution, after boiling for ten minutes to ensure complete saponification, is mixed with ether in a graduated cylinder till the volume is 100 cc. To the alcoholic and ethereal solution 1 gramme of very finely-powdered silver nitrate is added, and the contents of the cylinder are shaken thoroughly for ten or fifteen minutes. After the precipitate has settled, 50 cc. are measured off, and, if necessary, filtered into a second graduated cylinder. A little $AgNO_3$ is added to see if the precipitation is complete, and then 20 cc. of dilute hydrochloric acid (1 to 2) is added to decompose the silver resinate. An aliquot part of the ethereal solution in the cylinder is evaporated in a tared dish and weighed as resin, deducting a small correction for oleic acid (for 10 cc. deduct 0·00235 gramme). The amount of resin so found, subtracted from the combined fatty acids and resin as found before, gives the amount of fatty acids.

Estimation of Carbolic Acid.—J. Lewkowitsch has found the following method sufficiently accurate for all practical purposes : 50 grammes of the sample is dissolved in water, and about 20 cc. of 10 per cent. potash added to combine with the free phenols and cresols present. The solution is then treated with a large excess of strong brine. This will precipitate the soap as a granular mass. The supernatant liquid is then separated, and the soap again washed with a further quantity of brine, which is again repeated if necessary. The solution of the phenates and cresolates is evaporated to small bulk, and then introduced into a graduated tube (or, better, a Muter's carbolimeter). Add more salt if necessary, then acidify with hydrochloric acid ; the volume of the separated phenols and cresols is read off and taken as so many grammes.

Analyses of some Soaps of Commerce.

	Curd.	Curd.	Castile.	Castile (Mottled).	Tallow.	Tallow.	Soft.
Water	28·1	27·0	14·0	22·2	20·9	35·0	38·4
Fatty acids ...	67·4	68·0	77·0	67·7	71·0	45·0	48·4
Combined alkali	7·5	7·7	8·7	9·0	8·9	6·0	12·0
Free alkali ...	·1	·2	·3	·4	·3	2·1	3·2
Silica	·3	·8	none	·2	none	7·0	·2
Insoluble in alcohol			1·1	2·1	1·6	7·7	1·1
Total	103·4	103·7	101·1	101·6	102·7	102·8	103·3

URINE.

For the purposes of clinical diagnosis, it is absolutely essential to ascertain the total amount of urine voided during a period of twenty-four hours, especially in the case of morbid conditions, such as the presence of sugar, etc. The analysis of a urine is of very little use to the medical man unless it represents the composition of the urine passed during this period. A healthy human being of about 140 lb. weight excretes in twenty-four hours about 50 oz. of urine. This will have a specific gravity of about 1·020, and will contain about 4 per cent. of total solids, or nearly 20 grains to the ounce.

The data usually required are the specific gravity, reaction to litmus, colour, the amount of urea, the presence or absence of albumen and glucose, and if present, the amount. The characters of the deposit, if any, should be very carefully noted, both chemically and microscopically. Uric acid and bile acids are also tested for, and in some cases it is desirable to know the amount of total solids, chlorides, sulphates, phosphates, etc.

Urine is of a pale-yellow or reddish-yellow colour if normal. When blood is present, the urine has a brownish-red colour, and is often greenish or greenish-brown in tinge if bile acids are present.

Normal urine may be turbid, owing to the presence of urates, phosphates, or mucus. Urates will dissolve on warming with potash and phosphates on acidifying with acetic acid.

For general purposes, it is best to state the results of analysis in percentages, or better in grammes per 100 cc. in preference to grains per ounce or pint.

The **Specific Gravity** is taken at 15·5° by the bottle or Westphal balance. This ranges in healthy urine from 1·014 to 1·030. In diabetes the specific gravity occasionally reaches 1·062, whereas in albuminuria and diabetes insipidus it may fall as low as 1·005.

The **Reaction** to litmus is carefully ascertained by adding a few drops of litmus to a portion of the sample. Normal urine should be faintly acid. Healthy urine may, however, especially in warm weather, undergo an alkaline fermentation, caused by the micrococcus ureæ, which converts the urea present into ammonium carbonate.

Albumen may be tested for by the following tests :

Boiling Test.—The urine is rendered very faintly acid with acetic acid, and raised to the boiling point. Albumen, if present, precipitates in the form of a cloud or dense coagulum, dependent on the amount present.

Bodeker's Test.—To about 10 cc. of the urine faintly acidulated with acetic acid, a solution of ferro-cyanide of potassium is added drop by drop. If a precipitate forms, albumen is present.

Picric Acid Test.—An equal volume of clear saturated solution of picric acid is added to the urine. If a turbidity or precipitate forms, the tube is heated to boiling. If the precipitate is due to peptones or alkaloidal bodies, it will dissolve, while if the precipitate remains permanent, albumen is present.

Estimation of the Albumen.—An approximate estimation may be made by testing in an albuminometer. In this instrument the albumen is precipitated by various reagents, and the volume of the precipitate measured. There are many forms of this instrument—Esbach's is the best—but they are all very inaccurate. The albumen is

best estimated gravimetrically by boiling a measured volume of the faintly acidified urine until all the albumen is coagulated. The flocks of albumen are then filtered off, well washed with water, and dried at 100° C.

A quick, easy, and fairly accurate means of determining the coagulable proteids of urine is given by A. H. Allen: The specific gravity of the urine is carefully determined before and after coagulating the albumen by boiling. A quantity of the urine is filtered and faintly acidulated with acetic acid. The specific gravity is carefully noted, and the temperature of the liquid also noted. The liquid is next boiled, filtered through a dry filter, and the specific gravity again taken after cooling to the original temperature. The diminution in the specific gravity by boiling (water = 1000) multiplied by 0·4 gives the number of grammes albumen per 100 cc. of urine. Thus, if the original urine had a specific gravity of 1026, which was reduced to 1020 by boiling, the difference, multiplied by 0·4, equals 2·4 grammes of albumen per 100 cc. of sample.

Glucose may be tested for by the following tests:

Fehling's Test.—The urine is made alkaline with caustic potash; any phosphates which may be deposited are filtered off. Fehling's copper solution* is then added to the filtrate, which is then carefully raised to boiling. If cuprous oxide is precipitated, glucose is present.

If any albumen is present, this must be removed by boiling before applying the test. Fehling's copper

* Fehling's solution is best made as follows: 34·64 grammes of pure crystallised sulphate of copper is dissolved in distilled water and the solution diluted to 500 cc. 70 grammes of caustic soda and 180 grammes of recrystallised potassium sodium tartarate (Rochelle salt) are dissolved in about 400 cc. of water and the solution diluted to 500 cc. These two solutions are mixed in equal proportion just before use.

The following are the weights of the different sugars that will completely reduce 10 cc. of this solution:

10 cc.	Fehling's solution	=	·0500	grammes	dextrine, lævulose or invert sugar.
10 cc.	,,	,,	= ·0475	,,	cane sugar (after inversion).
10 cc.	,,	,,	= ·0678	,,	lactose (milk sugar).
10 cc.	,,	,,	= ·0807	,,	maltose (malt sugar).

solution, when properly applied, is the simplest and best means of detecting glucose in urine. It is absolutely necessary that the copper solution should be fresh and in good condition. It should remain perfectly clear when diluted with distilled water and boiled for a few minutes.

In some cases where only traces of glucose are present, particularly in abnormal urines containing notable quantities of creatinine, xanthin, uric acid, glycuronic acid, and other bodies reducing Fehling's solution, the indications given by this test are somewhat uncertain. Allen has devised a very ingenious modification of the Fehling test whereby these interfering bodies are removed by a preliminary treatment with copper sulphate. The testing is carried out as follows : From 7 to 8 cc. of the sample of urine to be tested is heated to boiling in a test tube, and without separating any precipitate of albumen which may be produced, 5 cc. of the solution of cupric sulphate used for preparing Fehling's test is added. This produces a precipitate containing uric acid, xanthine, hypoxanthine, phosphates, etc. To render the precipitation more complete, however, it is desirable to add to the liquid, when partially cooled, from 1 to 2 cc. of a saturated solution of sodium acetate, having a feebly acid reaction to litmus. The liquid is next filtered. To the filtrate, which will have a bluish-green colour, 5 cc. of the alkaline tartrate solution used for preparing Fehling's solution is next added, and the liquid boiled for from fifteen to twenty seconds. In the presence of more than 0·25 per cent. of sugar separation of cuprous oxide occurs before the boiling-point is reached ; but with smaller quantities precipitation takes place during the cooling of the solution, which becomes greenish, opaque, and suddenly deposits cuprous oxide as a fine orange-yellow precipitate.

Moore's Test.—The urine is slightly acidified with acetic acid, and boiled ; albumen, if present, is removed by filtration. An excess of 10 per cent. caustic potash solution is then added, and the liquid boiled. Normal urine will yield a reddish-brown liquid ; but if sugar is present, the liquid will become deep brown or black.

Nylander's Test.—1 cc. of the urine is added to 10 cc. of Nylander's reagent, and gently boiled ; if even traces of sugar are present, the solution will become black. The

reagent is made as follows : 2·5 grammes of pure bismuth oxynitrate (free from silver) and 4 grammes of Rochelle salt are dissolved in 100 cc. 8 per cent. solution of sodium hydrate.

Dr. Johnson's Test.—4 cc. of the urine is mixed with an equal volume of a saturated solution of picric acid in a test-tube ; to this mixture is added 2 cc. of a 6 per cent. solution of caustic potash. An orange-red colour instantly appears as a result of the incipient reducing action of the kreatinine upon picric acid at ordinary temperatures. The colour is deepened by boiling, and if after the liquid has been kept at the boiling point for about a minute a bright red colour appears through the test-tube when held up to the light, the urine for clinical purposes may be confidently pronounced free from sugar. If an aqueous solution of glucose in the proportion of not more than 2 grains to the ounce be tested in the manner described, the liquid will be rendered so dark that no light is visible through the full diameter of the tube.

The Estimation of Sugar in Urine.—Sugar in urine is best determined by Dr. Pavy's modification of Fehling's process.

The process is applied to the estimation of glucose in urine as follows : 10 grammes of the urine is diluted to 100 cc. with 10 per cent. ammonia solution. 20 cc. of the Pavy solution (=·010 grammes glucose) is diluted with 20 cc. of water, and placed in a flask of about 250 cc. capacity, which is fitted with an india-rubber stopper pierced with two holes. The flask is then fixed to the nozzle of a tapped burette, which is filled with the diluted ammoniacal urine. Through the second hole in the stopper is passed a short tube with a right angle bend, to which is attached a length of rubber tubing, which is used to convey the ammoniacal vapours given off during the titration out of a window.

The diluted Pavy solution in the flask is then gently boiled, and kept boiling during the titration with a low gas flame or spirit lamp. The saccharine liquid is allowed to run cautiously a little at a time into the flask until the blue colour is just discharged. Three titrations are made, and the mean result taken, when $\frac{100 \times \cdot 010}{\text{cc.'s used}} =$ per cent. of glucose in sample. Kreatinine, creatine, and traces of other substances normally present in urine, exercise a slight re-

ducing action on Pavy's solution, though they do not interfere to so great an extent as they do when Fehling's method is employed. As their removal is attended with great difficulty, and they are usually present in small but constant amounts, we prefer not to undertake the purification of the urine, but to make an allowance equal to 0·2 per cent., equal to ·75 grain to the ounce of glucose, which must be subtracted from the total glucose found. That this correction is sufficiently exact for all ordinary purposes we have proved by the comparative results yielded by a considerable number of diabetic and normal urines.

The Estimation of Urea.—The urea is estimated by the hypobromite of sodium method, which depends on the fact that when urea is treated with a solution of hypobromite of sodium the urea is decomposed with the liberation of nitrogen, as shown by the following equation :

$$CH_4N_2O+3NaBrO=3NaBr+CO_2+2H_2O+N_2.$$

The whole of the nitrogen of the urea is not always evolved, while some of the nitrogen may be yielded by the other nitrogenous constituents of the urine—for instance, uric acid, kreatinine, etc. This is of but little consequence, as a given specimen always yields the same quantity of gas.

The process is carried out as follows : 25 cc. of the sodium hypobromite solution (made by adding 2·5 cc. of bromine to 25 cc. of 40 per cent. soda hydrate and cooling) is placed in a 4-oz bottle fitted with a rubber stopper, through which passes a glass tube, to which is attached an indiarubber tube, the other end of which is connected with a nitrometer. Into the bottle containing the hypobromite solution is lowered a short test-tube containing 5 cc. of the urine, taking great care that none is spilt. The bottle is then carefully corked and connected with the nitrometer, which is best filled with water. The water-level is then set, and the generating bottle tilted, when, the urine mixing with the hypobromite solution, nitrogen and carbon dioxide will be evolved according to the equation already given, the carbon dioxide being absorbed by the excess of caustic soda present. After a few minutes the water-levels in the two tubes of the nitrometer are adjusted, and the increased volume of gas due to nitrogen noted. Each cc. of nitrogen at the normal temperature and pressure equals ·0029 grammes of urea in the 5 cc. of urine tested.

Normal urine contains from 2 to 3 per cent. urea.

The Determination of Uric Acid. — Uric acid is detected in urine by the addition of one-fifth of its volume of concentrated hydrochloric acid. The uric acid will deposit after standing some hours in the form of reddish-brown crystals. Uric acid can be estimated by Haycraft's method, which is based on the fact that uric acid combines with silver as silver urate, which is practically insoluble in water, ammonia, or acetic acid, but perfectly soluble in nitric acid. The necessary solutions are:

(*a*) A centinormal solution of ammonium thiocyanate, standardized by a silver solution of known strength. 1 cc. of this = 0·00168 gramme uric acid.

(*b*) An ammoniacal silver solution. 5 grammes silver nitrate in about 100 cc. of water, precipitated and re-dissolved in ammonia to a clear solution.

(*c*) Ferric indicator. A saturated solution of iron alum.

(*d*) Pure nitric acid. 25 cc. of the urine is placed in a small beaker, together with about 1 gramme of sodium bicarbonate, and two or three drops of strong ammonia. This precipitates ammonio-magnesic phosphate, and prevents reduction of silver; 1 to 2 cc. of ammoniacal silver solution is then added, which at once precipitates the silver urate. The mixture is now placed on a filter, and washed until the washings show no trace of silver on testing with salt solution. The precipitate is then dissolved in a few cc.'s of nitric acid, washed into a flask, a few drops of the ferric indicator added, and then titrated with the ammonium thiocyanate solution until a permanent red colour just appears. The number of cc.'s used, multiplied by 0 00168 × 4, gives the percentage of uric acid in the sample. (Sutton, 'Vol. Analysis.')

Normal urine contains from 0·3 to 0·7 per cent. uric acid.

The Detection of Bile.—*Pettenkofer's Test.*—Equal parts of the urine and sulphuric acid are mixed; after cooling the tube in cold water, a little powdered white sugar is then added; after well mixing, the tube is slightly warmed. If a reddish or violet coloration due to the liberation of cholalic acid is seen, bile is undoubtedly present.

Gmelin's Test.—When urine containing bile is cautiously mixed with an equal volume of nitric acid, a play of colours is seen, varying from green or blue to violet and red.

Oliver's Test.—1 cc. of the clear urine (which is filtered if necessary) is mixed with 3 cc. of Oliver's reagent. An opalescence appears if bile acids are present.

Oliver's reagent is made as follows: 2 grammes of peptone, 0·25 gramme salicylic acid, are dissolved in water, to which 2 cc. of 33 per cent. acetic acid had been added. The solution is diluted to 200 cc. The reagent must be rendered perfectly bright by filtration before using.

The Detection of Blood. — Urine containing blood generally has a characteristic smoky appearance. The urine may be examined by Dr. Day's test as follows: 2 or 3 cc. of tincture of guaiacum (which must be freshly made from the unoxidized resin), and a like amount of an ethereal solution of peroxide of hydrogen (ozonic ether), are added to the urine or the deposit. In the presence of blood, a beautiful sapphire blue colour will develop.

Note.—This test must be regarded with caution, as many other organic bodies yield a similar reaction.

Estimation of Phosphoric Acid.—After removal of albumen, if present, by boiling, 100 cc. of the urine is boiled with an excess of nitric acid; ammonium molybdate and ammonium nitrate, dissolved in nitric acid, is then added, the liquid well boiled, and allowed to stand some hours. The precipitate is filtered off, and washed with dilute alcohol until free from acidity. The precipitate is then redissolved in ammonia and a solution of ammonio-sulphate of magnesium (magnesia mixture), and allowed to stand over-night. The precipitate is collected on a filter, and washed with a mixture of one volume of ammonia and three of water. The precipitate is now ignited, allowed to cool, moistened with nitric acid, dried, and again ignited before the blowpipe. The magnesium pyrophosphate ($Mg_2P_2O_7$) is then weighed, the weight multiplied by the factor ·63964 to obtain the amount of P_2O_5.

Normal urine contains from 0·2 to 0·3 per cent. of P_2O_5.

Estimation of Sulphates.—100 cc. of the urine, free from albumen, is acidulated with hydrochloric acid, boiled, and barium chloride added; the liquid again well boiled, and the precipitated barium sulphate filtered off, the filter

well washed, dried, ignited, and weighed in the usual way. The weight of barium sulphate obtained is multiplied by the factor ·34335 to obtain the amount of SO_3 present.

Normal urine contains from 0·15 to 0·3 per cent. SO_3.

Estimation of Chlorides.—10 cc. of the urine is very carefully neutralized with sodium carbonate, and then diluted to 100 cc. 10 cc. are then taken and diluted to about 100 cc. in a white dish, one or two drops of pure neutral chromate of potassium solution added, and the solution titrated with decinormal silver nitrate, when ·00585 × cc. used × 100 = per cent. NaCl present.

Normal urine contains from 0·5 to 1·0 per cent. of sodium chloride.

Chemical Examination of the Sediment. — The urine is allowed to deposit in a conical test-glass, and the deposit examined both chemically and microscopically. Urinary deposits are generally simple in character, and their composition can usually be determined by the following tests:

1. Warm the urine containing the sediment, when if the latter should dissolve, it consists entirely of urates. In this case, let it once more crystallize out, and examine it by the ordinary course for Ca, Na, and NH_4 to ascertain the bases.

2. If the deposit be not dissolved by heating, let it settle, wash once by decantation with cold water, and warm with acetic acid. Phosphates will dissolve, and may be reprecipitated from the solution by excess of ammonia filtered out, well washed with boiling water, dissolved in acetic acid, and examined for Ca or Mg by the usual course for these metals in presence of phosphoric acid.

3. If the deposit is insoluble in acetic acid, warm it with hydrochloric acid. Any soluble portion is calcium oxalate, which may be precipitated by ammonia.

4. If the deposit is insoluble in hydrochloric acid, it is in all probability uric acid. In this case apply the 'Murexid' test as follows: Place it in a small white dish, remove moisture by means of filter paper, add a drop or two of strong nitric acid, and evaporate to dryness at a gentle heat. When cold, add a drop of ammonia, which will produce a purple colour, deepened to violet by a drop of caustic potash solution.

The urates are often of a pinkish colour, owing to the presence of the red pigment 'purpurin,' whence the name 'red gravel.' Phosphate of calcium and the ammonio-magnesium phosphate are generally both present in deposits, the magnesium salt usually forming the larger proportion. Free uric acid is nearly always crystalline, and yellow, red, or brown in colour.

Microscopical Examination of the Sediment.—The microscopical examination of urinary deposits requires much experience to be of value. In case of doubt, reference should always be made to typical slides. The best magnification to use is about sixty diameters. The following are the general characters of the most frequent forms :

Ammonio-Magnesium Phosphate (*Triple Phosphate*).—This is deposited as soon as the urine becomes alkaline, the ammonia being derived from the decomposition of the urea. The crystals are seen as regular and ragged stellate plates. Arborescent forms are also sometimes seen.

Uric acid occurs in flat plates, quadratic prisms, needles, rosettes ; spindle and star-like crystals are also of frequent occurrence. On testing with a drop of potash inserted under the cover-glass, uric acid will dissolve.

Amorphous deposits may be either phosphate of magnesium or calcium, or the urates of calcium, magnesium, ammonium, potassium, or sodium, chiefly the last. The urates may be detected by treatment on the slide with acetic acid, when in the case of the urates the characteristic forms of uric acid will develop.

The urates of magnesium and sodium sometimes occur crystalline in the form of small tufts or bundles of needles.

Oxalate of calcium occurs in very small octahedra. Oxalate of calcium is insoluble in acetic acid, but soluble in hydrochloric acid.

Cystin is somewhat rare as a urinary deposit ; it is seen in the form of flat hexagonal lamellæ superposed. Cystin is soluble in ammonia.

Hippuric acid occurs in characteristic pointed rhombic prisms and acicular crystals. The larger crystals may be mistaken for triple phosphate, and the smaller for uric acid ; but insolubility in acetic acid distinguishes them from the former, and solubility in alcohol from the latter.

Blood is easily recognised by the reddish circular

corpuscles ; the discs are seen, both singly and in strings, not unlike piles of coins. In stale urine, the blood corpuscles lose their round shape, and become angular.

Pus deposits on standing as a light-coloured layer, which easily diffuses through the liquid on shaking. Pus corpuscles are larger than those of blood, and are light coloured.

Mucus deposits not unlike pus, but has no definite structure ; it generally occurs with pus epithelium, etc. Mucus imparts a 'ropy' appearance to urine.

Epithelial debris are frequently present in urine in the form of nucleated cells, regular and oval when full, but angular and unsymmetrical when partially emptied of their contents.

Casts of uriniferous tubuli are fibrinous masses of various forms, and often of considerable length ; sometimes delicate and transparent, occasionally granular, and often containing fat globules.

Spermatozoa are liable to escape notice, on account of their extremely small size and transparency. Their tadpole-like appearance is characteristic.

Various micro-organisms are frequently met with in urine. *Sarcina*, *staphylococcus pyogenes aureus*, various *streptococci*, the *gonococcus*, etc., are those of frequent occurrence. Staining with aniline-gentian-violet will render their recognition more easy.

Foreign bodies, such as hair, fibres of wool and cotton, fragments of feathers, are amongst those most frequently seen, but are not easily mistaken for any substances that can actually occur in urine.

NOTES ON THE BRITISH PHARMACOPŒIA.

The 1898 edition of the British Pharmacopœia is a great improvement upon that of 1885 from the analytical point of view, owing to the fact that a much larger number of the preparations have now to conform to definite tests and standards. Many new official tests and processes of standardization have been added, and others improved, although some of them are defective in character. Since the British Pharmacopœia should be regarded as the standard authority for the definition of the characters, tests, strengths, etc., of all preparations intended for medicinal use, it is very desirable that the official tests and processes should be thoroughly mastered by all interested in the examination of drugs and pharmaceutical preparations, and for this purpose direct reference should be made to the Pharmacopœia.

Owing to lack of space, we are unable to reproduce or criticise the official processes here, but must confine ourselves to the following notes upon the tinctures, extracts, and alcoholic strengths of the Pharmacopœia.

The Tinctures of the British Pharmacopœia.

The composition of the tinctures of the British Pharmacopœia of 1898 has been entirely reconstituted, particularly with reference to the alcoholic menstrua employed in their preparation. The tinctures may be divided into five classes, according to their method of preparation, as follows :

1. *Prepared by Maceration.*—The powdered drug is treated with alcohol of specified strength in a closed vessel for a definite period with frequent agitation. When the exhaustion is complete the liquid is strained off, and the marc submitted to pressure ; the liquids are then mixed and filtered.

2. *Prepared by Maceration*, with the difference that the final product is made up to a definite bulk.

3. *Prepared by Percolation.*—The powdered drug is

moistened with alcohol, then exhausted in a percolator, with the rest of the spirit and the marc pressed, the expressed liquid mixed with the bulk, the resulting liquid being made up to definite volume with alcohol of the specified strength.

4. *Officially Standardized Tinctures.*—The following tinctures are required to show on assay the following percentages of active constituent:

Tinct. belladonnæ	... 0·05 gram. alkaloid in 100 cc.
Tinct. cinchonæ	... 1·00 gram. of total alkaloid in 100 cc.
Tinct. cinchonæ co.	... 0·50 gram. of total alkaloid in 100 cc.
Tinct. jalapæ	... 1·50 gram. of jalap resin in 100 cc.
Tinct. nucis vomicæ	... 0·25 gram. of strychnine in 100 cc.
Tinct. opii ...	... 0·75 gram. of morphine in 100 cc.

5. *Prepared by Admixture.*—The tinctures of this class are prepared by adding the required weights of ingredients to a given quantity of alcohol of specified strength.

The following table shows some results obtained on the more important tinctures prepared strictly according to the British Pharmacopœia of 1898. The first and second column give the specific gravity at 15·5° C., as given by Lucas ('Practical Pharmacy,' 1898) and Barclay (*Pharm. Journ.*, Dec. 17, 1898); the third and fourth columns give the specific gravity and alcoholic strengths on a series of tinctures, obtained by ourselves; the fifth column gives the method of preparation of the tincture according to the above classification which is partly founded on that suggested by Lucas.

Tincture.	Specific Gravity (Lucas).	Specific Gravity (Barclay).	Specific Gravity (P. & M.).	Absolute Alcohol by volume (P. & M.).	Mode of Preparation. Class.
Aconiti	·893	·890	·896	64·3	3
Aloes	·975	·970	·980	39·5	2
Arnicæ	·974	·894	·890	65·0	3
Asafetidæ	·915	·914	·912	63·7	2
Aurantii	·876	·885	·883	75·5	1
Belladonnæ	·917	·914	·919	58·8	4
Benzoini Co. ...	—	·900	·890	75·5	2
Buchu	·928	·934	·930	57·0	3
Calumbæ	·918	920	·917	56·0	1
Camphoræ Co. ...	·915	920	·917	57·0	5
Cannab. Ind. ...	·846	·846	·846	86·0	5
Cantharidis	·834	·838	·835	88·5	1
Capsici	·894	·896	·892	67·0	1
Cardamomi Co. ...	·945	·948	·946	57·0	1
Cascarillæ	·898	·900	·901	67·5	3
Chiratæ	·922	·920	·925	56·1	3
Cimicifugæ	·928	·924	·925	57·3	3
Cinchona	·916	·918	·918	64·6	4
Cinchona Co. ...	·913	·918	·918	65·0	4
Cinnamomi	·900	·904	·903	66·3	3
Colchici Sem. ...	·950	·953	·956	43·0	3
Conii...	·896	·896	·899	65·0	3
Cubebæ	·845	·840	·847	87·3	3
Digitalis	·928	·932	·936	57·0	3
Ergotæ Ammon. ...	·937	·934	·938	47·0	3
Ferri Perchlor. ...	1·086	1·086	1·086	22·3	5
Gelsemii	·925	·925	·922	58·0	3
Gentianæ Co. ...	·965	·966	·970	41·9	1
Guaiaci Ammon. ...	·899	·900	·887	79·8	2
Hamamelidis ...	·948	·952	·957	40·2	3
Hyoscyami	·950	·953	·955	42·5	3
Iodi	·878	·878	·879	87·3	5
Jaborandi	·950	·953	·956	42·2	3
Jalapæ	·910	·906	·906	67·1	4

Tincture.	Specific Gravity (Lucas).	Specific Gravity (Barclay).	Specific Gravity (P. & M.).	Absolute Alcohol by volume (P. & M.).	Mode of Preparation. Class.
Kino	·998	·995	·999	49·8	2
Krameriæ	·930	·938	·939	56·6	3
Lavandulæ Co. ...	·837	·836	·838	89·7	1
Limonis	·876	·888	·880	77·0	1
Lupuli	·931	·938	·934	56·0	1
Myrrhæ	·850	·854	·853	85·3	2
Nucis Vomic. ...	·912	·912	·914	63·7	4
Opii	·953	·958	·953	45·5	4
Opii Ammon. ...	·900	·895	·898	66·3	2
Podophylli	·844	·850	·846	87·5	2
Quassiæ	·941	·946	·947	44·5	1
Quininæ Ammon. ...	·928	·925	·926	54·2	5
Rhei Co.	·971	·970	·972	51·9	3
Scillæ	·972	·960	·973	53·8	1
Senegæ	·935	·938	·941	57·0	3
Sennæ Co.	·991	·988	·995	38·8	1
Serpentariæ	·895	·896	·900	68·3	3
Stramonii	·952	·962	·963	43·6	3
Strophanthi	·890	·892	·892	67·5	3
Tolutanæ	·867	·860	·867	81·0	5
Valerianæ Ammon.	·936	·942	·936	54·2	1
Zingiberis	·840	·835	·839	88·1	3

Specific Gravity of Tinctures.—This is obtained by use of the bottle or Westphal balance, the temperature of the samples being brought to 15·5° C.

Estimation of Alcohol in Tinctures. — The exact estimation of alcohol in tinctures is by no means easy. Any volatile oil or other body present comes over in the distillate and vitiates the result to this extent. This source of error is not readily overcome. Again, the resinous and other extractive matter retains the last traces of alcohol with great tenacity. The following

procedure will, however, be found to give very fair results in the case of most of the tinctures : 25 cc. of the sample carefully measured in an exact pipette at 15·5° is diluted with 80 cc. of water, and placed in a distillation flask attached to an efficient condenser. A spiral of platinum wire and a few fragments of pumice-stone are added to the flask to prevent bumping. Some of the tinctures give rise to excessive frothing on distillation. This is best overcome by suspending a small pipe-cleaning or bottle-brush in the neck of the distillation flask.

About 70 cc. of distillate is then collected, when a further quantity of 25 to 30 cc. of water is added to the distillation flask, and the distillation allowed to proceed until the distillate measures exactly 100 cc. at 15·5° C. The distillation should be allowed to proceed as slowly as possible. In the case of the ammoniacal tinctures of ergot, guaiaci, opii, quinine, and valerian, the ammonia should be neutralized with dilute sulphuric acid. In the case of tincture of iodine, neutralization of the iodine by means of alkali is of course inadmissible owing to the production of iodoform. The iodine is best fixed by means of a slight excess of sodium thiosulphate.

The compound tincture of camphor has to be specially treated as detailed below owing to the presence of camphor and the large quantity of essential oil.

50 cc. of the sample is taken, and made up to 350 cc. by the addition of water. This causes a precipitation of the oil and resinous matter. The liquid is then clarified by adding a few drops of a strong solution of calcium chloride, followed by some sodium phosphate. The resultant precipitate of calcium phosphate entangles the oily and resinous matter. The liquid is now made up to 400 cc. with water, filtered through a dry filter, and 250 cc. of the filtrate distilled until about 200 cc. have passed over. The distillate is then made up to 250 cc., and the specific gravity taken at 15·5° C. If the foregoing instructions be adhered to, the percentage of alcohol corresponding to the specific gravity of the distillate, multiplied by eight, will be the percentage by volume contained in the tincture.

There is a natural loss of spirit which is unavoidable in the manufacture of tinctures, and the moisture natural to drugs may, in some tinctures, also tend to lower the

percentage of alcohol in the finished product. It is therefore hardly possible to lay down a hard and fast rule as to the deficiency of alcohol that may reasonably be passed, but we think that in no tincture ought this loss to exceed 3 per cent. of absolute alcohol by weight.

Methylic Alcohol.—Methylated spirit is sometimes used partly or wholly in the preparation of tinctures, etc. This can be tested for by means of the Riche and Bardy test as given under 'Spirits.'

Opium.—The proportion of opium present in the compound tincture of camphor can be approximately judged by the depth of the red colour produced, when the sample previously diluted with water or proof spirit is treated with a solution of ferric chloride. By comparing the tint obtained with that given by a sample of known quality, a fair criterion of the proportion of opium may be obtained.

Benzoic Acid.—The benzoic acid in compound tincture of camphor can be estimated as follows: 25 cc. of the tincture is rendered alkaline with sodium hydrate, and evaporated to 10 cc.; a portion of the camphor and oil of anise will be volatilized. The liquid is then diluted slightly, and extracted with ether. This will remove the camphor and oil remaining from the liquid. The ether is separated, and the aqueous liquid rendered acid with hydrochloric acid; the free benzoic acid is then removed by agitation with ether. On allowing the separated ethereal solution to evaporate spontaneously in a small beaker, the benzoic acid is obtained in a fit condition for weighing.

An approximate determination of the benzoic acid may be made by determining the acidity of the tincture by titration with $\frac{N}{20}$NaHO, using phenol-phthalein as indicator. 10 cc. of the tincture is a convenient quantity to titrate. Each cc. of the soda required = 0·0061 benzoic acid.

Total Solids.—On account of the natural variation of drugs it is impossible to expect that the total solids obtained by evaporating the official tinctures to constant weight should absolutely accord, but we think it will be possible in the near future to lay down limits within which all genuine samples should fall.

The Pharmacopœial Extracts.

The extracts of the British Pharmacopœia may be divided into five classes, according to their method of preparation, as follows :

(1) Evaporated infusions ; (2) evaporated decoctions ; (3) evaporated aqueous extracts obtained by infusion, followed by percolation ; (4) evaporated plant juices ; (5) evaporated strong tinctures. The following extracts are officially standardized :

Alcoholic extract of belladonna	to contain 1·00 per cent. of alkaloid.
Liquid extract of belladonna ...	to contain 0·75 gram. of alkaloid per 100 cc.
Liquid extract of cinchona ...	to contain 5·00 gram. of alkaloid per 100 cc.
Liquid extract of ipecacuanha ...	to contain 2 to 2·25 gram. of total alkaloid per 100 cc.
Extract of nux vomica	to contain 5·00 per cent. of strychnine.
Extract of opium	to contain 20·00 per cent. of morphine.
Liquid extract of opium ...	to contain 0·75 gram. of morphine per 100 cc.

The official processes given in the Pharmacopœia for the assay of the alkaloidal extracts and tinctures are on the whole fairly satisfactory, with the exception of that given for the liquid extract of ipecacuanha, which certainly gives results below the truth, owing to the bulky lead precipitate obtained in the second stage of the process taking down with it a notable proportion of alkaloid which is thus lost.

This defect of the official process has been pointed out by H. Wilson (*Pharm. Journ.*, July 2, 1898), who has suggested the following process, which is speedy, simple, and accurate :

'Take 20 cc. of the strong liquid extract, dilute with 20 cc. water, place in a porcelain dish, and dissipate the alcohol by evaporating the mixture to rather less than half its bulk ; allow to cool. Now add 1 cc. dilute sul-

phuric acid, and transfer to a separator, washing the dish with 20 cc. water, and adding these washings to the liquid in the separator. Add 10 cc. ether-chloroform (ether and chloroform equal volumes), agitate, warm to promote separation ; run off and reject the ether-chloroform layer, and twice repeat the treatment with the same quantity of ether-chloroform. Add now 10 cc. ether-chloroform and excess of solution of ammonia, agitate, warm, and run off the separated ether-chloroform layer into a tared dish ; agitate with two more similar quantities of ether-chloroform, separate as before, adding these solutions to that in the tared dish. Evaporate the mixed solutions and dry the residue below 80° C. until of constant weight. This weight, less that of the dish, will give the weight of total alkaloids present in the quantity of liquid extract operated on.'

The drying of the alkaloidal residue till of constant weight is tedious, but no means can at present be devised for shortening this operation, as cephaëline has been shown by Paul and Cownley to lose weight at 100° C., and hence to guard against this loss the residue must be dried below 80° C.

The Alcoholic Strengths of the Pharmacopœia.

The alcoholic strengths of the new British Pharmacopœia are entirely different to those of the old edition. Absolute alcohol is the name given to a mixture of ethyl-hydroxide, containing not more than 1 per cent. of water. Alcohol (90 per cent.) is the name given to a mixture containing in 100 volumes 90 volumes of ethyl-hydroxide. This is 6 per cent. stronger than the rectified spirit of the edition of 1885, and the name *spiritus rectificatus* is retained for it alone. Four other strengths of alcohol are also employed, namely : alcohol of 70 per cent., 60 per cent., 45 per cent. and 20 per cent. These dilutions are the outcome of the inquiry as to the best menstrua for the extraction of the crude drugs in the preparation of tinctures. Proof spirit is now omitted. Spirituous solutions of essential oils are now termed spirits. They are nearly in every case made of the

strength of 1 volume of the oil to 9 volumes of the alcohol (90 per cent.).

Fifty-eight over-proof (more exactly 57·7 to 57·8 over-proof) will probably become the recognised proof strength for 90 per cent. alcohol, just as 56 over-proof was the common expression for the 55·4 over-proof rectified spirit of the 1885 Pharmacopœia. It is a matter for congratulation that 'proof-spirit' as a standard of alcoholic strength, with its complications of 'over' and 'under' proof, is now likely to become quite obsolete pharmaceutically. However convenient the proof system may be for the purposes of the Excise, the simpler system of volume percentage adopted in the Pharmacopœia must commend itself as being better adapted to the requirements of pharmacy.

So much depends upon accuracy in the prescribed strength of the alcoholic menstrua employed for certain pharmaceutical preparations, that in order to guard against error (especially when the method of mixture by volume has been adopted) it is often advisable to check the specific gravity of a diluted alcohol before use. But before a true reading can be taken, the liquid must either be brought to the standard temperature of 15·5°, or a correction made for temperature by observing the temperature and apparent specific gravity at the time of the experiment, and ascertaining the real specific gravity at 15·5° C. by reference to the following useful table, compiled by F. C. J. Bird, which embraces the extremes of temperature likely to be met with in this climate :

Apparent Specific Gravities of the Official Diluted Alcohols at Temperatures from 2° to 25° C. (35° to 77° F.)

Degrees C.	90 per cent.	70 per cent.	60 per cent.	45 per cent.	20 per cent.	Degrees F.
2	·8454	·9010	·9242	·9529	·9795	35·6
3	·8446	·9002	·9234	·9522	·9793	37·4
4	·8438	·8994	·9226	·9515	·9790	39·2
5	·8429	·8986	·9218	·9508	·9787	41·0
6	·8421	·8977	·9210	·9502	·9785	42·8
7	·8412	·8969	·9203	·9495	·9782	44·6
8	·8404	·8961	·9195	·9488	·9780	46·4
9	·8395	·8953	·9187	·9481	·9777	48·2
10	·8387	·8946	·9179	·9474	·9774	50·0
11	·8378	·8936	·9171	·9468	·9772	51·8
12	·8370	·8928	·9164	·9461	·9769	53·6
13	·8361	·8920	·9156	·9454	·9767	55·4
14	·8353	·8912	·9148	·9447	·9764	57·2
15	·8344	·8904	·9140	·9440	·9761	59·0
15·5	**·8340**	**·8900**	**·9135**	**·9436**	**·9760**	**60·0**
16	·8335	·8895	·9132	·9433	·9759	60·8
17	·8327	·8887	·9124	·9426	·9756	62·6
18	·8318	·8879	·9116	·9419	·9754	64·4
19	·8310	·8871	·9108	·9412	·9751	66·2
20	·8301	·8863	·9100	·9405	·9748	68·0
21	·8293	·8854	·9093	·9399	·9746	69·8
22	·8284	·8846	·9086	·9392	·9743	71·6
23	·8276	·8838	·9078	·9385	·9741	73·4
24	·8268	·8830	·9070	·9378	·9738	75·2
25	·8259	·8822	·9062	·9371	·9735	77·0

The figure in the fourth decimal place is not intended to be scientifically accurate, but merely serves to qualify the one preceding it.

ALCOHOL TABLES.

Specific gravity, 15·5°.	Absolute Alcohol by weight. Per cent.	Absolute Alcohol by volume. Per cent.	Proof Spirit. Per cent.	Specific gravity, 15·5°.	Absolute Alcohol by weight. Per cent.	Absolute Alcohol by volume. Per cent.	Proof Spirit. Per cent.
1·0000	0·00	0·00	0·00	·9689	22·15	27·04	47·39
·9999	0·05	0·07	0·12	·9679	22·92	27·95	48·98
·9989	0·58	0·73	1·28	·9669	23·69	28·86	50·57
·9979	1·12	1·42	2·48	·9659	24·46	29·76	52·16
·9969	1·75	2·20	3·85	·9649	25·21	30·65	53·71
·9959	2·33	2·93	5·13	·9639	25·93	31·48	55·18
·9949	2·89	3·62	6·34	·9629	26·60	32·27	56·55
·9939	3·47	4·34	7·61	·9619	27·29	33·06	57·94
·9929	4·06	5·08	8·90	·9609	28·00	33·89	59·40
·9919	4·69	5·86	10·26	·9599	28·62	34·61	60·66
·9909	5·31	6·63	11·62	·9589	29·27	35·35	61·95
·9899	5·94	7·40	12·97	·9579	29·93	36·12	63·30
·9889	6·64	8·27	14·50	·9569	30·50	36·76	64·43
·9879	7·33	9·13	15·99	·9559	31·06	37·41	65·55
·9869	8·00	9·95	17·43	·9549	31·69	38·11	66·80
·9859	8·71	10·82	18·96	·9539	32·31	38·82	68·04
·9849	9·43	11·70	20·50	·9529	32·94	39·54	69·29
·9839	10·15	12·58	22·06	·9519	33·53	40·20	70·46
·9829	10·92	13·52	23·70	·9509	34·10	40·84	71·58
·9819	11·69	14·46	25·34	·9499	34·57	41·37	72·50
·9809	12·46	15·40	26·99	·9489	35·05	41·90	73·43
·9799	13·23	16·33	28·62	·9479	35·55	42·45	74·39
·9789	14·00	17·26	30·26	·9469	36·06	43·01	75·37
·9779	14·91	18·36	32·19	·9459	36·61	43·63	76·45
·9769	15·75	19·39	33·96	·9449	37·17	44·24	77·53
·9759	16·54	20·33	35·63	·9439	37·72	44·86	78·61
·9749	17·33	21·29	37·30	·9429	38·28	45·47	79·68
·9739	18·15	22·27	39·03	·9419	38·83	46·08	80·75
·9729	18·92	23·19	40·64	·9409	39·35	46·64	81·74
·9719	19·75	24·18	42·38	·9399	39·85	47·18	82·69
·9709	20·58	25·17	44·12	·9389	40·35	47·72	83·64
·9699	21·38	26·13	45·79	·9379	40·85	48·26	84·58

ALCOHOL TABLES—*continued.*

Specific gravity, 15·5°.	Absolute Alcohol by weight. Per cent.	Absolute Alcohol by volume. Per cent.	Proof Spirit. Per cent.	Specific gravity, 15·5°.	Absolute Alcohol by weight. Per cent.	Absolute Alcohol by volume. Per cent.	Proof Spirit. Per cent.
·9369	41·35	48·80	85·53	·9079	54·52	62·36	109·28
·9359	41·85	49·34	86·47	·9069	55·00	62·84	110·12
·9349	42·33	49·86	87·37	·9059	55·45	63·28	110·92
·9339	42·81	50·37	88·26	·9049	55·91	63·73	111·71
·9329	43·29	50·87	89·15	·9039	56·36	64·18	112·49
·9319	43·76	51·38	90·03	·9029	56·82	64·63	113·26
·9309	44·23	51·87	90·89	·9019	57·25	65·05	113·99
·9299	44·68	52·34	91·73	·9009	57·67	65·45	114·69
·9289	45·14	52·82	92·56	·8999	58·09	65·85	115·41
·9279	45·59	53·29	93·39	·8989	58·55	66·29	116·18
·9269	46·05	53·77	94·22	·8979	59·00	66·74	116·96
·9259	46·50	54·24	95·05	·8969	59·43	67·15	117·68
·9249	46·96	54·71	95·88	·8959	59·87	67·57	118·41
·9239	47·41	55·18	96·70	·8949	60·29	67·97	119·12
·9229	47·86	55·65	97·52	·8939	60·71	68·36	119·80
·9219	48·32	56·11	98·34	·8929	61·13	68·76	120·49
·9209	48·77	56·58	99·16	·8919	61·54	69·15	121·18
·9199	49·20	57·02	99·93	·8909	61·96	69·54	121·86
				·8899	62·41	69·96	122·61
·9198	49·24	57·06	100·00P.s.	·8889	62·86	70·40	123·36
				·8879	63·30	70·81	124·09
·9189	49·68	57·49	100·76	·8869	63·74	71·22	124·80
·9179	50·13	57·97	101·59	·8859	64·17	71·62	125·51
·9169	50·57	58·41	102·35	·8849	64·61	72·02	126·22
·9159	51·00	58·85	103·12	·8839	65·04	72·42	126·92
·9149	51·42	59·26	103·85	·8829	65·46	72·80	127·59
·9139	51·83	59·68	104·58	·8819	65·88	73·19	128·25
·9129	52·27	60·12	105·35	·8809	66·30	73·57	128·94
·9119	52·73	60·56	106·15	·8799	66·74	73·97	129·64
·9109	53·17	61·02	106·93	·8789	67·17	74·37	130·33
·9099	53·61	61·45	107·69	·8779	67·58	74·74	130·98
·9089	54·05	61·88	108·45	·8769	68·00	75·12	131·64

ALCOHOL TABLES—*continued.*

Specific gravity, 15·5°.	Absolute Alcohol by weight. Per cent.	Absolute Alcohol by volume. Per cent.	Proof Spirit. Per cent.	Specific gravity, 15·5°.	Absolute Alcohol by weight. Per cent.	Absolute Alcohol by volume. Per cent.	Proof Spirit. Per cent.
·8759	68·42	75·49	132·30	·8439	81·80	86·96	152·40
·8749	68·83	75·87	132·95	·8429	82·19	87·27	152·95
·8739	69·25	76·24	133·60	·8419	82·58	87·58	153·48
·8729	69·67	76·61	134·25	·8409	82·96	87·88	154·01
·8719	70·08	76·98	134·90	·8399	83·35	88·19	154·54
·8709	70·48	77·32	135·51	·8389	83·73	88·49	155·07
·8699	70·88	77·67	136·13	·8379	84·12	88·79	155·61
·8689	71·29	78·04	136·76	·8369	84·52	89·11	156·16
·8679	71·71	78·40	137·40	·8359	84·92	89·42	156·71
·8669	72·13	78·77	138·05	·8349	85·31	89·72	157·24
·8659	72·57	79·16	138·72	·8339	85·69	90·02	157·76
·8649	73·00	79·54	139·39	·8329	86·08	90·32	158·28
·8639	73·42	79·90	140·02	·8319	86·46	90·61	158·79
·8629	73·83	80·26	140·65	·8309	86·85	90·90	159·31
·8619	74·27	80·64	141·33	·8299	87·23	91·20	159·82
·8609	74·73	81·04	142·03	·8289	87·62	91·49	160·33
·8599	75·18	81·44	142·73	·8279	88·00	91·78	160·84
·8589	75·64	81·84	143·42	·8269	88·40	92·08	161·37
·8579	76·08	82·23	144·10	·8259	88·80	92·39	161·91
·8569	76·50	82·58	144·72	·8249	89·19	92·68	162·43
·8559	76·92	82·93	145·34	·8239	89·58	92·97	162·93
·8549	77·33	83·28	145·96	·8229	89·96	93·26	163·43
·8539	77·75	83·64	146·57	·8219	90·32	93·52	163·88
·8529	78·16	83.98	147·17	·8209	90·68	93·77	164·33
·8519	78·56	84·31	147·75	·8199	91·04	94·03	164·78
·8509	78·96	84·64	148·32	·8189	91·39	94·28	165·23
·8499	79·36	84·97	148·90	·8179	91·75	94·53	165·67
·8489	79·76	85·29	149·44	·8169	92·11	94·79	166·12
·8479	80·17	85·63	150·06	·8159	92·48	95·06	166·58
·8469	80·58	85·97	150·67	·8149	92·85	95·32	167·04
·8459	81·00	86·32	151·27	·8139	93·22	95·58	167·50
·8449	81·40	86·64	151·83	·8129	93·59	95·84	167·96

ALCOHOL TABLES—*continued.*

Specific gravity, 15·5°.	Absolute Alcohol by weight. Per cent.	Absolute Alcohol by volume. Per cent.	Proof Spirit. Per cent.
·8119	93·96	96·11	168·24
·8109	94·31	96·34	168·84
·8099	94·66	96·57	169·24
·8089	95·00	96·80	169·65
·8079	95·36	97·05	170·07
·8069	95·71	97·29	170·50
·8059	96·07	97·53	170·99
·8049	96·40	97·75	171·30
·8039	96·73	97·96	171·68
·8029	97·07	98·18	172·05
·8019	97·40	98·39	172·43
·8009	97·73	98·61	172·80
·7999	98·06	98·82	173·17
·7989	98·37	99·00	173·50
·7979	98·69	99·18	173·84
·7969	99·00	99·37	174·17
·7959	99·32	99·57	174·52
·7949	99·65	99·77	174·87
·7939	99·97	99·98	175·22
Absolute Alcohol.			
·7938	100·00	100·00	175·25

Alcohol Calculations.—Proof spirit is defined by Act of Parliament to be a liquid of such a density that at 51° F. 13 volumes shall be equal in weight to 12 volumes of water of the same temperature. Such a spirit has a density of ·91984 at 60° F., and contains 49·24 per cent. by weight of alcohol and 50·76 of water. Spirits weaker than this are said to be *under* proof, U P ; when stronger *over* proof, O P. A spirit is said to be 20 degrees or 20 per cent. under proof when it contains at 60° F. 80 measures of proof spirit and 20 of water ; and 50° O P when 100 measures at 60° F. would require to be diluted to 150 measures to form proof spirit.

To find the percentage of alcohol by volume, multiply the percentage of alcohol by weight by the observed specific gravity, divide the product by ·7938.

Ex. $\frac{\cdot 8979 \times 59}{\cdot 7938} = 66\cdot 74$ per cent. of alcohol per volume.

To find the percentage volume of proof spirit multiply the percentage of alcohol by volume contained in the samples by 1·7525. Ex. 66·74 × 1·7525 = 116·96, or we may divide by ·5076 instead of multiplying by 1·7525.

Suppose it is required to find what proportion of proof, or any other strength of spirit a particular sample of alcohol contains or would contain when diluted, proceed as follows :

$$\frac{\text{P.C. of proof spirit in alcohol required} \times 100}{\text{P.C. of proof spirit in sample.}} = \left\{\begin{array}{l}\text{The number of vols. of the stronger spirit which will produce or be contained in 100 measures of the dilute spirit.}\end{array}\right.$$

Required the percentage of gin at 35° U P contained in a watered sample of 44° U P.

$$\frac{56 \times 100}{65} = 86{\cdot}15 \text{ per cent. per volume.}$$

Required the proportion of water which must be added to spirit of 25° O P to reduce it to 20° U P.

$\frac{80 \times 100}{125} = 64$. Hence 64 measures at 25° O P must be diluted to 100 to obtain a spirit of 20° U P.

To reduce spirit to a required strength. If the proof strength is known, note that each degree above proof of the dilution required *plus* 100 is the factor to start with. Thus, if we want to reduce 60° O P spirit to 20° O P, we take 120 volumes of the 60° O P, and add water to make it 160 volumes. If only the specific gravity is known, refer to our alcohol tables, and compare the overproof with the specific gravity, when the sum can be worked out. The following are some of the more useful figures in that connection :

Specific Gravity.	Percentage of Proof Spirit.
0·829	160 or 60° O P.
0·833	158 „ 58° „
0·837	156 „ 56° „
0·8405	154 „ 54° „
0·8475	150 „ 50° „
0·856	145 „ 45° „
0·864	140 „ 40° „
0·872	135 „ 35° „
0·886	125 „ 25° „
0·9005	115 „ 15° „
0·907	110 „ 10° „
0·920	100 „ proof

Owing to the contraction of volume when alcohol and water are mixed, there is no regular increment of difference in the relation between the degrees and the specific gravities. This is evident from the above figures, but the following are approximately correct factors :

There is an increase in specific gravity of

0·0018	for each	degree	between	60°	and	50° O P.
0·0017	„	„		50°	„	40° „
0·0015	„	„		40°	„	30° „
0·00147	„	„		30°	„	20° „
0·00137	„	„		20°	„	10° „
0·0013	„	„		10°	„	0° „

With the aid of the factors in the first column it will be possible to work out the above rule without referring to an alcohol table. Were it not for the contraction which occurs when water and alcohol are mixed, the following rule as to mixtures of liquids of different specific gravity could be used with certainty. The object of the rule is to determine what proportions of two liquids differing in density must be taken to produce a liquid of a required density.

Let the specific gravity of the lighter liquid be A, *the specific gravity of the heavier* B, *and the required specific gravity* C, *then—*

C – A = volume of B to be taken.
B – C = volume of A to be taken.

If strict exactitude is not required, the rule may be used for spirit mixtures, ammonia solution and water, etc.

CORRECTION FOR TEMPERATURE in specific gravity of mixtures of alcohol and water can be determined from the following formula :

$$w = w' \pm t \left(\cdot 00014 \times \frac{1 - w'}{150} \right)$$

Where w' = the observed density
w = the density at 15·5° C.
t = the difference between the normal and observed temperatures in degrees Centigrade
·00014 = the coefficient of expansion for alcohol.

The + sign is used if the temperature of the experiment is above 15·5° C. ; the – sign if below that point.

THERMOMETRIC SCALES.—To convert Fahrenheit-heat degrees to Centigrade terms, subtract 32, multiply by 5, and divide by 9.

To convert Centigrade degrees into Fahrenheit multiply by 9, divide by 5, and add 32.

°C.	°F.	°C.	°F.	°C.	°F.	°C.	°F.
1	33·8	14	57·2	27	80·6	40	104·0
2	35·6	15	59·0	28	82·4	41	105·8
3	37·4	16	60·8	29	84·2	42	107·6
4	39·2	17	62·6	30	86·0	43	109·4
5	41·0	18	64·4	31	87·8	44	111·2
6	42·8	19	66·2	32	89·6	45	113·0
7	44·6	20	68·0	33	91·4	46	114·8
8	46·4	21	69·8	34	93·2	47	116·6
9	48·2	22	71·6	35	95·0	48	118·4
10	50·0	23	73·4	36	96·8	49	120·2
11	51·8	24	75·2	37	98·6	50	122·0
12	53·6	25	77·0	38	100·4	51	123·8
13	55·4	26	78·8	39	102·2	52	125·6

WEIGHTS AND MEASURES OF THE METRICAL SYSTEM.

Weights.

1 Milligramme = the thousandth part of one gramme or 0·001 gramme.
1 Centigramme = the hundredth part of one gramme or 0·01 gramme.
1 Decigramme = the tenth part of one grm. or 0·1 grm.
1 Gramme = weight of a cubic centimetre of 1·0 „ water at 4° C.
1 Decagramme = ten grammes „ 10·0 „
1 Hectogramme = one hundred grms. „ 100·0 „
1 Kilogramme = one thousand grms. „ 1000·0 „

Measures of Capacity.

1 Millilitre =	1	cub. centim.	or the mea. of	1	grm. of water
1 Centilitre =	10	„		10	„ „
1 Decilitre =	100	„		100	„ „
1 Litre =	1000	„		1000	„ (1 kilo.)

Measures of Length.

1 Millimetre = the thousandth part of one metre or 0·001 metre.
1 Centimetre = the hundredth part of one metre or 0·01 metre.
1 Decimetre = the tenth part of one metre or 0·1 metre.
1 Metre = the ten-millionth part of a quarter of the meridian of the earth.

Relation of the Weights of the British Pharmacopœia to the Metrical Weights.

1 Pound = 453·5925 grammes
1 Ounce = 28·3495 „
1 Grain = 0·0648 „

Relation of Measures of Capacity of the British Pharmacopœia to the Metrical Measures.

1 Gallon	=4·543487	litres		
1 Pint	=0·567936	,,	or 567·936	cub. centims.
1 Fluid Ounce	=0·028396	,,	28·396	,,
1 Fluid Drachm	=0·003549	,,	3·549	,,
1 Minim	=0·000059	,,	0·059	,,

Relation of the Metrical Weights to the Weights of the British Pharmacopœia.

1 Milligramme	=	0·015432	grs.
1 Centigramme	=	0·15432	,,
1 Decigramme	=	1·5432	,,
1 Gramme	=	15·432	,,
1 Kilogramme	=2 lb. 3 oz. 119·8 grs.	or 15432·348	,,

Relation of the Metrical Measures to the Measures of the British Pharmacopœia.

1 Millimetre	=	0·03937	inch
1 Centimetre	=	0·39371	,,
1 Decimetre	=	3·93708	inches
1 Metre	=39·37079	,,	or 1 yard 3·7 inches

1 Cubic centimetre = 15·432 grain-measures
1 Litre=1 pint 15 oz. 2 dr. 11 m. or 15432·348 grain-measures

CO-EFFICIENTS REQUIRED IN VOLUMETRIC ANALYSIS.

Normal Acid Solution.

Oxalic acid	·063
Sulphuric acid	·049
NH_3	·017
NH_4HO	·035
NH_4HCO_3 . $NH_4NH_2CO_2$	·0523
$Na_2B_4O_7$. $10H_2O$	·191
Ca2HO	·037
CaO	·028
$CaCO_3$	·05
$Ba(HO)_2$	·0855
$Ba(HO)_2 8H_2O$	·1575
$BaCO_3$	·0985
MgO	·02
$MgCO_3$	·042
KHO	·056
K_2CO_3	·069
$K_2C_4H_4O_6$ (converted into K_2CO_3)	·113
$KHC_4H_4O_6$	·188
$K_3C_6H_5O_7$	·102
$KC_2H_3O_2$	·098
NaHO	·04
Na_2CO_3	·053
Na_2CO_3 . $10H_2O$	·143
$NaHCO_3$	·084

Normal Soda Solution.

Sodium hydrate	·04
$HC_2H_3O_2$ (acetic)	·06
$H_3C_6H_5O_7H_2O$ (citric)	·07
HCl	·0365
HBr	·081
HI	·128
HNO_3	·063
H_2SO_4	·049
$H_2C_4H_4O_6$ (tartaric)	·075
$HC_3H_5O_3$ (lactic)	·09
$H_2C_2O_4 2H_2O$ (oxalic)	·063

$\frac{N}{10}$ Nitrate of Silver Solution.

Argentic nitrate	·017
CN	·0052
HCN	·0054
KCN	·01302
NH_4Cl	·00535
KCl	·00745
NaCl	·00585
KBr	·0119
NaBr	·0103
Cl	·00355

$\frac{N}{10}$ Iodine Solution.

Iodine	·0127
SO_2	·0032
H_2SO_3	·0041
As_2O_3	·00495
$Na_2S_2O_3 5H_2O$	·0248
$Na_2SO_3 7H_2O$	·0126
$K_2SO_3 2H_2O$	·0097

$\frac{N}{20}$ Bichromate of Potassium.

Potassium bichromate	·01475
Fe (ferrous)	·0168
$FeSO_4$	·0456
$FeSO_4 2H_2O$	·0564
$FeSO_4 7H_2O$	·0834
$Fe_3(AsO_4)_2$	·0446
$Fe_3(PO_4)_2$	·0358
$FeCO_3$	·0348
Fe_3O_4	·0696
FeO	·0216

$\frac{N}{10}$ Hyposulphite of Soda Solution.

Hyposulphite of sodium	·0248
I	·0127
Cl	·00355
Br	·0080

$\frac{N}{10}$ Permanganate of Potassium.

Potassium permanganate	·00316
Fe (ferrous)	·0056
$FeSO_4$	·0152
$FeSO_4 . 7H_2O$	·0278
$FeCO_3$	·0116
FeO	·0072
$H_2C_2O_4 2H_2O$	·0063
$CeC_2O_4 . 9H_2O$	·354

Nitrometer Analysis.

Each cc. of NO at N.T.P. equals gramme of	·00281	HNO_3
	·00241	N_2O_5
	·00450	KNO_3
	·00334	$C_2H_5 . NO$
	·0026	Urea
Each cc. of CO_2 at N.T.P. equals gramme of	·0042	$(NH_4)_2CO_3$
	·001967	CO_2

Nitrogen × by factor 6·33 = Albuminoids.

USEFUL DATA.

1 metre=39·37 inches, or 1 yard and 3⅓ inches (nearly).
25 millimetres=1 inch (nearly).
30 centimetres=1 foot (nearly).
8 kilometres=5 miles (nearly).
6½ square centimetres=1 square inch (nearly).
9¼ square decimetres=1 square foot (nearly).
1 kilogramme=15432·3 grains, or 2·2 pounds (nearly).
28 grammes=1 ounce avoirdupois (nearly).
455 grammes=1 pound avoirdupois (nearly).
65 milligrammes=1 grain (nearly).
1 litre=35·2 ounces, or 1·76 pints (nearly).
100 cubic centimetres=3½ ounces (nearly).
1 cubic centimetre=15·4 grains, or 16·7 minims (nearly).
1 cubic inch=16·4 cubic centimetres (nearly).
1 gallon=4·5 litres (nearly).
Cubic feet × 6·2355=gallons.
Cubic inches × ·003607=gallons.
To reduce ounces to grammes, multiply by 28·349.
To reduce grains to grammes, multiply by ·0648.
To reduce grammes to grains, multiply by 15·432.
To reduce pints to cubic centimetres, multiply by 567·936.
To reduce gallons to litres, multiply by 4·548.
To reduce litres to gallons, multiply by ·22.
To reduce kilogrammes to pounds, multiply by 2·2046.
To reduce centimetres to inches, multiply by ·3937.
To reduce inches to centimetres, multiply by 2·54.
To convert grammes per 100 cubic centimetres to grains per fluid ounce, multiply by 4·375.
To convert grammes per 100 cubic centimetres to grains per gallon, multiply by 700.
To convert parts per million into grains per gallon × ·07.
To convert grains per gallon into parts per million ÷ ·07
To convert parts per 100,000 into parts per million × 100.
To convert grains per gallon (=parts per 70,000) into parts per 100,000 ÷ ·7.

ATOMIC WEIGHTS OF THE ELEMENTS.

Element.	Symbol and Atomicity.	Atomic Weight.
Aluminium	Al III. IV.	27·5
Antimony	Sb III. V.	120
Arsenic	As III. V.	75
Barium	Ba II.	137
Beryllium	Be II. III.	9·4
Bismuth	Bi III. V.	208
Boron	Bo III. V.	11
Bromine	Br I. III. V. VII.	80
Cadmium	Cd II.	112
Cæsium	Cs I.	133
Calcium	Ca II.	40
Carbon	C IV. II.	12
Cerium	Ce III. IV.	138
Chlorine	Cl I. III. V. VII.	35·5
Chromium	Cr IV. VI.	52·5
Cobalt	Co II. IV.	59
Copper	Cu II.	63
Didymium	Di IV.	145
Erbium	E II.	169
Fluorine	F I.	19
Gallium	Ga IV.	69
Gold	Au I. III.	196·7
Hydrogen	H I.	1
Indium	In III.	113·4
Iodine	I I. III. V. VII.	127
Iridium	Ir II. IV. VI.	193
Iron	Fe II. IV. VI.	56
Lanthanum	La IV.	139
Lead	Pb II. IV.	207
Lithium	Li I.	7
Magnesium	Mg II.	24
Manganese	Mn II. IV. VI. VII.	55
Mercury	Hg II.	200
Molybdenum	Mo VI.	96

ATOMIC WEIGHTS OF THE ELEMENTS—

continued.

Element.	Symbol and Atomicity.	Atomic Weight.
Nickel	Ni II. IV.	58·8
Niobium	Nb V.	94
Nitrogen	N III. V.	14
Osmium	Os II. IV. VI. VIII.	199
Oxygen	O II.	16
Palladium	Pd II. IV. VI.	106·5
Phosphorus	P III. V.	31
Platinum	Pt II. IV. VI.	194·3
Potassium	K I.	39
Rhodium	Rh II. IV. VI.	104
Rubidium	Rb I.	85
Ruthenium	Ru II. IV. VI. VIII.	104
Selenium	Se II. IV. VI.	79
Silicon	Si IV.	28
Silver	Ag I.	108
Sodium	Na I.	23
Strontium	Sr II.	87·5
Sulphur	S II. IV. VI.	32
Tantalum	Ta V.	182
Tellurium	Te II. IV. VI.	128
Thallium	Tl I. III.	204
Thorium	Th IV.	231·5
Tin	Sn IV.	118
Titanium	Te IV.	48
Tungsten	W IV. VI.	184
Uranium	U VI. IV.	240
Vanadium	V III. V.	51·2
Yttrium	Y IV.	89
Zinc	Zn II.	65
Zirconium	Zr IV.	90

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