The accelerated freeze-drying (AFD) method of food preservation / a report on some work of the Research Establishment and Experimental Factory of the Ministry of Agriculture, Fisheries and Food, Aberdeen, Scotland, between 1955 and 1960.

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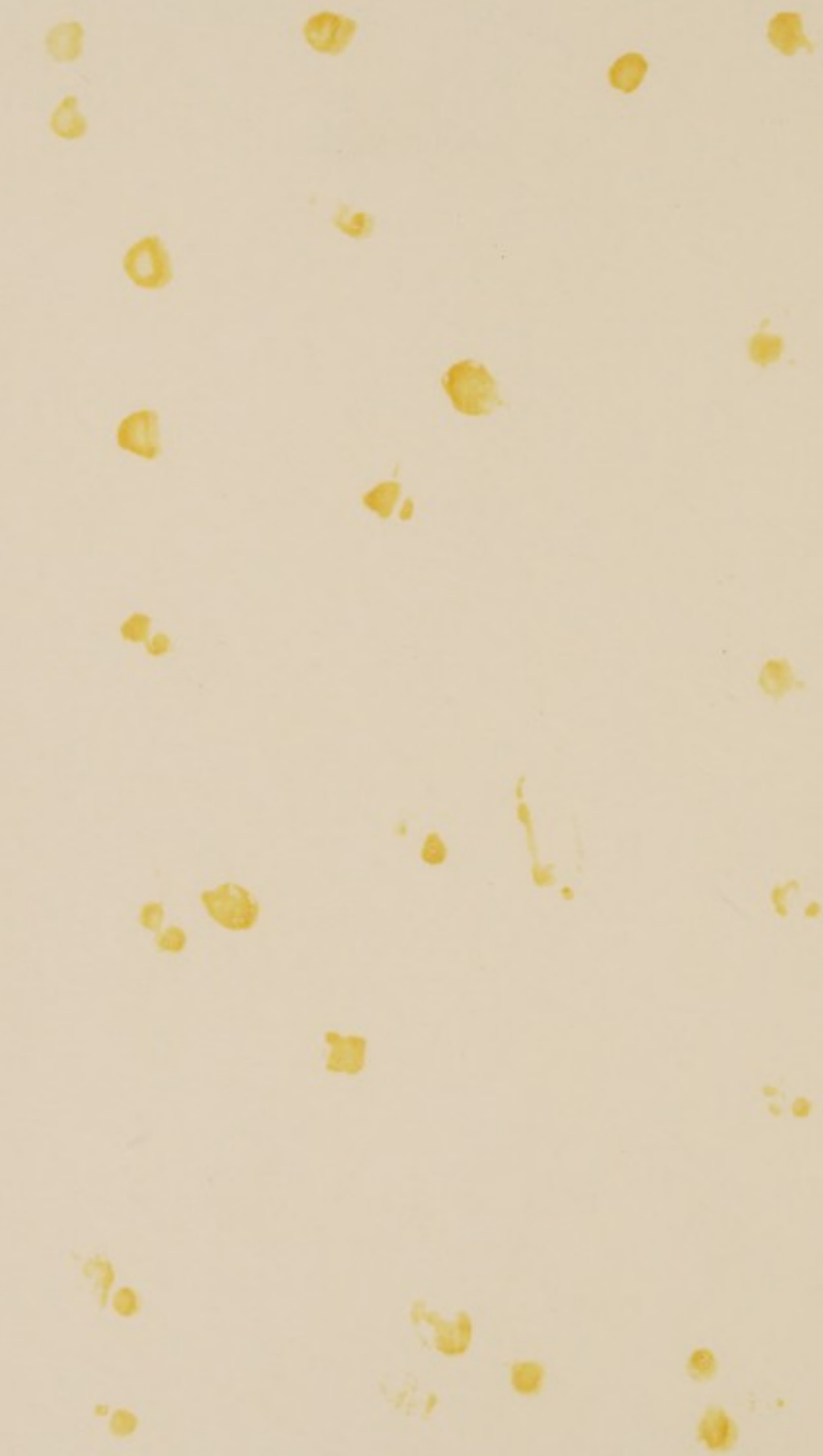

THE ACCELERATED FREEZE-DRYING METHOD OF FOOD PRESERVATION

MINISTRY OF
AGRICULTURE, FISHERIES AND FOOD

THE ACCELERATED FREEZE-DRYING (AFD) METHOD OF FOOD PRESERVATION

A report on some work of the Research Establishment and Experimental Factory of the Ministry of Agriculture, Fisheries and Food, Aberdeen, Scotland, between 1955 and 1960

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CHAPTER I

Introduction

Historical

Since man first killed animals and harvested plants he has had the problem of keeping the food from day to day and from season to season. From the earliest times, therefore, he has had recourse not only to fresh foods but to processed foods. With increasing scientific knowledge new methods of preservation have been developed and still newer ones are fast coming into existence.

The most obvious and widespread forms of deterioration are due to microbial attack. Bacteria, yeasts and fungi will compete with man for his food if permitted, and the most elementary forms of preservation have been, without knowledge of their nature, methods of dealing with this problem. Drying is probably the oldest form of preservation, developed as a practical art which in fact deprived the microbial population of the water necessary for growth and reproduction. In such a way fish has been split and slowly dried in the wind, grain has been dried, and products such as biltong and pemmican have been made from meat cut thin and dried in the sun, for many thousands of years.

Salting, another ancient process, in effect does the same by depriving microbes of available water, and is usually associated with a measure of drying. Smoking adds to the drying effects the additional benefits of chemical antibiotics and antioxidants. In due course there evolved pickled and fermented products which, by changing the conditions of acidity or salinity, either prevented the growth of microbes or suppressed undesirable ones and encouraged acceptable ones. These processes, together with natural freezing or chilling, which retard microbial growth, were substantially the total resources of mankind for the preservation and transportation of food from prehistoric times until comparatively recently.

Only 150 years ago, as a direct result of its appreciation of the problems of the armed forces, the French government sponsored the development of an improved process of preserving food which, starting from Nicolas Appert's method of hermetically sealing it after heating in glass jars in a water-bath, led to the food canning industry of today. It is interesting to note in this context that a tremendous impetus was given to the development of canning as a result of military use during the American Civil War. In even more recent times mechanical refrigeration has moved into a foremost place as a method of food preservation in places and under conditions where its somewhat exacting requirements can be met.

As has been said, dehydration is of a venerable antiquity; and the British Food Manufacturing Industries' Research Association has samples of dried foods from Jericho, four thousand years old. Seven hundred years ago Marco Polo found the Tartars drying milk in goatskin bottles in the smoke of their camp fires; and in the seventeenth century the American Indians' process for making pemmican by drying lean meat in the sun, powdering it and mixing

it with melted fat, was adopted by the early settlers, and later by Arctic explorers.

In 1780 in England the first patent for dehydration was granted to J. Graefer, who scalded vegetables in boiling salt water and then dried them in a heated room. The Royal Navy expedition which set out to search for Franklin in 1852 carried carrots and potatoes which had been dried in hot air for 20 to 30 hours. They were then compressed into cakes, wrapped in tinfoil and packed in air-tight cases of tin or zinc. They were not popular. One report said 'The carrots require too much attention and therefore are not fit for sea service as rations' and 'The seamen did not take to the potatoes'.

During the Crimean War an attempt was made to combat scurvy by using potatoes dried by Edwards' patented process; which was to boil them, press them through small holes as fine threads and dry them on steam-heated plates. They were disliked as much as the carrot and meat powders also provided.

The first World War saw a considerable production of air-dried vegetables and the second an extension to milk, eggs and meat. Hardly three years had elapsed after the 1939–45 war, however, when Commonwealth defence scientists realized, with some alarm, that both research and production on the dehydration of foodstuffs had, to all intents and purposes, ceased. From a defence viewpoint this was a matter of some importance, particularly to the United Kingdom, where the quantity of food necessarily imported each year contains something like 3 million tons of water, and any reduction in the water content would reduce the amount of shipping required. Since about three-quarters of meat and fish and possibly nine-tenths of fruit and vegetables are water the potential saving in packaging, storage and transportation resulting from dehydration could be immense.

Dehydration has a further advantage, especially in periods of emergency, in that the biological material we call food is rendered considerably less perishable by this process. In the first instance, spoilage of foodstuffs by microbes is prevented by the removal of water and, secondly, the processes normally used for the dehydration of food stop the activity of the enzyme systems which are responsible for the metabolism of the living tissues. Under normal conditions these systems persist to a large extent in the food, causing first a 'ripening' and eventually a complete breakdown or rotting. Even the purely chemical reactions which can and do occur among the myriad components of the food are inhibited by lack of water. The result of dehydration is therefore a more or less stable food product, which can be kept without freezing or refrigeration.

During the war this process, with its fundamental advantages in defence, had been applied on a large scale to the feeding of the populace, and on a relatively smaller scale, but no less significant, to the production of military rations for use throughout the world. Nevertheless, despite an advance in knowledge of blanching and sulphiting of vegetables, the war had ended with little more than a full realization of the problems and limitations of the process, when production and research ceased.

It was in 1948 then, that the attention of Commonwealth countries was drawn to this hiatus in food research, and the advice to member-governments to stimulate further progress was acted upon by the British Government and resulted in the opening, in 1951, of the Research Establishment and Experimental Factory in Aberdeen. This latter provision was based on the hard-won experience of the war years, when it had been found difficult, if not impossible, to conduct adequate scientific experiments in a factory devoted to production.

In the case of dehydration, for example, the processes then available had been devised on a laboratory scale, and in some cases on a small pilot scale, by 1940 or thereabouts; but it was some years before factory production got into gear. It was considered to be common prudence under modern conditions to develop processes right up to factory scale; and, indeed, to establish an industrial potential in peace time.

The Experimental Factory, therefore, was designed to be capable of developing a food process from laboratory, through pilot-scale, to full commercial scale demonstration. This made it unique in the world and it is hardly surprising that it attracted thousands of visitors, representing scientific, commercial, governmental and defence interests, from all over the world.

Coincident with an investigation of hot-air drying techniques, in which significant improvements were made, the possibilities of vacuum-drying (with its advantages in quality, stemming from the reduced heat treatment) were examined. The most promising device was one developed by the firm A/S Atlas, of Copenhagen, towards the end of the war, and a vacuum-contact-dehydration (VCD) plant was purchased from them for the Experimental Factory. In the event, although it did make a substantial advance in technique, the elusive qualities of ease and completeness of reconstitution were missing from its products. The engineers of the Research Establishment were, however, able to adapt it so that hand-in-hand with the biological scientists involved they reached the stage when food could be freeze-dried in it.

Now freeze-drying had long been known as a highly desirable, but rather academic and expensive process, taking 24 hours or longer, which could only reasonably be applied to high-priced pharmaceuticals and biological materials. In essence the same process occurs when wet sheets are hung out to dry on a frosty day. The water in them freezes, making the sheets as stiff as boards, then 'sublimes' (i.e., passes from the solid to the vapour state without passing through a liquid phase) and the sheets become dry without the ice ever melting.

This Report discloses in detail how a process was devised whereby a wide variety of foods could be dried in this fashion, hundreds of pounds at a time and in a period of about 8 hours. The process was termed Accelerated Freeze-Drying (AFD) and seemed for the first time to bring freeze-drying within reach of commercial feasibility for foods.[1] [2]

This was a big advance. Foods of all kinds can be treated in this fashion. By quick freezing, the constituents of the tissues, solids and fluids, are immobilized; and thereafter the water can be removed by sublimation at sub-freezing temperatures so that practically no concentrations of salts occur, and little denaturation of proteins or gelling of starches or twisting of fibres. As a result the final product is as little changed as it can be, and when water is added it instantly perfuses the microscopic honeycomb of sponge-like tissue, swelling it out and reconstituting the food in substantially its original condition within a few minutes.

By using freeze-dried foods the military requirements of lightweight, stable, attractive and nutritious meals can be met, and field trials continue to take place. Well over one hundred expeditions have been supplied with a variety of dehydrated meat, fish, fruit and vegetables from Aberdeen, including the Everest and trans-Antarctic expeditions, and interest in the products has been shown by the U.S. space-flight agencies.

The ultimate in these new foods is the 'instant meal', made up of pre-cooked freeze-dried foods, in lightweight plastic containers, to which only hot water need be added to produce a full and attractive meal within 15 minutes. This appears to have advantages for the highly mobile field forces of modern armies, and may well also have an impact on civilian life. In any case it has always been an objective of the project to provide the technical basis for a peace time industry.

Although the principles of accelerated freeze-drying had been established by 1958, however, it was not possible to demonstrate a 'Mark I' commercial prototype plant until 1960, when Armour and Company, of Chicago, provided the Experimental Factory with the means for doing so. During the ensuing year, up to the closure of the Research Establishment in March, 1961, the opportunity was seized to make a considerable volume of samples and to demonstrate the method and equipment to the food industry, many leading firms in which are now further developing the process.

Principles of the process

A proper understanding of the technical arguments contained in the appropriate chapters of the Report must be based on an appreciation of what is going on during freeze-drying and what the limitations and objectives are. Particularly for the non-specialist, therefore, it is as well to consider these points first.

The lyophilization, or freeze-drying, of biological materials generally calls for true, rapid and deep freezing, followed by sublimation of the ice without thawing. The objective is to establish the whole mass as a frozen entity without the growth of large ice-crystals to disrupt cells and membranes and change ionic concentrations; to avoid supercooling with consequent violent heat movements and volume and concentration changes; and then to be able to hold the mass without low-temperature enzymic or chemical activity. Thereafter the frozen water only is to be evaporated, without melting and without undue heating of the dried tissue resulting.

If all these requirements are observed, the resulting product can be so little altered that the mere addition of water almost immediately restores it to its original condition so that viruses and microbes can actually resume living processes, sperm can be successfully used for artificial insemination, and human tissues retain such subtle characteristics as specific antigenic properties.

The first practical requirements resulting from the above are to determine the maximum temperature of true freezing and the minimum temperature of melting. In many biological materials (and especially those containing sugars) it is common to experience supercooling, from which state true freeze-drying cannot take place, to the detriment of quality. There are methods, including electrical conductivity methods, of detecting this; and it may be necessary to take the temperature very far indeed below the melting point to prevent it. Thereafter, it is advantageous to dry at as high a temperature as possible consistent with not melting the frozen mass.

In general, then, it is not sufficient to say that a foodstuff is at, say, $-20°C$. Its condition may be quite different if it has arrived at this temperature by cooling down from room temperature, from what it would be if it had cooled first to $-100°C$ and then warmed up to $-20°C$. (And the speed of freezing is also important as it could make significant differences in the cellular conditions of the foodstuff.)

This exercise establishes fixed points to be arrived at in the freezing and subsequent drying cycles, and any departure from them carries penalties in quality. Some departure may be necessary to make the process economically feasible, and it is important to recognize the point of compromise, which cannot be judged rationally without the necessary experimental data, which will vary with each commodity.

Another point of reference which defines the optimal process is the temperature (and time) to which the dried parts of the food can be subjected. While it is true that less heat damage is suffered by the dry than by the wet material, nevertheless protein denaturation, for example, (and so insolubility, dryness and toughness) proceeds at a rate depending upon both time and temperature. To quote another example, if the highest temperature permitted to the outer surface of beefsteak during a drying run is 60°C, the product of even uncooked meat is brown. Reduced to a 30° maximum it may remain pink, but 2 hours may be added to the drying time.

To return to the actual phenomenon of freeze-drying. At the ice surface in the frozen food there is a vapour pressure of molecules escaping. This is directly proportional to the temperature of the ice, since it is a measure of the activity at the surface. Whether the removal of these water molecules is in the form of a viscous flow from a region of higher pressure to one of a lower pressure, or whether it is purely random molecular motion made more free by the removal of impeding molecules, the requirement is the same. To get the water away the pressure in the cabinet must be less than the vapour pressure at the ice surface, and therefore the evacuating equipment must be capable of removing the maximum output of vapour without a build-up in pressure.

One more fact is basic to this review. The escape of the faster-moving water molecules from the ice surface causes a loss of energy from the ice, whose temperature would then fall if it were not replaced ('evaporative cooling'), with consequent lowering of vapour pressure and slowing of the process. The amount of water which comes off is proportional to the energy supplied, and it is for this reason that the aim has to be to supply heat energy as fast as possible, always consistent with not melting the ice. This transmission of heat energy is always a problem and is increased in the late stages of drying, as discussed below, by the intervention of an insulating layer of dried foodstuff round the ice.

This dry layer appears, of course, the moment the surface is dehydrated, and it increases until the last of the ice sublimes. Its properties determine the whole course of events during drying. For one thing it is a porous mass whose characteristics depend on the commodity, its rate of freezing, and even the direction of cutting; but it inevitably and increasingly inhibits the outflow of vapour from the retreating ice surface, as it increasingly insulates the ice from applied heat. In the balance between heat input and vapour outflow one or the other may be the limiting factor, according to the material and sometimes the limitation swings from the one factor to the other during the drying cycle. If the vapour diffusion is limiting, the applied heat must be reduced to avoid melting the ice, and the process is prolonged. If the resistance to heat transfer is the limiting factor there is no danger of melting the ice, but the temperature gradient cannot be increased beyond the limit imposed by the critical temperature for the dried outside material, so once again the process is prolonged.

The optimum drying period is observed when these two factors are in balance by a judicial processing involving particle size and direction of cut; pretreatment

including blanching, rate of freezing, and cooking; and design of equipment to correlate heat input and vapour egress. All these points are the subject of the Report.

The Report

All that can be offered in this Report is the experience of the Aberdeen staff within the limitations of time and facilities that were available to them. They are well aware, therefore, that much remains to be done in the realm of further engineering advances and the never-ending striving for the ultimate in product quality. Product development is, of course, very much the responsibility of the food manufacturer, but indications of the possible scope of application are given in detail. Process development will no doubt be shared by the food manufacturers and the food machinery manufacturers, but it is hoped that the experience reported here will be of value to them. It is certain that the Mark I commercial equipment will not be available in the future, for it naturally became obsolescent as soon as it was created; but there is much that was done with it that will refer directly to any future equipment for accelerated freeze-drying, and this is also true of the work reported on the research plant EVE (Experimental Vacuum Equipment).

SECTION A

Engineering

CHAPTER II

Theoretical considerations

THE investigation of dehydration, as it was understood at the Experimental Factory, was taken to mean the application of special techniques to the reversible removal of water from a foodstuff, with the minimum damage to the appearance, flavour, texture and nutritive value. Drying may be by evaporation from the liquid phase, brought about by exposure of the foodstuff to streams of heated air (hot-air drying), or in the case of purées or solutions by atomization into heated air (spray-drying) or by contact with heated rollers (drum-drying). It may be by a combination of heat and low pressure (vacuum-drying). None of these seemed to meet the requirements, however, anything like as nearly as freeze-drying, the drying of the frozen food by sublimation under conditions of very low pressure.

Hot-air drying was indeed further developed for many foodstuffs, and has reached the stage where it can yield acceptable products, especially with certain vegetables. Reasonably good colour, texture and flavour retention are possible, but to achieve this and maintain the drying time within acceptable limits, the dimensions of the pieces to be dried must be small; in practice a minimum dimension of not more than $\frac{1}{4}$ in. is necessary if satisfactory drying (and satisfactory rehydration) is to be achieved, and it is preferable to keep it below this value, e.g. $\frac{3}{16}$ in. or even $\frac{1}{8}$ in. A root vegetable can thus be cut to slices of any size, provided the thickness does not exceed $\frac{1}{4}$ in., or into strips or dice. The smaller the dimension of the piece the faster will be the drying operation (and the more rapid the rehydration when the product is being prepared for eating), but unfortunately the rate of throughput in the cutting machine falls, the leaching losses during scalding increase, the spillage in handling increases, and the appearance of the final product is less attractive, since thinness of cut gives a rather translucent appearance lacking in the normal intensity of colour. In the majority of cases reconstitution of air-dried foods is slow, for example, root vegetables cut to a minimum thickness of $\frac{3}{16}$ in. require 1 to 2 hr soaking in water before they can be cooked for eating. Thinner pieces reconstitute more quickly and if very thin ($\frac{1}{8}$ to $\frac{3}{32}$ in.) can be prepared for eating in about 10 min. Air-drying of vegetables was therefore found to be rather limited in application though it is capable of yielding certain quite attractive products.

Meat products can be air-dried in the form of cooked minces only, and are characterized by a marked darkening of colour, toughening of texture, roast off-flavours and prolonged reconstitution time. The same characteristics are evident in fruits, with the possible exception of apple which yields a fairly attractive product.

In drying from the liquid phase heat is required not only to increase the water-carrying capacity of the air but, more important during the later stages of drying, also to increase the kinetic energy of the water molecules in the foodstuff by virtue of which the water is able to migrate from the interior of the

pieces to the outer surfaces from which evaporation actually takes place. Thus the higher the temperature the more rapid will be the rate of drying. However, high temperatures accelerate chemical reactions, some of which lead to deleterious changes in the colour, texture and flavour of the product. Time as well as temperature is important; excessive temperatures may be avoided by using lower temperatures for a longer time, but prolonged exposures even to these lower temperatures can still cause the various deteriorative reactions. Ideally, then, a system of drying should employ low temperatures for short times, and the idea of drying at low pressures, i.e., in a partial vacuum, has been attractive for this reason. At reduced vapour pressure, other things being equal, water evaporates more rapidly than under normal pressure, and thus it might be expected either that drying times could be reduced, or that drying might be carried out at lower temperatures without any prolongation of drying time. Also there is the advantage that oxidation of components of the foodstuff should be minimized in the absence of air.

In practice these expectations were not entirely fulfilled, because for a large part of the drying cycle the uncontrollable factor limiting the rate of drying at reduced pressures was, just as in hot-air drying, the rate at which water could migrate from the interior to the dried surface of the material.

Drying from the liquid phase had other undesirable characteristics. The liquid in tissues is a solution containing mineral and organic substances; rapid evaporation is apt to lead to deposition of these substances on the surface of the material, particularly in meats, where the deposit forms a pellicle which retards subsequent dehydration. There is also reason to believe that the local high concentrations of salts brought about in this manner caused denaturation of the proteins and resulted in the noticeable toughening of texture. In vegetables there was no evidence of the formation of substantial surface deposits, but there was some indication of a movement of solutes from the surface to the interior of the piece, along the concentration gradient produced as a result of the increasing concentration of the solution in the outer layers by surface evaporation. In the more concentrated solution finally resulting at the centre of the piece deteriorative reactions were more liable to occur, e.g., the browning often exhibited by the cores of dehydrated potato strips.

Shrinkage of the tissue is also inevitable when evaporation takes place from the liquid phase; the minute pores of the cell walls or between meat fibres and fibrils become closed, and the whole tissue becomes hard and glazed or horny in appearance. Penetration of water is greatly retarded by this shrinkage—hence the relatively slow reconstitution of foods that have been dried in this manner, whether in hot air or in vacuum. Although vacuum-drying did in general give a better product than air-drying, the increase in quality did not seem to justify the substantially higher cost.

When a foodstuff is frozen the water in it forms crystals of ice, smaller or larger according to whether the rate of freezing is rapid or slow. If the ice could be sublimed the food should, theoretically, not change in volume, but retain its original shape, essentially as a skeleton with cavities left by the evaporated ice. Such a food would take up water very rapidly when reconstitution was required. Further, in order for ice to sublime, and not to be thawed, drying would have to be carried out at sub-zero temperatures, and thus heat damage hould be virtually impossible. Again, since the water in the food is solid throughout the period of drying, no complications should arise because of

migration of solids. In addition it appears that foodstuffs may well be most susceptible to heat damage when the moisture content is about 20 per cent. Since the part containing ice holds its original proportion of water, and when the ice is sublimed away it is well below the danger level, it is possible that this feature would further reduce the possibility of thermal deterioration.

In practice an extension of vacuum-drying technique enabled this process of freeze-drying to be carried out. If a vacuum dehydration cabinet is operated at pressures of 1 mm Hg or less, the moisture in the foodstuff freezes and subsequent application of heat, carefully controlled, results in sublimation of the ice. Dry tissue is in most cases fairly strong mechanically, and since the 'wet' tissue is in the frozen state, the whole foodstuff can withstand fairly rough treatment during the process, and in fact shrinks little if at all during drying. The resulting product was in every case (with the possible exception of potatoes) superior to that obtainable with vacuum or hot-air drying. However, it should be stressed that the improvements obtained with freeze-drying, as compared with the alternative methods, were much more marked with some foodstuffs than with others, e.g., thick pieces of freeze-dried meat were infinitely better than their equivalent air-dried counterpart, but the differences between samples of cabbage treated by the two techniques were far less marked. The cost of the dehydration stage in freeze-drying is more expensive than in hot-air drying, and it is therefore apparent that food processors should be highly selective in deciding which raw materials should be handled by the different methods.

PHYSICAL REQUIREMENTS IN FREEZE-DRYING

Since the method involved the sublimation of ice, it was evident that the natural liquid component of the foodstuff must be converted to ice before freeze-drying could commence. In some cases it was preferable to pre-freeze the material before loading into the cabinet, while in other foods it was possible to cool the raw material sufficiently simply by loading it into the drying cabinet and evacuating the system to a low absolute pressure. In the latter case water evaporated from the cut surfaces; the latent heat absorbed from the foodstuff by this evaporation progressively lowered the temperature of the remaining water, until eventually freezing took place. It should be remembered, however, that during this period a substantial amount of liquid-phase drying had occurred, and as had been mentioned above this is always accompanied by some undesirable changes, so that if the method is to be acceptable the depth of liquid-phase drying into the foodstuff must be limited. For almost any product the quantity of water which must be evaporated to freeze the remainder was found to be about 20 per cent of the initial content, and it was necessary to ensure that it was readily available at, or near, the surface: thus material with a high surface/bulk volume ratio, such as peas, minced meats, small vegetable dice, etc., would freeze readily, but steaks of fish or meat would not.

In some cases, however, simply cutting material into small picees was not satisfactory if the moisture content of the material was so low as to limit the amount of relatively free water on the surface. In such an instance the effect was much less pronounced and although the product was cooled, the minimum temperature attainable failed to ensure complete freezing of the water fraction. However, where a sufficiently high quality of product can be obtained by evaporative freezing, this method should be adopted, since the elimination of

a pre-freezing stage gives a significant saving in costs. In addition the rapid removal of 20 per cent of the water reduces the weight to be removed by freeze-drying and generally shortens the drying cycle.

Ice Formation

The true freezing-point of a foodstuff is rather difficult to establish accurately, but the most practical experimental technique appears to be to measure the electrical conductivity of the tissue during cooling. As the temperature is lowered, ice crystals begin to separate out, so concentrating the salt and sugar solutions, and eventually a eutectic point is reached when the mass of the remaining solution freezes. Since ice has a much higher electrical resistance than liquid water, this point can be estimated with a probe inserted in the foodstuff and connected to a megohmmeter. A figure of 100 megohms with a probe having electrodes $\frac{3}{8}$ in. apart was found to give satisfactory results, indicating the temperature to which the food must be reduced to produce true freezing. It is quite possible to get an appreciable degree of super-cooling if the freezing cycle is rapid, so that on a warming-up cycle the electrical characteristics are found to change at a different temperature, the melting-point. For example, apple slices when frozen by evaporation in the cabinet dropped to $-26°C$ before the electrical resistance indicated freezing, but subsequent raising of the temperature to $-10°C$ gave no noticeable difference in the reading.

No two foodstuffs have exactly the same freezing characteristics and it is essential to know the true freezing point before effective freeze-drying can be undertaken. (As well as the apple mentioned above, carrot and banana were two other materials found to require a temperature of about $-30°C$ for freezing.)

The rate of freezing has a marked effect on the size of the ice crystals formed, their location in the foodstuff, and the drying and reconstitution characteristics. With rapid freezing, e.g., by evaporation in the cabinet, where it is possible to pass through the stage of thermal arrest with pieces of meat $\frac{3}{4}$ in. thick in about 20 min, the ice crystals are extremely small and mostly inside the cells (intra-cellular). As the speed of freezing is reduced the size of ice crystal increases, becoming more and more extra-cellular in location, and eventually quite severe mechanical damage can occur to the cell structure. As this ice is sublimed away pores of large diameter remain in the dry tissue, offering a reduced resistance to the escape of vapour from the ice surface as compared with material that has been frozen rapidly; and, on rehydration, readily admitting water to the centre of the piece. Unfortunately the structural changes accompanying slow freezing adversely affect the appearance and texture of the product, and with fibrous materials such as meat and fish the water is very loosely held and can easily be squeezed out. It is therefore desirable, at any rate with some foodstuffs, to use an intermediate rate of freezing, such as that obtained by blast-freezing, to obtain the optimum crystal size. There is, in fact, little precise knowledge of what constitutes optimum crystal size, or of the conditions which produce it; this is a matter on which further research is required.

Physics of Drying

When the material is placed in an evacuated cabinet sublimation will continue until the temperature of the ice is reduced to a value which is related to the partial pressure of water vapour in the system. At this point sublimation would cease unless heat were applied and, generally speaking, with previously developed

techniques the rate of heat application was the factor which controlled the rate of drying.

The so-called classical freeze-drying method was to load the material in trays on the shelves of a cabinet which was connected to a suitable vacuum system. Heating fluid was then circulated through the shelves, and the heat transfer was thus largely by conduction to the lower side of the tray, and by radiation from the shelf above to the upper surface. Even if the temperature of the heating medium was comparatively high (possibly 100°C maximum but frequently much less), the amount of radiant heat transferred in this way was rather small, and in addition there was a tendency for large pieces of food to curl away from the supporting tray, thus giving only point contact and consequently little conducted heat. A further consideration was that even with a substantially flat surface on the foodstuff it could not be assumed that there was a high percentage of contact, since vapour was escaping from the lower surface at a high rate. This meant that the material was virtually cushioned on a layer of high velocity water vapour with only point contact here and there, with consequent reduction in direct conduction of heat into the tissue.

In addition to this inefficiency of heat transfer, another factor is evident, as has been verified experimentally by interrupting the drying cycle of 15 mm thick cod steaks after 2 hr drying in such a system, when it was found that the thickness of the dry layer on the underside was only 4 mm, while that on the upper side was 5½ mm. There are two possible explanations for this since both heat and mass transfer are involved, but it is reasonable to suppose that the heat input to the lower face was as great, if not greater, than on the upper surface since the radiant heat flux will be substantially the same on each surface, and in addition there is some conduction to the lower surface. It is therefore most likely that a greater resistance to the escape of the high velocity vapour stream had limited the rate of drying from the bottom surface, this resistance being dependent on the weight of the fish.

The physical problem, simply stated, was to improve the rate of heat transfer to the product during drying, whilst still maintaining an adequate vapour escape path. The work in Aberdeen was based on a type of heat application technique called the 'contact-plate system' (using the Atlas VCD plant). Essentially this required the application of mechanical pressure to the foodstuff between two horizontal heated plates, so permitting the conduction of heat to both top and bottom surfaces simultaneously. With such an arrangement much higher rates of heat transfer were possible, not only because of the presence of the second conducting surface, but also because the application of pressure to the product increases the heat transfer coefficient and the contacting area. Unfortunately, however, with such a system the situation which exists on the underside of pieces of food in a simple vacuum shelf-dryer is also encountered to a much more serious extent and on both surfaces of the foodstuff. The resistance to escape of water vapour is much higher, and the stifling of this flow can cause a local rise in vapour pressure which may lead to thawing of the ice in the material. With such equipment it was thus impossible to apply the full potential rate of heat transfer in the early stages of a drying run, and normally the plate pressure had to be limited, while the temperature of the heating plates could not be raised very quickly. In the earlier stages of development of the AFD technique the method was to maintain the plate temperature at its lowest level for about 2 hr until a layer of dry tissue was formed, with very light contact pressure on

the product. The dry tissue then acted as a vapour escape path, and the plate pressure and temperature could be raised with little chance of thawing the ice in the material.[3] In such a system, then, it was the vapour flow which was the limiting factor, in contrast to the situation in classical freeze-drying described above, where heat input is the limiting factor.

The point in the drying cycle when most benefit could be obtained from a high heat transfer rate was at the beginning, when the ice was at or near the surface, and it was desirable to achieve this while still permitting ready escape of the water vapour. The method evolved in Aberdeen, which led to the realization of the Accelerated Freeze-drying process, was to interpose between the foodstuff and the heating surface a sheet of expanded aluminium.[1] [2] The mesh of the expanded metal conducted heat directly to the foodstuff at numerous points, while the vapour escaped fairly readily through the channels formed by the mesh; and pressures of up to 8 p.s.i. could be applied by the heating plates to rigid pre-frozen materials, though lower pressures had to be used with vegetables and fruit.

When this technique was used, an extremely high rate of heat transfer was possible in the initial stages since there was little resistance to the escape of water vapour. The temperature of the ice in the product rose until the related vapour pressure was in an equilibrium condition with the cabinet pressure, and thereafter all the heat transferred to the ice was absorbed as latent heat of sublimation. Under these conditions the pumping capacity of the evacuating system caused some problems, since the sublimation rate could rise as high as 0·45 lb/hr/sq. ft of material surface (both top and bottom surfaces being effective). The actual pumping mechanism does not theoretically affect the freeze-drying operation, but it must be of a suitable size to handle the evolved vapour in the peak quantity expected, at the designed operating pressure.

At any time later than this initial stage the situation is complicated by the presence of the enveloping layer of dried material. In animal tissues, in general, the cells form elongated clusters or fibres, and in such tissues the resistance offered to the escape of vapour through the dried layer is not sufficiently high to cause thawing unless the escape path is excessively long (e.g., as a result of incorrect cutting-up techniques). However, in plant materials the cells are not normally arranged in such a way as to form, during drying, easy channels for the escape of vapour; thus the resistance of the dry layer may be much higher, and the application of too much heat can cause thawing, with undesirable results. This problem can be tackled in two ways—either the heat input must be limited, thereby prolonging the drying cycle, or alternatively a smaller piece must be used. With apple dice, for example, a maximum thickness of $\frac{5}{16}$ in. was established for reasonable drying times. Theoretical considerations would suggest another method of dealing with this situation—by improving the cabinet vacuum, and therefore the pressure gradient across the porous barrier. In the vapour flow conditions probably existing under these conditions the mathematical formulae indicate that the rate of flow is proportional to the square of the pressure differential, but on the other hand Harper and Tappel's[4] curves show no significant improvement in vapour flow with such variations, say from 1 mm to 0·1 mm, as might reasonably be possible in practice.

(As a corollary to this it will be appreciated that if the maximum cabinet pressure has been decided by the melting-point temperature (and so vapour

pressure) of the ice in a particular foodstuff, then as drying proceeds and the dry layer introduces a progressively increasing resistance to vapour flow, the absolute pressure in the cabinet should be reduced to maintain the same vapour pressure at the ice surface, so maintaining the ice temperature at the established level. So if 1 mm Hg is considered to be a suitable starting pressure it may be necessary to lower the value to 0·5 mm Hg or less in the final stages. Fortunately, such a characteristic is inherent in a batch system, whether the evacuating system consists of steam jets or a refrigerated condenser/rotary pump arrangement. With the former designed to pump a predetermined large quantity of water vapour at 1 mm Hg, the pressure will automatically drop when the rate of sublimation falls off in later stages of the cycle; similarly, where a refrigerated condenser is employed, the refrigerating compressor has to be designed to deal with the maximum rate, and as drying progresses the condenser temperature will drop, so lowering the water vapour pressure in the system.)

In other cases, the limiting factor is not the passage of water vapour through the porous material, but the transmission of heat through it. This has been proved quite conclusively by the fact that it is possible in such cases to apply sufficient heat to scorch the surface of the foodstuff while the ice temperature in the centre remains at sub-freezing temperatures. Prevention of this requires limitation of the heating plate temperature, with consequent reduction in the speed of the process. The only way to improve the temperature gradient into the food is to lower the ice temperature, which can be done by lowering the cabinet pressure, but it is doubtful whether this would be economical below a temperature of −30°C. Nevertheless, it is significant that theoretically 28 per cent more heat passes over a temperature gradient of 60°C to −30°C, than over a gradient of 60°C to −10°C. This does not mean that the drying time would be reduced by this amount, since the latent heat of sublimation is greater at the lower temperature, and in addition more specific heat would be absorbed by the vapour passing from the lower temperature ice to the surface. It does, however, suggest that with those foodstuffs where rate of heat transfer is the controlling factor, it might be worth considering the installation of larger capacity evacuating equipment to achieve some reduction in drying time. Time has not permitted a detailed study of this feature, but one load of fillet steak 15 mm thick took 8½ hr to dry at 0·8 mm Hg as compared with about 7 hr at 0·2 mm Hg, the final moisture contents in both cases being about 2 per cent.

As to the maximum permissible temperature for the surface of the dried material, 60°C was generally adopted as an arbitrary limit in the Experimental Factory. This appeared to be quite satisfactory for vegetable tissues, provided the pieces of material were small enough to maintain the ice temperature at the desired level, but with fish and meat products the use of so high a temperature caused some toughennig and impaired the water-retaining power of the tissue, also leading to loss of the natural pink colour of raw meats. The quality became increasingly better as lower surface temperatures were used, but the drying time became proportionately longer, making the process more costly. It is therefore necessary to compromise between product quality and drying time when setting these temperature limits, and for lean meat products it is suggested that 50°C might be a better level. A further deciding factor which must be taken into account is the possibility of the melting of the fat in such foods as pork, bacon, cheese, etc., where the dry layer temperature should never exceed 40°C. Experience has shown that if the fat is rendered during the

process it will coat the dried material and make rehydration a tedious process if, indeed, not totally impossible.

The highest permissible temperature cannot be judged by appearance alone, since changes in flavour and texture may also occur. This was particularly noticeable with fish, which can be raised to 90°C for at least 2 hr during an 8 hr drying cycle with no visible heat damage but with very noticeable toughening and off-flavour. There is also a time effect; the longer the tissue is held at an elevated temperature, the more severe the damage. Again fish provides an example; a double thickness of fish (two 15 mm layers) was dried in 11½ hr (with an intermediate layer of expanded metal) which meant that the surfaces adjacent to the heating plates, and the tissue extending some way in from the surfaces, were maintained at 60°C for several hours. The resulting product looked perfect, but was unacceptable because of its tough and dry texture.

The storage properties of the product depend on a number of factors which are dealt with more fully in Chapter XI, but not the least important is its moisture content. In liquid-phase drying the cycle can be stopped at any time and the final moisture content will be fairly uniform throughout the piece, but with freeze-drying this is not the case. Here, any portion of the product still containing ice has the moisture content of raw material, and if such material is stored in a sealed container there is a grave risk of spoilage of these wet pieces before the water migrates to the dry tissue. Moreover, this in-package desiccation will be from the liquid phase and the product will be characteristic of such material.

It is well known that the removal of water from biological material becomes progressively more difficult as the end of drying is approached. After the 'free' water is removed there remains 'bound' water, and it has been suggested that the mono-molecular layer of water adsorbed on to the pore surface of material such as meat amounts to a moisture content in the region of 5 per cent.[5] This value is too high for long term storage which requires something below 2 per cent. Experience in Aberdeen has indicated that with the AFD process employing fairly high temperatures in the dry tissue (ca 60°C) the secondary drying of this adsorbed moisture appeared to occur at the same time as the main drying was proceeding. When even small areas of ice remained in the centre of a piece of food the temperature of the surface layers was depressed and this could be taken as an accurate indication of the presence of residual ice. It was often found that slabs of meat or fish could feel cool to the touch on removal from the plant and yet have a moisture content of below 5 per cent, so indicating that even while some ice remained secondary drying must also have taken place. All material which was warm on the surface had a moisture content of below 2 per cent, and often below 1 per cent.

No two pieces of meat or fish are exactly alike. There may be irregularities in chemical composition and in physical characteristics, e.g., the nature of fibres, whether the fibres run at right angles to the cut surfaces, distribution and quantity of fat, etc. Other foodstuffs are equally heterogeneous and thus it was not permissible to assume that all pieces would dry at the same rate in any one run. In general the process was monitored by keeping track of the temperatures at different points in the foodstuffs, by means of thermocouples leading to a recorder. This natural heterogeneity of foods, however, suggested that thermocouples might not be the best control sensing device, since it was unlikely that they were placed in the slowest drying pieces in every case. In

practice it was necessary to extend the drying time by 20–30 min after the thermocouples had risen to the heating plate temperature in order to ensure that no wet pieces remained. This might well have been prolonging the cycle unnecessarily, and some saving in operating costs could be obtained by using a device which would sense the presence of ice by some other means, and which would give an overall estimate of conditions instead of the spot check which is, in fact, all the thermocouples accomplished.

On the other hand, it was found that after a number of runs had been carried out on any one commodity, the length of the drying cycle for standard processing conditions could be predicted with fair accuracy. It might be possible, therefore, to operate from prior knowledge alone, using thermocouples as a check or even dispensing with them altogether.

It is perhaps worth mentioning that experience with a variety of processes suggested that it might not be necessary to complete the freeze-drying cycle with all commodities. As already indicated adequate quality may in some cases be obtained by other, less expensive, procedures and it may be desirable in some cases, for various reasons, to combine freeze-drying with other methods; for example colour retention of garnishes for soup mixes may be improved if the material is freeze-dried down to 20 per cent and subsequently air-dried instead of being air-dried throughout. Again the woolly texture of some freeze-dried chicken may be improved by removing the final water by vacuum-drying from the liquid phase. This results in a relatively tougher centre to the pieces which gives a sensation of bite and is more acceptable to some people. Similarly with cabbage it was shown that air-drying tended to make the midrib of the leaves tough and chewy, while freeze-drying made the leaves mushy but gave a better texture in the midrib. Partial air-drying followed by freeze-drying gave the best of both methods and the leaf and midrib had optimum texture properties. These examples indicate some possible combinations of methods and it is, probable that some other products would react favourably to similar techniques.

CHAPTER III

Development of specifications

Preliminary Tests

The original freeze-drying tests were carried out on the Atlas VCD (Vacuum Contact-Dehydration) Plant[1] [2] installed in Aberdeen in 1950. This was designed to dry 1 ton of fish fillets at 10–15 mm Hg, and under these conditions drying took place from the liquid phase. The steam-jet evacuating equipment consisted of two stages, with the spray condenser maintained at the operating pressure by water ejectors. Between the cabinet and the condenser was a single stage steam augmentor. Large heavy non-return valves were fitted between the water ejectors and the spray condenser, and it was found that these limited the attainable vacuum in the condenser and consequently, therefore, in the cabinet as well. When they were removed it was possible to operate the cabinet at an absolute pressure of 1–2 mm Hg, and provided the load of foodstuff was limited, it was possible to freeze-dry under these conditions.[6] Many tests were carried out with a variety of products, but since there was only limited instrumentation available, it was impossible to make systematic studies.

A small pilot plant having a single tray 15½ in. × 29½ in. and using the vacuum contact principle was in operation during this period, but the method of construction and the inability to measure process variables accurately limited its usefulness for studying the freeze-drying requirements. In addition it proved to be impossible to relate results obtained from this pilot plant to conditions in the Atlas VCD Plant, and it was felt that the greatest cause of this was probably the scaling up effect arising mainly from the very different vapour-escape path-lengths from the centre of the tray. Also the quantity of material which could be produced per batch in the pilot plant was inadequate, since it was desirable to carry out storage tests and obtain taste-panel results as well as routine laboratory analyses. For these reasons it was decided to construct a fully instrumented pilot plant having a single pair of heated plates 4 ft (the smaller dimension of the Atlas trays) square.

Experimental Vacuum Equipment (EVE)

This plant (Fig. 1) was manufactured in the Factory workshop, and consisted of a cabinet with full size doors at front and back to give maximum accessibility. The top heating plate was fixed to the roof of the cabinet, while the lower plate was mounted on four screw jacks, bolted to the floor of the cabinet and driven by a single shaft passing through a vacuum seal in the cabinet wall. The bottom plate could then be raised or lowered as required without breaking the vacuum; and located between the jack heads and the bottom plate were electrical strain-gauge load cells measuring the applied thrust. This was indicated on a gauge outside the cabinet, and an accurate assessment of applied plate pressure could then be obtained.

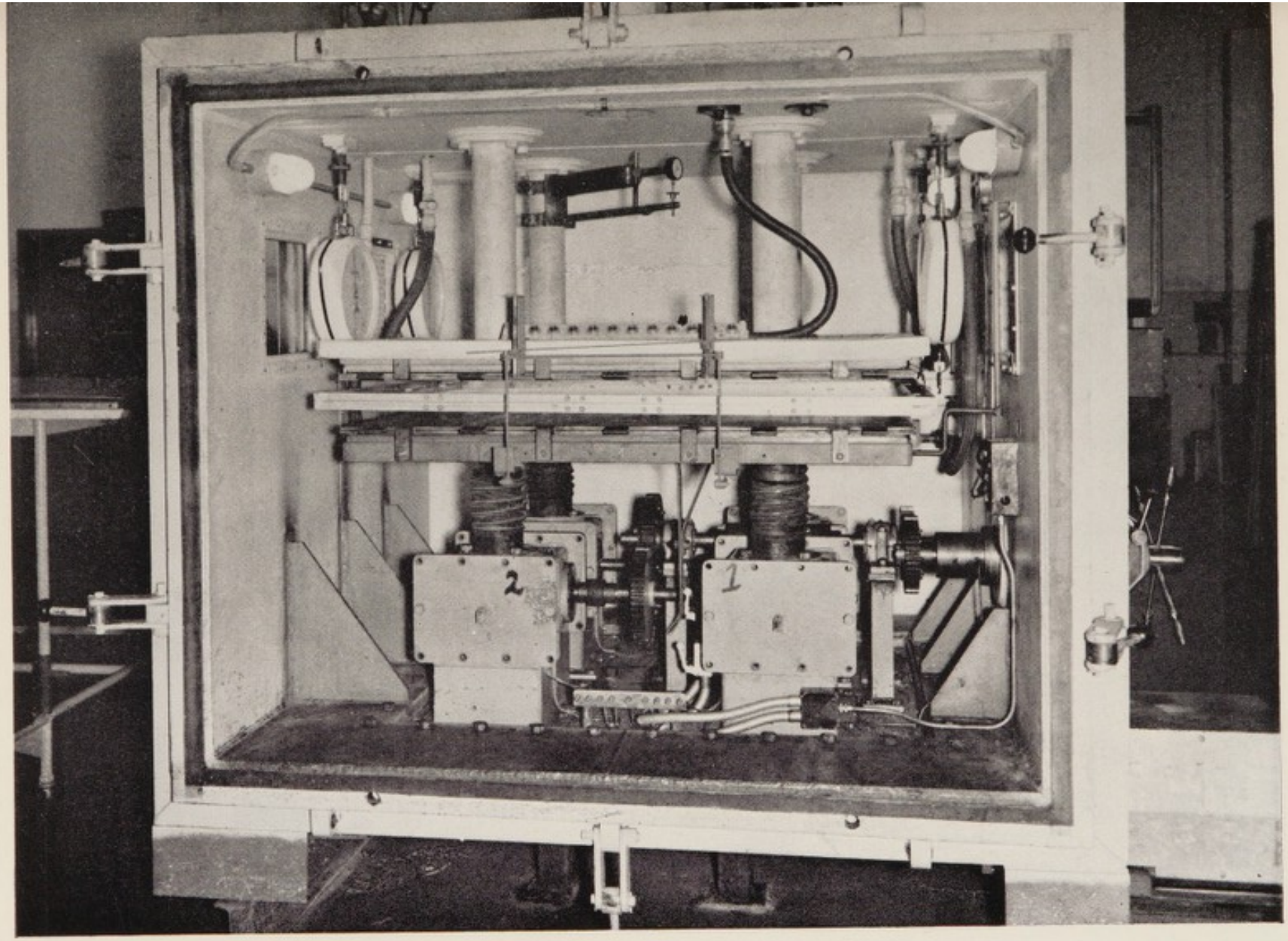
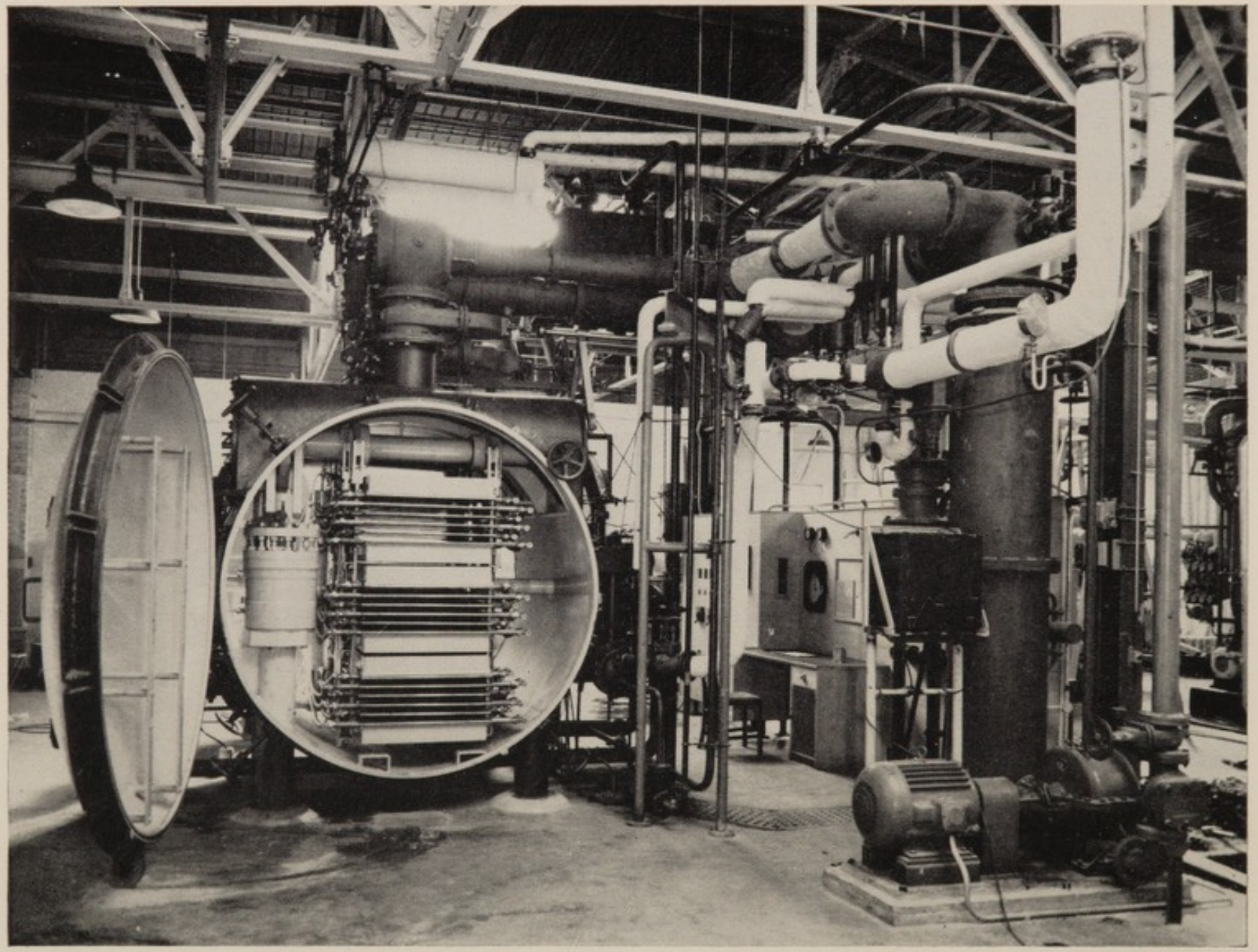


FIG. 1. *EVE pilot plant fully instrumented for investigational studies of the AFD process*



The heating plates were manufactured from stainless steel with an internal multi-channel labyrinth, all-welded, construction. The location lugs on both plates were fixed at right angles to the heating surface, and the plates were held in position by bolts passing through holes in these lugs into the plate support frame. This proved to be unsatisfactory since thermal expansion caused the plates to buckle, and it was necessary to permit some freedom of movement to overcome this difficulty. Attachment lugs in the same plane as the plate, with slotted holes, would have been preferable.

The plates were heated by circulating water by a pump through a steam-heated calorifier and a changeover valve. The rate of circulation was approximately 28 gal/min and the water temperature could be raised from 10°C to 140°C in about 20 min. Also included in the circuit were an expansion tank and a water cooled indirect heat exchanger which by means of two 3-way valves could be used to cool the circulating water. The entire system was suitable for operation at 45 p.s.i.g. in order to permit the pressurization necessary for the use of temperatures as high as 140°C. A water flow-rate meter was fitted with an orifice plate in the connecting pipe-work to indicate the rate of circulation of the heating water.

During a drying run the bottom plate was lowered until the drying tray was held by support lugs on a weighing frame suspended from four spring balances. A window was fitted in the cabinet in front of each spring balance, permitting readings to be obtained without interrupting the vacuum, and consequently causing minimum alteration to the shape of the drying curve (i.e. sublimation rate). Experience has led to the belief that this is probably the most important facility in such an experimental plant. A plate-spacing indicator was also fitted having a magnified linear scale, mounted so that it was visible through one of the viewing ports.

The absolute pressure in the system was measured with an Alphatron cold ionization gauge. This instrument was chosen after extensive enquiries had been made into alternative types suitable for factory scale operations with the freeze-drying of foods. The major factors which are peculiar to this type of process, and which may possibly lead to incorrect measurement of absolute pressure, are the large proportion of water vapour in a superheated state, and the presence of volatiles evolved from the foodstuff. Theoretically, the simplest and most fundamental type of gauge is the McLeod, but with water vapour present this invariably reads low. For operation under laboratory conditions, this can be overcome by utilizing an efficient vapour trap, but this method was thought to be unsuitable for semi-skilled factory employees. A Pirani type gauge uses a hot wire as the indicating element, this being particularly susceptible to contamination by organic volatiles, with consequent loss of calibration. In addition, one must be extremely careful to ensure that the gauge is not left switched on when the vacuum is broken, and normally the calibrations for air and water-vapour are quite different. This implies that an accurate assessment has to be made of the proportions of air and water-vapour present before a reliable estimate of absolute pressure can be derived. The Alphatron gauge appeared to be the most satisfactory for this type of plant since it does not suffer from the disadvantages listed above and in addition is robust, covers the range 1–10,000 microns on a linear scale (with range selector switch), gives a continuous reading of pressure and, since it employs radium as the source of alpha particles, has a long term stability.

Considerable difficulty was encountered in obtaining load cells which were not affected by temperature and pressure changes. The original cells employed strain gauge elements manufactured from circular sectioned wire, and these were extremely susceptible to changes of pressure in the cabinet. The elements were formed by bonding the wire to a paper backing sheet, and it was found that this technique resulted in small bubbles of air being trapped between the wire and the paper. Subsequent reduction of the air pressure round the element caused the occluded air bubbles to expand, straining the wire and altering the calibration of the gauge. In addition the orientation of the temperature-compensating element was unsatisfactory, and when the temperature of the heating plates was raised, the resulting temperature gradient across the cell introduced further errors. Eventually cells were obtained, made from etched foil elements and having a modified temperature compensating system, which gave good results. Each cell had a rated loading of 2 tons giving an overall load on the 16 sq. ft tray area of 8 tons. The indicator was calibrated from 0 to 4,500 lb by 25 lb divisions, and provision was made for reading each cell individually in succession, or alternatively of indicating the mean value.

For reasons of economy it was decided initially not to purchase separate evacuating equipment for this plant, but to connect it to the Atlas VCD plant mentioned above. A considerable number of runs were carried out in this way, but it soon proved to be an extremely inconvenient arrangement, since it was undesirable to operate EVE while semi-production runs were carried out in the parent plant and some conflict of requirements naturally occurred. Consequently it was decided to install separate evacuating equipment to overcome this difficulty.

Evacuating Equipment

It was intended that this plant would be reserved for experimental work on the physical aspects of the AFD process, and at the time it was expected that this would entail scaling up the equipment, so that it was decided that the evacuating equipment should be capable of removing the water vapour sublimed from *eight* trays measuring 4 ft × 4 ft, the maximum number which could be fitted conveniently into EVE. Previous experiments had indicated that cod steaks gave the highest sublimation rate of all products, and the specification of capacity was based on this commodity, requiring a pumping rate of 100 lb/hr at 1 mm Hg total absolute pressure.

Experience had already been obtained with the operation of steam ejectors, and it appeared that the claims made for this type of equipment as regards the low maintenance requirements were correct. However, enquiries indicated that there was a dearth of information on the design and operation of refrigerated condenser and rotary pump evacuating systems, and accordingly it was felt that installation of this type of plant would be rewarding. Various firms were invited to offer quotations for suitable equipment in each category and, as expected, those offering rotary pump systems estimated costs which varied widely, but generally were in excess of the figures given for steam ejectors. The lowest price quoted for a rotary pump system was £6,500 as compared with £3,800 for a four-stage steam-ejector layout. These two figures cannot be compared directly, since the rotary pump system was self-contained whereas the steam ejectors required a boiler installation to supply steam at about 2,500 lb/hr at 120 p.s.i.g. The cost of such an installation would partially close the gap

between the two systems, and since the apparent running cost of the rotary pump layout was only slightly less than the steam ejectors, it did not appear that either should be preferred on financial grounds. The deciding factor in the final selection was therefore the apparent ignorance of design criteria for this size of refrigerated condenser when operating at about 1 mm Hg. It was intended primarily to study the physical problems of water removal from the foodstuffs, and not the capabilities of the evacuating equipment. Consequently, it was felt that the installation of a rotary pump system might possibly result in prolonged delays, due to the need to carry out equipment modifications, and so imperilling the programme of processing experiments. The 4-stage steam-ejector system was therefore ordered and installed.

The layout consisted of two 2-stage steam augmentors in parallel designed to pump 67 lb/hr and 33 lb/hr of water vapour respectively at an inlet absolute pressure of 1 mm Hg. These discharged into the main condenser, cooled by sea water at 55°F maximum, and operating at about 20 mm Hg. The non-condensible gases were removed from the condenser by two 2-stage steam ejectors again in parallel, with inter- and after-condensers. One of the ejector sets was designed to act as a booster during the 4 min evacuating time to 1 mm Hg, while the other was intended to maintain the operating pressure after this point had been reached. With this arrangement some economy of operating steam could have been effected if the cabinet had been fitted with 8 tray loads of material, but in actual fact, since all the development work was carried out with only one tray load, only the one-third capacity augmentors were employed.

In general good results were obtained with this equipment, but some fluctuation in cabinet operating pressure occurred due to the build-up of ice in the primary augmentor combining-tube. The symptoms of this phenomenon were that after the drying cycle had been proceeding for some time, the absolute pressure in the cabinet would slowly rise by as much as 0·3 mm Hg. Suddenly a large piece of ice would fall into the cabinet, and the pressure would then revert to its original level. A mirror and lamp were mounted inside the primary augmentor suction port, and it was then possible to study the course of this build-up of ice through the cabinet windows. The high velocity steam discharging from the nozzles impinged on the sides of the combining tube walls, and since the vapour pressure at this point was well below the critical value this soon cooled the mass of metal to below freezing point. Ice was then deposited on both the outside and inside of the tube. On the outside this took the form of a uniform layer about $\frac{1}{16}$ in. thick, but on the inside it grew in the form of horns extending from the combining tube wall towards the three nozzles. These continued to grow until they could no longer support their own weight, at which time they became detached and fell into the cabinet. The solution to this problem was to wrap the outer surface of the combining tube with closely coiled copper tube, insulated from the atmosphere and heated with live steam.

As already indicated the rated capacity of the augmentors was greatly in excess of that encountered when drying a single tray load and consequently there was no reason to assume that the equipment was incapable of achieving the design condition. Some time later, however, when difficulty was experienced with the Mark I plant (see Chapter IV) it was thought desirable to check this feature on EVE. It was then shown that the evacuating equipment on this plant, although supplied by a different manufacturer, was also incapable of removing water vapour at the specified rate and pressure. Subsequent tests

and modifications carried out by the manufacturers resulted in a final capacity of 85 per cent of the rated value.

Process Control

The basic control element in the process is the thermocouple indicating the product temperatures both at the centre and surface. As indicated in the previous chapter the desirable centre temperature is decided by the eutectic point in each food, and the surface must not exceed a safe maximum value to prevent thermal damage to the tissues. Some difficulty was encountered originally with uncertainty of the true temperature level in the centre of the foodstuff, due to the fact that the wire used was of too heavy a gauge, and also because the method of insertion was to drill a hole and push the wire home. The heavy gauge wire conducted heat into the junction causing its temperature to rise, and in addition, since the drill-hole was a free surface, some drying of the tissue round the thermocouple occurred. These features, together with the distinct possibility that the junction might not be pushed right to the bottom of the drilled hole, resulted in the junction eventually being quite remote from the nearest ice, and consequently giving a false impression of the temperature of the undried material.

With material which is to be frozen by evaporation no difficulty arises with the insertion of the wire, but with pre-frozen food the thermocouple must be inserted into a pre-formed hole, either by freezing in a skewer of suitable dimensions which is afterwards withdrawn, or alternatively by drilling a hole. Whereas the latter technique was unsuitable with a heavy gauge wire as mentioned above, the adoption of a 33 S.W.G. copper constantan thermocouple wire with glass fibre insulation gave good results. This wire required to be stiffened by dipping the end 3 in. into a cold setting resin adhesive so that it could be easily inserted in the tissues. In addition, since it was mechanically weak, the overall length was limited to about 9 in. with a plug and socket assembly completing the circuit to the cold junction outside the cabinet, through a P.V.C. insulated copper constantan cable ($14 \times 0{\cdot}0076$ in.).

Measurement of the surface temperature of the product introduced different problems, and no entirely satisfactory solution has yet been found. Originally, attempts were made to insert the thermocouple at an angle into the block so that the junction was immediately below the surface. Not only was this extremely difficult to locate correctly, but in addition the temperature indicated was affected by the relative proximity to one of the points of the adjacent expanded-metal sheet. If it happened to be immediately below one of the points a higher temperature would be indicated than would be the case if it happened to be under an aperture in the sheet. Various alternative methods were suggested, the method eventually adopted being to solder a standard 33 S.W.G. thermocouple to a piece of brass gauze. It was soon obvious that if this was applied to the surface of the product with the thermocouple junction on the product side of the gauze, good agreement could be obtained between a number of points. However, it is reasonable to suppose that the indicated temperature was higher than the mean temperature of the product surface, though by how much is uncertain. In any event, from the point of view of process control the actual level of temperature measurement is not important, so long as it is reproducible from run to run, but whatever technique is used it is desirable that the measuring element should cause the least possible

interference with the natural heat and mass transfer characteristics at the point of measurement.

On this plant, twelve thermocouples were provided within the cabinet, one being utilized for an icicle, and the remaining eleven being distributed throughout the foodstuff, usually with four on the surface and the remainder in the centre of the product. The readings, along with another two thermocouples in the heating water circulation system at inlet and outlet to the plates, were recorded on a 16-point electronic recorder having a symbol- and colour-code of marking, a chart speed of 3 in./hr and a temperature range from −40°C to 140°C.

Preparation of Prototpye Specifications

When, in 1959, Armour and Company offered to provide the Research Establishment with a commercial prototype AFD drier, it became necessary to write a Specification for its purchase. The specification of what became known as the Mark I AFD plant was worked out from the experience on EVE.

One of the major problems that had been encountered when freeze-drying in the Atlas VCD plant was the difficulty of transferring pre-frozen food to the drying trays and establishing the vacuum before any thawing of the ice in the surface layers occurred. The original trays were 8 ft by 4 ft and it was not possible to load the trays prior to freezing, i.e., when pre-freezing was required. Consequently it was necessary to freeze the material on smaller trays, transfer to the large drying trays, and then load the cabinet and pull the vacuum. Under these circumstances a considerable amount of thawing occurred and an inferior product was obtained. The adoption of a tray size 4 ft by 2 ft, which would be inserted into a blast-freezer, permitted the freezing of the product on the drying trays, and thus speeded up the transfer of the foodstuff into the drier. It was therefore essential that the prototype machine should have a tray dimension which would suit the design of an ancillary blast-freezer. A/S Atlas were by now producing a Vacuum Contact Dehydration cabinet with heating plate dimensions 750 mm by 2,000 mm, and it was decided to specify this size in the prototype, although, since it was felt that expanded metal inserts of this size would be extremely liable to be damaged, it was arranged that the actual tray size would be approximately 750 mm × 1,000 mm (i.e., 2 trays on each plate).

The VCD plant required mechanical loading gear, and again bearing in mind the necessity to load the cabinet as quickly as possible, it was considered that such a device should not be employed with the AFD prototype. This meant that the top tray should be no more than 6 ft from the ground, and it was therefore decided to specify 15 loading stations (i.e., 30 tray loads) with the stack divided up into three banks, as in the original Atlas plant, to limit the required overall travel of the plate movement mechanism.

A large number of runs was carried out on EVE to establish the required range of all the process variables on a wide variety of products. The basic knowledge required before the specification could be finalized was:

Maximum plate pressure and optimum expanded-metal dimensions.
Maximum water temperature in the heating plates, the maximum rate of change required, and the permissible temperature drop across the plates.
Range of absolute pressures required in the cabinet.
Maximum sublimation rate.
Maximum pump-down time to operating pressure.
Instrumentation required.

Applied Plate Pressure

It was soon obvious that leafy vegetables, fruit and meat minces could not withstand a high plate pressure, either because the resulting compaction of the bed caused excessive stifling of the vapour flow, or because the structure of the tissues was too weak mechanically. Furthermore, since it was advisable to increase the pressure gradually from zero, it followed that it was necessary to establish only the maximum useful pressures for the range of foods considered.

It seemed possible that this feature would require to be studied in conjunction with the temperature of the heating plates, and a series of experiments was carried out using pre-frozen cod steaks. The technique evolved was to raise the heating water temperature to its maximum as soon as possible, maintaining the plate pressure at a low value, since if this were increased too rapidly some thawing always occurred on the surface of the product. The maximum plate pressure was thus not applied until after about 2 hr, and the effect was to drive the points of the expanded metal sheet into the surface of the dry layer, so reducing the heat path from the metal mesh to the internal ice front, and further increasing the heat flux by increasing the contact area.

Expanded metal was obtainable in a fairly large range of mesh formations and dimensions, but in order to achieve significant penetration of the dry tissue with a practical applied plate pressure, it was necessary to select those forms which had points on one side of the diamond configuration. The other side should preferably have been flat to give good contact with the metal tray and lid but this form was not commercially available and the cost of producing small test pieces for experimental purposes was prohibitive. The dimension of the aperture in the mesh was important, since it had to be small enough to ensure a high proportion of contact area with the foodstuff and yet large enough to permit easy escape of the water vapour. In addition the overall thickness of the mesh, as measured between two flat sheets placed on either side, had to be adequate to allow easy escape of the evolved water vapour to the edge of the heating plate. The metal used to form the mesh did not appear to affect the rate of drying. Both stainless steel and aluminium were tried, and although these materials have very different thermal conductivities, the main barrier to heat transmission was the dry layer of tissues on the product surface, and both metals were equally effective.

A number of different mesh forms were tested, and eventually it was decided to standardize on an aperture size $\frac{7}{8}$ in. $\times$ $\frac{5}{16}$ in. with a strand $\frac{1}{16}$ in. wide formed from 20 G. aluminium sheet, and having an overall thickness of about 2·5 mm. Aluminium was chosen because it was only one-third the price of stainless steel, it was readily available and, although it was more easily damaged by careless handling, it could be obtained in perfectly flat sheets whereas the samples of stainless steel which were tried were in most cases badly buckled.

Using this mesh it was then shown to be advantageous to employ a final maximum applied plate pressure on pre-frozen blocks of some materials, e.g., meat and fish, in the region of 8 lb/in.[2] This value gave a penetration into the dry layer of about 1 mm on both top and bottom surfaces, giving a significant reduction in heat path to the ice front. As the expanded metal was driven into the product the available cross-sectional area, through which water vapour could escape to the edge of the tray, was progressively reduced, and it is probable that the use of higher plate pressures with this particular mesh would have resulted in stifling of the vapour flow again.

Time did not permit a detailed study of optimum expanded-metal meshes or plate pressures for all foodstuffs, but where expanded metal was used the data reported in these chapters all refer to the mesh size given above. The actual plate pressures used on each type of product (which are often modified by the particle size, depth of bed, etc.) are given in Chapter V.

Temperature of Heating Water

It was clear from the outset that plate temperatures in excess of 100°C could be used to advantage, and some thought was given to the possibility of employing some other fluid, apart from water, as the heating medium. Oils or various chemical solutions can be heated to these temperatures without having to use a pressurized system, but generally their specific heat is lower, and consequently higher fluid velocities would have been necessary to maintain a comparably small temperature drop across the heating plate. On the other hand low pressure steam could give the range of temperatures required with no temperature differential, but its use in flat horizontal plates might have entailed some problems in condensate removal, with consequent relatively cold pockets within the plates. Some considerable experience had been gained with a pressurized water system on the Atlas VCD plant and, since the aim was the production of a prototype to prove the process rather than the development of completely novel equipment, it was decided to retain this method of heating.

As has already been explained, the control of the process was basically control of the surface temperature of the product at the safe maximum (usually about 60°C). The faster this condition could be reached at the beginning of the cycle the higher would be the initial sublimation rate and therefore the shorter the process; and it had been shown that, unlike a comparable air-drying process, an increase of rate at this stage would have no effect on the later sublimation rates, provided that no thawing occurred. Again most of the work was carried out with pre-frozen raw cod steaks, and it was shown that the heating water could be raised to 140°C with advantage. This was therefore the maximum temperature specified although subsequently it transpired that with most materials the heat input had to be stopped as soon as this level was reached, whereas a maximum of 120°C gave better control and very little change in overall drying time.

In order to take full advantage of these higher drying temperatures it was reckoned advisable to design for a high initial rate of heating, the limitations being thermal shock to the system and the high rate of energy input (in this case steam) required. The specified rate of 15–140°C in not more than 14 min was a compromise taking into account these various considerations. Since Mark I was to be in the nature of an experimental unit, it was desirable to permit rapid cooling of the heating plates in the event of unpredictably high rates of temperature rise in the product, and the rate of cooling specified (100°C to 60°C in not more than 15 min) appeared a reasonable value. In actual fact it was never necessary to utilize this feature.

The permissible temperature differential across the plates was a difficult value to specify, since it appeared that it was possible to get bigger variation in drying rate due to variations in the pieces of product being dried, than was likely to occur due to reasonable temperature differences. However, it was decided to call for a water circulation rate which would theoretically result in a drop of 1°C across the heating plates at the maximum rate of sublimation. In

addition a changeover valve was requested to reverse the direction of the water flow, but it has since been found that either the changeover valve could have been omitted with the same high flow rate, or alternatively the allowable temperature drop could have been increased to about 5°C.

The only other consideration which could have been specified in connection with the heating system was associated with the desirability of loading the cabinet with its plates as cool as possible. For shift operation it would have been essential that there should be minimum delay between unloading one batch of dry material and inserting the next charge, so that the plates should drop from the final drying temperature (60°C) to be as near as possible the cooling water temperature (say 10°C) in not more than 20 min. This was not in fact specified for the Mark I, but was attainable.

Absolute Pressure in the Cabinet

As explained earlier the ice temperature within the product was decided by the partial pressure of water vapour in the system, and the resistance offered by the expanded metal sheets and dry tissue to the escape of water vapour. The operating cost for an evacuating system is increased quite considerably as the absolute pressure is lowered, and consequently, from this point of view, it appeared desirable to design for the highest permissible value. A pressure of 1·5 mm Hg would maintain the ice temperature in meat and fish products below −10°C (eutectic temperature), but was not low enough for some vegetables and fruit. However, the sublimation rate for fish was higher than for other products, and the steam ejector system was thus able to produce lower absolute pressures with the same tray load of these other products.

A pressure of 1 mm Hg at the maximum sublimation rate was therefore specified in order to err on the safe side, and this proved to be satisfactory for most materials, although with some fruits a lower pressure would have permitted higher heat input rates, and consequently faster drying times.

Sublimation Rates

During each experimental run carried out in EVE the product was weighed at frequent intervals, and a drying curve was plotted. The maximum rate of sublimation occurred near the beginning of the drying cycle, and theoretically it was desirable to obtain as many weight readings as possible during the first hour to obtain an accurate plot of the drying curve over this period. However, the weighing procedure took about 3 min, and it was necessary, as already indicated, to suspend the tray from the scales away from the heating plates, so reducing the heat input for that time. This altered the shape of the drying curve, and introduced a greater error than was to be expected due to the necessity of estimating the true shape from a limited number of points. It was therefore common practice to weigh the tray load hourly from the start and every quarter of an hour towards the end, although in some runs three readings were taken in the first hour to check the shape of the curve at that most significant period of drying.

The gradient of the drying curve was then measured, and from this the maximum sublimation rate was obtained. Originally this figure was referred to the actual tray area, but since with pre-frozen blocks it is affected almost exclusively by the larger surfaces of the foodstuff (and practically not at all by the thickness of the piece) it was later decided to relate the sublimation rate

as deduced above to these surface areas of the product on the tray. The proportion of tray area covered by discrete blocks of material depended on the shape of the pieces. With meat slices formed in a rectangular mould the coverage could be 100 per cent, whereas with cod steaks an average figure was about 60 per cent.

With those materials which were frozen *in situ* by evaporation the initial vapour load was extremely high, but since the rate of evaporation was progressively reduced as the temperature of the product dropped, eventually the design condition was reached.

It was not possible to establish the peak sublimation rates for all the main groups of foodstuffs, but in the experiments carried out different materials could be broadly characterized as shown in Table I.

TABLE I

Peak Sublimation Rates

PEAK SUBLIMATION RATE	PRODUCTS
0·3 to 0·5 lb/hr/sq. ft of tray covered	Halved or uncut fruit, apple rings, potato chips, carrot slices, meat slices (cooked or raw), fish flakes (cooked), beans (broad and green)
0·5 to 0·7 lb/hr/sq. ft of tray covered	Diced carrot or apple, peas, leaf celery, minced meat, whole egg (slabs)
0·7 to 0·9 lb/hr/sq. ft of tray covered	Scampi, raw cod steaks, cabbage (scalded)

These should be taken as a rough guide only, since it was not possible to carry out sufficient tests on all these products to establish whether the rates quoted are in fact the highest attainable. In addition, modification of the structure of the raw material was found to change the relevant rate, e.g., slicing of the skin on beans instead of pricking, or reduction of particle size in dice or minces, increased the maximum sublimation rates.

PUMP-DOWN TIME

Several tests were carried out to determine how long it took for the surface temperature of pre-frozen materials to rise sufficiently for thawing to occur after the blast-freezing operation. It was found to be extremely difficult to measure the surface temperature accurately, but visual examination could distinguish between frozen or 'wet' tissues sufficiently closely for all practical purposes. With cod steaks 15 mm thick pre-frozen to a centre temperature of $-15°F$ ($-26°C$) in an air stream at $-30°F$ ($-37°C$), it was noticeable that the surface became wet after about 10 min exposure to an ambient temperature of about 65°F (18°C). If the dimensions of the pieces were smaller than this, faster warming of the product occurred, and since it was essential that no thawing should take place if a good product were to be obtained, it was decided to call for a pump-down time (to 1 mm Hg) of 4 min. Experience eventually suggested that some relaxation of this feature was possible, as the actual pump-down time attainable in the plant was 5 min in a dry system, and since this proved more than adequate it is probable that a value of 5 min to 4·5 mm Hg would have been quite acceptable.

The other consideration, apart from influence on drying time, was the rate of removal of water by evaporation while freezing in the cabinet. The product would eventually freeze no matter what the initial pumping capacity might be (assuming the heat input was restricted at this stage), but the removal of water would have taken place from the liquid phase with all the attendant disadvantages

of this type of drying. The slower the pump-down, the more liquid-phase drying would have occurred, and an inferior product would have resulted. However, if the 5 min period to 4·5 mm Hg had been specified even this second consideration would doubtless have been met.

Instrumentation

Apart from the normal gauges and meters which were necessary for the operation of the evacuating equipment, plate heaters, electric motors, etc., and measuring the energy consumption, it was necessary to decide on the most suitable instrumentation to control and record the actual dehydration process.

This could be subdivided as follows:

Temperature
Absolute Pressure
Plate Spacing

Temperature

It was realized that control of the process by measuring product temperature at isolated points was not ideal, but in view of the fact that considerable experience had been gained with this method, and since no better alternative system had been evolved, the use of thermocouples was specified. As on previous plants, this consisted of 12 plug-and-socket adaptors inside the cabinet together with 2 thermocouples in oil-filled pockets at inlet and outlet heating-plate connections. These were all connected to a 16-point electronic recorder similar to that fitted to EVE. As a check on the water temperatures, a protected glass thermometer was called for in the connecting pipe work.

Absolute pressure

For experimental work it was essential to have a reliable means of indicating accurately the operating pressure within the cabinet. Three $\frac{1}{2}$ in. B.S.P. connections were asked for to permit the use of the Alphatron gauge mentioned previously, and also a tilting McLeod and a capsule gauge. In addition, it was intended that one of the internal thermocouple sockets should be used for an icicle as discussed in the next chapter.

Plate spacing

It was originally hoped that the hydraulic system for moving the heating plates would be sensitive enough to indicate when contact had just been made between the product and the expanded metal, and consequently no spacing indicator was called for. However, the frictional resistance of the moving parts was found to be equivalent to an applied hydraulic pressure of about 3 atmospheres, and it was therefore necessary to fit a spacing indicator. This took the form of a variable resistance mounted on the hydraulic cylinder and rotated by the movement of the piston, indicating the position electrically on a recorder mounted outside the cabinet. This method was selected since it was undesirable to make holes in the cabinet wall for a mechanical drive, as not only would the inner protective coating be damaged, but it would require a vacuum-tight seal which would be a potential source of leakage.

Conclusion

The full Specification derived for the Mark I Cabinet, together with the design figures provided by the manufacturers, appears in the following chapter.

As indicated at various points throughout this text, some relaxation of requirements proved possible, but the Specification for Mark I has served as a basis for the initiation of the commercial interest in the AFD process for foodstuffs in many countries, and in addition has provided a basis for comparison with other equipment now being designed for the purpose throughout the world.

CHAPTER IV

The Mark I prototype AFD plant

THE specification of a Mark I commercial prototype AFD drier was completed, as outlined in the previous chapter, by 1959, and made available to all enquirers. Armour and Company had a cabinet conforming to these specifications built by A/S Atlas of Copenhagen, and installed at Aberdeen in 1960. The evacuating equipment, again manufactured to Ministry of Agriculture, Fisheries and Food performance specifications, was supplied by Messrs. Hick, Hargreaves and Company, Ltd., of Bolton, England.

Although the details of the Atlas-built Mark I unit will not be the same as those of subsequent Marks, or of plants of other manufacturers, they have been described at some length because it is thought that they include much of basic importance and potential value to engineers and food technologists alike. Similarly some space is given to problems which arose when the equipment was installed and tested; similar difficulties may be encountered by other workers in the field.

In the following pages it will be seen that in some cases equipment did not function as specified, but just as the whole concept and process of accelerated freeze-drying is in its infancy, so are the engineering techniques associated with it; the problems of, for example, obtaining and maintaining absolute pressures of the extremely low orders required, on the scale demanded, where such large quantities of water vapour are involved, are as new to the engineering world as are AFD foods in the realm of food technology. In such cases, therefore, no criticism of the manufacturers is intended; but equally the mention of a manufacturer's name should not be taken to imply that his products are preferred.

It has already been indicated that the design was based on drying conditions for pre-frozen cod steaks, since this material had proved to give the highest sublimation rate of all foodstuffs handled, but the design was based on the premise that the equipment should be capable of dehydrating a wide range of foodstuffs. Accordingly, if it were intended to concentrate on one type of product some of the physical variables specified could be modified. In addition it was essential that where any doubts existed as to the necessary values of any physical variable, the tendency should be towards over-design.

Operation of the plant showed that the design was successful with all meats and vegetables, but it appears that some fruits require a lower absolute cabinet pressure to ensure adequate depression of flesh temperature during drying. (It may be that, owing to supercooling, they were never truly frozen.)

DEHYDRATION CABINET

The plant consisted of a vacuum cabinet in which trays of foodstuffs to be freeze-dried were placed between contact plates under strictly controlled conditions of vacuum, temperature and mechanical pressure (Fig. 2, facing page 19).

Capacity

The cabinet was capable of holding 15 trays (nominally 750 mm × 2,000 mm) (2 ft 5½ in. × 6 ft 6¾ in.) giving approximately 22·5 m² (242 sq. ft) of tray area. The space between heating plates when fully open was 40 mm (1⅝ in.) and closed to a minimum opening of 10 mm (⅜ in.). Depending on the nature of the foodstuff the total input load could be varied between 450 lb and 900 lb with 650 lb as a fair average.

Cabinet Construction

The cabinet was a mild steel welded cylindrical vessel about 2,200 mm (7 ft 2 in.) diameter and 2,000 mm (6 ft 7 in.) long, with a hinged door at each end, each door being supported by a roller wheel on floor rails. The doors were fitted with a ½ in. diameter elastomer 'O' ring vacuum seal supplied by Edwards High Vacuum, Ltd., Crawley, Sussex. This seal was located in a special machined trapezoidal groove and location dowels were fitted. Each door was fitted with three viewing ports and the shell and attachments were suitable for operation at an absolute pressure of 1 mm Hg.

Ejection suction ports were 304 mm (12 in.) diameter and 457 mm (18 in.) diameter attached to and located on top of the cabinet, and flanged to suit isolating valves supplied by the manufacturer of the evacuating equipment.

The internal surfaces of the shell and all internal parts not made of stainless steel were protected by an air drying epoxy resin coating ('Marinat'). All external surfaces on the shell and auxiliaries received a protective covering of two coats of red oxide paint prior to assembly.

Heating Plates

Arranged in tiers, with intermediate stiffening of adequate rigidity, these plates were flat and hollow with internal twin-path labyrinths for the circulation of hot water. The plates were connected by ample lengths of flexible stainless steel tubing in parallel flow to headers within the shell, and there was a manually operated regulating valve for each plate.

Heating plates were of all-welded construction in stainless steel to British Standard Specification E.N. 58 G (C–0·15; Si–0·2; Mn–2·0; Ni–9·0 to 12·0; Cr–17 to 20; S–not more than 0·05; phosphorus not more than 0·05). The plate pressure mechanism was hydraulically operated from an oil cylinder within the shell capable of giving a uniform controlled pressure to each tray of 0·545 kg/cm² (7¾ lb per sq. in.) of plate area.

The heating system, designed for an operating pressure of 3·16 kg/cm² gauge (45 p.s.i.g.), consisted of 1 heat exchanger and expansion tank with requisite pumping equipment, changeover valve for flow reversal, and piping. A second 4-way valve was fitted to permit isolation of the expansion vessel and connecting piping so that the water in the plates could be cooled more quickly between batches. Heating water flow-rate was 1·1 m³/min (240 g.p.m.), and the heater was designed for a working pressure of 8·44 kg/cm² gauge (120 p.s.i.g.) on the steam side. Provision was made for direct injection of steam so that the circulating water could be heated as quickly as possible but the heat exchanger alone was capable of raising the heating water temperature from 15°C to 140°C in not more than 15 min, when using dry saturated steam at the design pressure. Connections were provided for flushing the system with cold water to permit lowering of the water temperature at the full flow-rate from 100°C to 60°C in

not more than 15 min, when using cooling water at a mean temperature of 13°C and a maximum pressure of 3·52 kg/cm² gauge (50 p.s.i.g.). The complete heating water system was hydraulically tested to twice the operating pressure.

General

The plant was supplied with pipe fittings to British Standard specification and electrical equipment suitable for a power supply of 415 volts, 3 phase, 50 cycles.

Since two of the first Mark I cabinets were to be shipped to U.S.A. the design complied with the A.S.M.E. code of practice for pressure vessels.

Guarantee

An assurance was given that the plant would conform to the specified physical conditions and the mechanical construction was guaranteed for a period of six months from the date of delivery at purchaser's premises.

It was specified that on final assembly, the cabinet should suffer a rate of air leakage not exceeding 10 g/min when evacuated to an internal pressure of 3 mm. The actual test leakage rate was well below this value, being about 0·1 g/min.

EVACUATING EQUIPMENT (Fig. 3)

Evacuation was by a steam ejector system, consisting essentially of steam augmentors which removed air and vapour from the cabinet and transferred them to a condenser, and ejectors which removed non-condensible gases (i.e., air) from the main condenser. In more detail the equipment comprised two 2-stage vacuum augmentors, in parallel, the units having $\frac{1}{3}$ and $\frac{2}{3}$ capacity respectively. The discharge from these augmentors was condensed in a low level direct-contact condenser, the vacuum in which was maintained by means of a 2-stage steam operated ejector, initial boosting being provided by a separate quick-starting 2-stage ejector. The condensed steam and cooling water was removed from the condenser by means of a motor-driven centrifugal extraction pump.

The evacuating equipment had been designed in accordance with the following technical particulars:

Suction pressure in cabinet	1 mm Hg abs.
Total vapour duty	200 lb/hr
Permissible air leakage rate	11 lb/hr
Operating steam pressure	115 p.s.i.g. D. & S.
Steam consumption of $\frac{1}{3}$ duty Primary Augmentor	160 lb/hr
Steam consumption of $\frac{2}{3}$ duty Primary Augmentor	320 lb/hr
Steam consumption of $\frac{1}{3}$ duty Secondary Augmentor	208 lb/hr
Steam consumption of $\frac{2}{3}$ duty Secondary Augmentor	415 lb/hr
Steam consumption of 2-stage Maintaining Ejector	170 lb/hr
Main condenser operating temperature	72°F (22°C)
Cooling water temperature	55°F (13°C)
Peak cooling water consumption	11,570 gal/hr
External head on extraction pump	15 ft
B.H.P. absorbed	9
B.H.P. of motor	10
Speed of pump and motor	960 r.p.m.
Volume of cabinet to be evacuated	346 cu. ft
Time to attain full vacuum	5 min
Steam consumption rate of quick-starting booster ejector	1,900 lb/hr

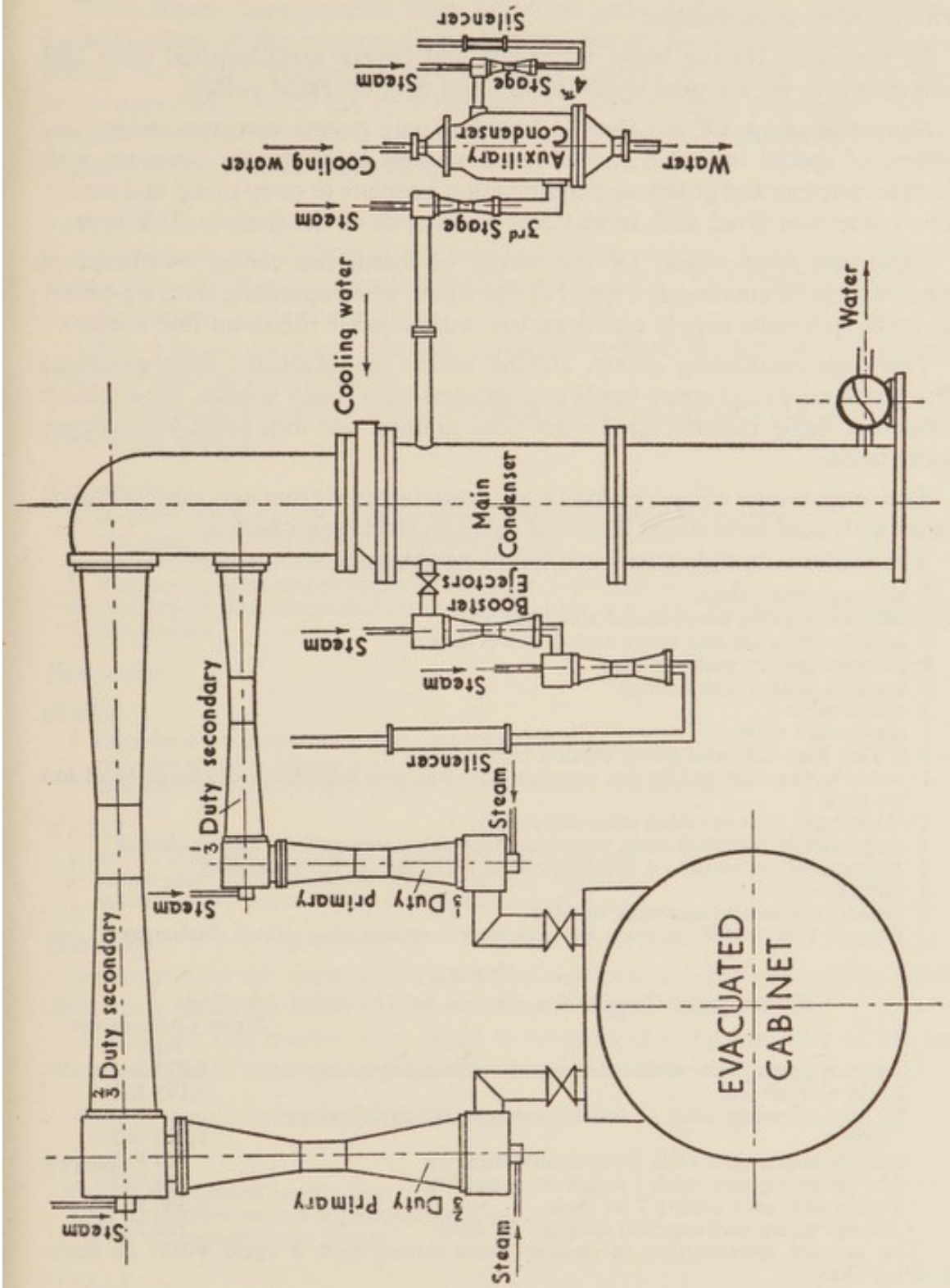


FIG. 3. *Schematic four-stage evacuating system*

MATERIAL SPECIFICATIONS

Multi-nozzle augmentors. Having suction head and combining tube made from cast iron with mild steel steam heads, nozzle plates and covers, complete with stainless steel nozzles.

Jet condenser. Having body, water-box and covers manufactured from cast iron complete with copper spray nozzles and float operated switch.

Extraction pump. Of the Double Impellor type having cast iron casing, impellors of special bronze mounted on a stainless steel spindle complete with flexible coupling and guard, and combination bedplate to carry pump and motor. The pump was fitted with mechanical gland seals of the Seatrist T-Y type.

Extraction pump motor. Of the totally enclosed, fan cooled weatherproof type suitable for running at a speed of 960 r.p.m. when operating from a 3-phase, 50 cycle, 415 volts supply and complete with suitable direct-on-line starter.

Two-stage maintaining ejector. Having bodies manufactured from gunmetal fitted with mild steel steam heads and stainless steel steam nozzles, the auxiliary condenser being manufactured from close grained cast iron fitted with copper spray nozzle.

Two-stage booster ejector. Having body manufactured from cast iron/gunmetal fitted with mild steel steam head and stainless steel steam nozzle.

The unit was complete with the following fittings:

7 steam control valves
2 cabinet isolating water-sealed sluice valves
3 cooling-water control water-sealed sluice valves
6 pressure gauges with syphons
2 vacuum gauges with fittings
1 sluice valve
1 non-return valve
3 ft 7 in. bore cast iron pump suction piping
1 water-level gauge and fitting, together with cast iron augmentor discharge bend and tee pipe
1 'Mowbrey' float operated magnetic switch
1 magnetically-operated main steam isolating valve arranged for spring closing
1 magnetically-operated air breaking valve, arranged for spring closing
1 rectifier set
2 bronze vapour test metering nozzles
2 Burgess type AMW silencers for booster and maintaining ejector discharges

TABLE 2

Ejector Steam Consumption Analysis for Theoretical 7 hr Cycle

	Steam Consumption Rate
3 min operating time with booster and maintaining ejector	3,070 lb/hr
2 min with all jets	3,173 lb/hr
55 min operating time at full augmentor capacity (booster OFF)	1,273 lb/hr
2 hr operating time with $\frac{2}{3}$ augmentor capacity	905 lb/hr
4 hr operating time with $\frac{1}{3}$ augmentor capacity	538 lb/hr
Total steam used during 7 hr cycle	5,338 lb
Average steam consumption during 7 hr cycle	760 lb/hr

(The *average* consumption of cooling water during such a cycle would be about 6,500 gal/hr.)

It should be noted that the steam consumption shown above referred only to the evacuating equipment. In addition to this there was a demand of 2,500 lb/hr for the cabinet water circulation system for a period of 15 min; this demand normally occurred from 5 to 20 min after the beginning of the cycle. The overall *maximum* steam demand was therefore 1,273+2,500 lb/hr = 3,773 lb/hr. The

initial heater demand added to the subsequent requirements to maintain the operating temperature level in the heating plates gave a mean usage of 170 lb/hr, i.e., an *overall average* consumption of 760+170 = 930 lb/hr.

These steam consumptions were based on estimated figures given by the manufacturers of the evacuating equipment, but under actual operating conditions the $\frac{1}{3}$ duty augmentor did not prove capable of dealing with the load at the expected point in the cycle (see above) and the $\frac{2}{3}$ duty augmentor had to be used throughout, which raised the average steam consumption from the theoretical 930 lb/hr to 1,100 lb/hr.

Instrumentation

A faced hole was drilled in the side wall of the cabinet to take a bulkhead fitting (No. 2CZ84269) manufactured by Plessey Co., Ltd., Ilford, Essex, for 25-way thermocouple connections.

Three $\frac{1}{2}$ in. screwed and plugged connections were welded into the cabinet shell for vacuum measurement.

The following instruments were mounted either on the panel or on the interconnecting piping:

Temperature

1 16-point thermocouple recorder with an ice reference cold junction
1 dial gauge on main condenser discharge
1 mercury-in-glass thermometer on main condenser discharge
1 mercury-in-glass thermometer on auxiliary condenser discharge
2 mercury-in-glass thermometers on inlet and outlet circulating water connections to the cabinet.

Flow rates

STEAM

1 recorder on augmentors and maintaining-jets supply line
1 recorder on plate heater 1 in. supply line
1 indicator on booster ejectors and 2½ in. diameter heater supply.

WATER

1 orifice plate and U-tube on circulating water system
1 orifice plate and U-tube on main condenser discharge
1 meter on cold water supply for forced cooling.

Electricity

Ammeters on the drive motors for the condensate extraction pump, water circulating pump, hydraulic pump and forced supply pump.

In addition two meters were fitted to measure the consumption of the sea water pumps for cooling water and the remainder of the demand.

Pressure gauges were fitted as follows:

STEAM

On the main steam supply line
Before all 8 ejector and augmentor nozzles
Before circulating-water heater

WATER

On the circulating-pump discharge
On the forced cooling-water supply line
On the expansion vessel

OIL

Two gauges were fitted to indicate hydraulic pressure within the main oil cylinder. One read from 0–10 kg/cm² while the second read 0–60 Kg/cm²

Vacuum gauges were fitted as follows:

Absolute pressure recorder
(0–30 in. Hg) and (0–40 mm Hg) on main condenser
(0–30 in. Hg) on auxiliary condenser
(0–760 mm Hg) and (0–40 mm Hg) for cabinet but mounted on instrument panel
(0–20 mm Hg) on cabinet
Vacustat gauge on cabinet
Alphatron gauge on cabinet
(0–40 mm Hg) on both primary augmentor discharges

Safety Precautions

A rupture disc diaphragm, 2 in. diameter, designed to fail at a positive pressure of 0·3 Kg/cm² (5 p.s.i.g.) was fitted to the cabinet shell, and a flanged pipe connection 2½ in. I.D. was also fitted for a vacuum breaker valve. Two spring-loaded safety valves were fitted on the water circulation system; one on the expansion vessel and the second one near the steam heater to give protection when the expansion vessel was isolated.

In the vacuum system, in order to prevent accidental flooding of the cabinet with water from the main condenser, a float-operated magnetic switch was fitted. In the event of the level of the cooling water in the condenser rising excessively, this switch was to close a solenoid-operated valve on the steam line to the jets, and simultaneously open a second valve to admit air above the nozzle ring on the main condenser, so reducing the suction head on the cooling water supply line.

The electricity consumption of the dehydration cabinet ancillaries was 19 kW, of which 4–5 kW was absorbed by the superheaters on the primary augmentors, but it is not absolutely certain that the superheaters were essential (see below).

A further 9·5 kW was consumed by the sea water pump but it is considered that a smaller pump, consuming about 7·5 kW, would have been adequate.

The cooling water flow rate to the main condenser was verified in practice as being approximately 11,500 gal/hr when using both augmentor sets and 8,000 gal/hr when the ⅔ augmentor was used alone.

Installation

The straightforward procedure would have been to erect the cabinet and then to assemble the augmentors, finishing with the main condenser; but since the evacuating equipment was delivered first, it was decided to install the condenser and second stage augmentors, and to support the primary augmentors in their proper positions. This permitted the erection of all the evacuating system service lines and the bulk of this work was in fact completed prior to the delivery of the cabinet. The cabinet (weighing approximately 10 tons) was then manoeuvred into position, jacked up and grouted in. It was found that the ⅔ augmentor mated exactly with its isolating valve flange, but the ⅓ primary suction flange, although parallel to its valve flange, required a packing ring about $\frac{3}{16}$ in. thick. This was machined from mild steel and, as with all other flanges on the evacuating system, was sealed with high-pressure jointing liberally coated with graphited grease.

The steam line dimensions used were as recommended by the manufacturers, and precautions were taken to ensure adequate trapping to make the nozzle steam supply as dry as possible. Steam was generated at 150 p.s.i.g., and originally was reduced to 115 p.s.i.g. through a reducing valve. Subsequent tests showed that this valve would not pass the required weight of steam without an

excessive pressure drop, and the valve was therefore removed. The steam supply from the stop valve was through a drier trap to the second stage maintaining ejector with the booster primary stage fed through a T-connection. Opening the second stage maintaining ejector admitted full steam pressure to the first stage maintaining ejector control valve, which in common with the other valves was mounted direct into the line with an extension spindle. When the first stage maintaining ejector was brought into commission, steam was automatically admitted to a supply manifold mounted at a convenient level, with supply valves feeding firstly the secondary augmentors and the primary augmentor control valves. Finally, when the valves to the primary augmentors were opened the steam supply to the plant was complete.

As mentioned in Chapter III, the possibility of ice building up inside the combining tube of the primary augmentor could not be ignored, and consequently copper tracing tubes fed with steam at line pressure were wrapped round the entire outer surface of this tube. In addition superheaters were fitted on the steam supply lines to the primary augmentor nozzles, rated to give 50°F (28°C) superheat at 115 p.s.i.g. These were electrical units with maximum capacities of 6 kW and 12 kW respectively, and were controlled by bi-metallic thermostats in the discharge steam supply lines to the nozzles. During the later proving runs on the plant the superheaters were not used, and it was possible to maintain stable vacuum conditions without them. However, previous attempts to isolate the steam tracing tubes had introduced an intermittent pressure fluctuation similar to that experienced in EVE, and it would therefore appear that some form of heat application is required to prevent ice accumulation. It is also possible that prolonged operation with wet steam might result in erosion of the $\frac{1}{16}$ in. bore nozzle throats, but the test period was not long enough to verify this.

Acceptance Tests

Cabinet

It was found that the heating-plate pressure frames were out of alignment. These should have been parallel and equal on all three banks to within 1 mm but discrepancies of about 6 mm were found. The method of correction was to fit new support-bar connecting links between the pressure-frame support bars and the operating levers on the overhead transverse shafts. With careful attention to fitting detail the plates were made parallel within the required tolerance.

Oil leakage from the main hydraulic cylinder was corrected by increasing the load on the hydraulic ram packing ring. This increased the frictional resistance offered to movement of the ram, so making it more difficult to relate oil pressure to pressure on the product. However, it was thought that the possible risk of flooding the cabinet with oil and contaminating the foodstuff was less desirable.

Evacuating equipment

The greatest deviation from design conditions was found to exist in the pumping capacity of the steam ejector system.[7] The initial proving runs were carried out with cabbage, and it soon became obvious that vapour was not being removed from the cabinet at the required rate. This caused the absolute pressure in the cabinet to rise above 1 mm Hg in the initial stages, and subsequent interchange of the $\frac{1}{3}$ and $\frac{2}{3}$ augmentor sets could not be made at the predicted times.

Calibrated test orifice plates had been provided by the manufacturers, and these were used to check the augmentor characteristic curves which had also been supplied. The initial tests were made with an Alphatron gauge to measure the absolute pressure in the cabinet, and this indicated a pumping capacity in the $\frac{2}{3}$ augmentor of about 50 per cent of the design figure and slightly less than 50 per cent in the $\frac{1}{3}$ set. The manufacturers had used a McLeod gauge with a silica gel trap, but no correlation could be obtained with a similar instrument in Aberdeen. Further tests with an icicle, consisting of distilled water frozen round a thermocouple and suspended in the evacuated cabinet, gave an indication of wet bulb temperature in the cabinet which was very close to the equilibrium value with the partial pressure of water vapour present. This, in addition to a McLeod gauge with a refrigerated glass-wool trap indicating partial air pressure, suggested that the Alphatron reading was substantially correct. As a further check, an oil-filled U-tube was connected between the cabinet and an auxiliary evacuated system to which was connected a McLeod gauge. With this arrangement the McLeod gauge would be expected to give a correct reading in the dry auxiliary vessel, and by adding to this value the differential on the U-tube, an indicated cabinet pressure was obtained which again agreed with the Alphatron. A waxed Vacustat gauge was also utilized,[8] and this corroborated the Alphatron results.

The first indication thus was that the evacuating equipment was capable of pumping only about half of the designed figure, but subsequent tests indicated that the temperature of the pumped vapour was much higher than had been assumed. Measurement of the temperature at the primary augmentor suction port indicated about 60–80°C (140–176°F) superheat, and since this would have a pronounced effect on the specific volume of the vapour, it partially accounted for the discrepancy in pumping capacity. However, it was quite evident that considerable care should be exercised also in the selection of a suitable vacuum gauge. For testing purposes the Alphatron type of gauge has proved to be invaluable, but since this is a fairly expensive instrument one could not justify its use on a production plant. Probably the most suitable unit for this type of installation would be a Vacustat gauge treated internally with paraffin wax, in conjunction with an icicle suspended inside the cabinet. Unfortunately the stability of the waxed gauge is not all that might be desired, since after about two months there was some lack of agreement at high sublimation rates. In addition to these gauges it was felt advisable to have at least one dial type gauge to cover the range 0–760 mm Hg.

Some trouble was experienced with the booster ejector which as originally supplied was a single stage unit. Preliminary tests indicated a pull-down time to 1 mm Hg of 15 min as opposed to the desired figure of 4 min. Subsequently a second stage was added, and with a load of pre-frozen material the best pull-down time was 5 min. Since there was no visible sign of thawing in the product under these conditions it was agreed that this was acceptable.

Control of the water level in the main condenser proved to be difficult, since the float-operated switch tripped while the indicated water level in the sight glass was well below the danger level. It was found that the gauge-glass cocks had an extremely small orifice which restricted the rate of flow into the glass, so that when the level was rising in the condenser it did not rise so quickly in the glass. This was rectified by installing new gauge-glass fittings with larger orifices.

Test Results

The most serious fault in the equipment as installed was the discrepancy in pumping capacity. As already mentioned this was based on the maximum sublimation rate of about 0·9 lb/hr/sq. ft of tray area which had been found possible on EVE with raw cod steaks. Since the evacuating system could not handle vapour at this rate, the absolute pressure tended to rise excessively at the beginning of the cycle and consequently the rate of heat input had to be reduced. The comparable sublimation figure for cabbage appeared to be about 0·8 lb/hr/sq. ft, and most meat products, root vegetables and fruit gave a figure of about 0·4 lb/hr/sq. ft. It was therefore proved to be quite possible to handle foodstuffs in the latter category very successfully at the maximum rate, and the general result was that it was proved conclusively[6] that a successful product could be obtained from this type of plant.

CHAPTER V

The drying operation

REFERENCE to the combination of graphs in Fig. 4 gives a picture of the general operation which was applied to the Mark I AFD prototype. The main controllable variables were the temperature of the heating plate water and the pressure applied by the plates on the material; and the programme of variation of these differed for each foodstuff. Since subsequent equipment will differ in design, all that can be offered here is a review of the principles involved, as they were applied to practical drying runs.

It was not possible to measure the actual pressures applied by the plates, and recourse was made to the manufacturers' calculations which, making allowance for friction, yielded the information that the ratio of hydraulic ram pressure to plate pressure was about 120 : 1. It will be seen that Fig. 4 shows a maximum of about 4 p.s.i. on the tray, which is a fairly representative example. It is important to realize, however, that the pressure on a foodstuff was greater than this if the tray was not completely filled. For example, beefsteaks covered about 60 per cent of the area of the tray and at a plate pressure of 4 p.s.i. suffered an actual pressure of about 6½ p.s.i.

As to plate temperatures, they naturally varied but could be highest in the case where evaporation was most rapid (e.g., with codfish steaks) and the plates could be run at a temperature of 120–140°C, with a pressure of 6½ p.s.i. In such a case the evaporative cooling was sufficient to keep the ice from melting and the dried surface from reaching 60°C. An example of the temperature gradient which could be set up is: heating water temperature 140°C; tray side of expanded metal 60°C; food side of expanded metal 40°C; ice temperature inside food −20°C.

LOADING

The material for drying was evenly distributed on the 30 trays used for one complete load of the cabinet. In the Mark I cabinet there were 15 tray spaces, but for convenience in handling, half-plate size trays were used, loading taking place simultaneously at each end of the cabinet. Normally the trays were loaded using expanded metal sheets on both sides of the material. For some materials this could be dispensed with (e.g., those foods which of themselves formed an open bed, such as cooked meat minces). When pre-frozen material was being dried, it was usual to load it at a temperature of −25°C or lower. A blast freezer was specially designed for this purpose, so as to take a full load of material (on the drying trays) in two trolleys. Each trolley took the 15 trays for loading one end of the cabinet.

As has been stated earlier, the average wet-weight loading of the cabinet was in the region of 2 lb/ft², utilizing some 60 per cent of the total tray area in the case of steaks of meat or fish. It was essential for the material to be loaded in such a manner that variations in thickness of the layer were as small as possible.

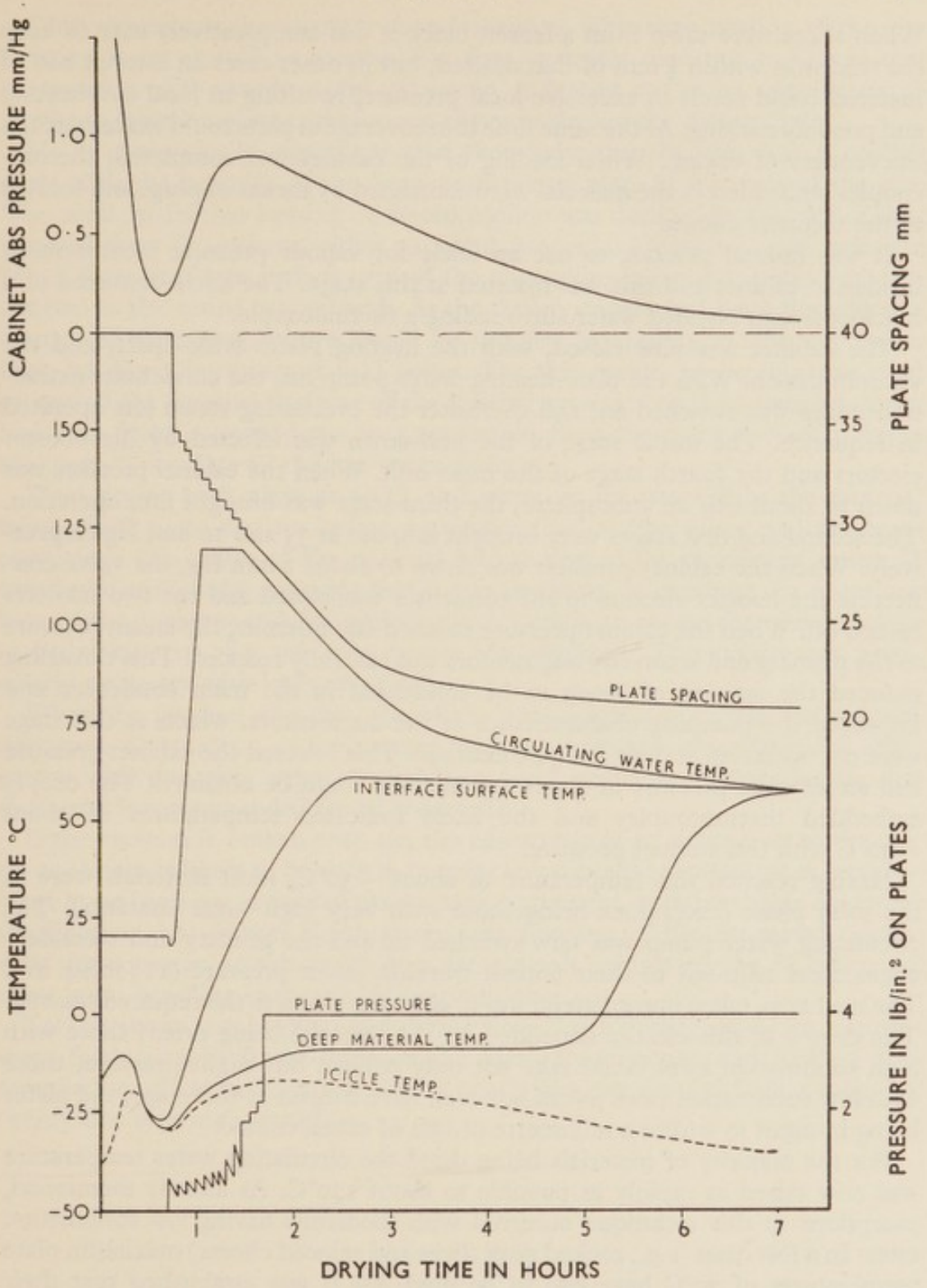


FIG. 4. *Typical AFD drying conditions*

Two factors govern the temperature to which it is normal to pre-freeze the foodstuff:

The temperature should be low enough to bring it into the solid phase and allowance must be made for the rise in temperature during the time taken to load the cabinet and to achieve a suitable vacuum to hold down the temperature of the material. In practice the time involved is about 15 min, the temperature rise during this period being about 15°C. The blast-freezer must therefore deliver the foodstuff at about 15°C below melting point.

When slices were sawn from a frozen block it was comparatively easy to keep the thickness within ½ mm of that desired, but in other cases an uneven bed of material could result in excessive local pressure, resulting in local overheating and possibly melting. At the same time thin coverage in parts could exacerbate the unevenness of drying. When loading of the cabinet was completed, thermocouples embedded in the material were connected by means of plugs and sockets to the recorder circuit.

It was normal practice to use an icicle for vapour pressure measurement inside the cabinet and this was inserted at this stage. The icicle consisted of a block of frozen distilled water surrounding a thermocouple.

The cabinet was now closed, with the heating plates wide apart, and the vacuum drawn. With the plate-heating water pump off, the condensate extraction pump was switched on, and thereafter the evacuating steam jets operated in sequence. The initial stage of the pull-down was effected by the booster ejectors and the fourth stage of the main unit. When the cabinet pressure was down to about half an atmosphere, the third stage was brought into operation. The second and first stages were brought into use at 35 and 10 mm Hg respectively. When the cabinet pressure was down to about 2 mm Hg, the valve connecting the booster ejectors to the condenser was closed and the two boosters turned off. When the cabinet pressure reached 600 microns, the steam pressure to the primary and secondary augmentors was carefully reduced. This throttling reduced the amount of steam to be condensed in the main condenser and improved the pumping characteristics of the augmentors, which at this stage were not operating at their design condition. This lowered the cabinet pressure and an absolute pressure of about 250 microns could be obtained. The deeply embedded thermocouples and the icicle indicated temperatures of about −30°C with this cabinet pressure.

Having reached this temperature of about −30°C, most materials were in the solid phase (exceptions being those with very high sugar contents). The circulating water pump was now switched on and the primary and secondary augmentors adjusted to their normal working steam pressure (115 lb/sq. in.). The next step, taken immediately, was to close the plates to the required spacing. The degree of this closure depended on the material being dried; those with high sublimation rates could take not only contact but slight pressure, those with low sublimation rates would not even have contact at this stage, the plates being brought to within a millimetre or two of actual contact.

For the majority of materials being dried the circulating water temperature was now raised as rapidly as possible to about 120°C. As already mentioned, exceptions to this technique occurred with foodstuffs having low sublimation rates. In a few cases (e.g., cooked pork slices and minced cheese) maximum plate temperatures of 70°C have had to be used, for it was established that their dried surfaces could not be permitted to reach a higher temperature than 40°C for fear of rendering out their fat.

The circulating water temperature was normally held at the maximum value until the surface temperature of the material being dried rose to about 55°C, which was usually a period of between 30 and 90 min. Thereafter it was allowed to drop by natural cooling. The plate closures continued steadily until resistance was met (as indicated on the hydraulic pressure gauge). Continuous pressure was now applied, at the appropriate level determined by experiment, and pressures of about 7 lb/sq. in. have been used for some materials without damage.

With experience the operator soon learned at what time heating of the circulating water should be stopped for any particular foodstuff to ensure that overheating of the surfaces did not occur. It turned out to be unnecessary to employ a forced cooling technique at any time during the drying cycle.

(It is economically significant that thermodynamically this was a cooling cycle with the majority of the heat input taking place at the start of the run. The need for further heating or forced cooling was decided by the rate of heat removal from the system, either as latent heat of sublimation or as radiation losses from the large surface area of the connecting pipework, including conduction to the actual cabinet wall. At the design stage it had been thought that control of plate temperature would be simplest by cooling in this way rather than by direct injection of cold water. For this reason no lagging was used on any of the external surfaces of the heating system; but if an indirect cooler had been fitted it would have been preferable, from the point of view of steam economy, to insulate all the hot-water surfaces.)

Final drying took place at 60°C, and when no ice was left in the material being dried there was no longer an evaporative cooling effect and the temperature in the centre of the material rose to that of the circulating water. It was found with some materials that it was necessary to continue the operation beyond the apparent end, for periods up to 30 min, to ensure that the desirable moisture content of 2 per cent or less was obtained. This tailing was particularly necessary with leafy vegetables.

When the product was dry, the plates were opened and then the vacuum destroyed. Destruction of the vacuum with dry nitrogen was considered obligatory with materials especially liable to rapid oxidative changes in the dry state, and is recommended for all foodstuffs.

If the vacuum is broken with air, the adsorption of its oxygen on to the dry tissue surface is likely to be such as to resist any subsequent attempts at replacement by an inert gas in normal gas-packing equipment, with inevitable oxidative deterioration on storage. Furthermore, the flooding of the evacuated tissues with dry nitrogen at this point goes far towards mitigating the ill effects of subsequent handling in the air before packaging.

Table 3 shows tray loadings and drying times (with drying ratios) for twenty-seven foods actually freeze-dried on the Mark I equipment, and Table 4 is a compilation of the freezing and drying conditions experienced with a range of twenty-one typical foodstuffs.

TABLE 3

Typical Wet-weight Loadings and Drying Times
(Actual experimental runs)

Material	Drying Ratio	Loading (lb/ft^2)	Drying Time (hr)
Raw Beef	*3·6 : 1	2·5	6½
Raw Pork	*3·2 : 1	2·5	7½
Raw Lamb	*2·9 : 1	2·5	7½
Cooked Beef Mince	*3·0 : 1	3·0	6½
Cooked Beef Slices	*2·9 : 1	3·0	7
Cooked Pork Slices	*2·7 : 1	3·0	7½
Cooked Lamb Slices	*2·9 : 1	3·0	7½
Cooked Ham Mince	*2·5 : 1	3·0	9
Cooked Veal Mince	*3·3 : 1	3·0	7
Cooked Chicken	2·9 : 1	2·5	7½
Raw Cod Steaks	5·1 : 1	2·5	8½
Cooked Cod Flakes	4·0 : 1	2·5	9
Cooked Prawns	5·1 : 1	1·5	6
Cheese Mince	1·6 : 1	2·0	8
Broad Beans	4·5 : 1	1·5	8½
French Beans	10·0 : 1	2·0	8½
Brussels Sprouts	8·6 : 1	2·0	9
Cabbage	13·2 : 1	2·0	9½
Carrot Dice	10·0 : 1	2·0	9
Cauliflower	11·0 : 1	1·5	8½
Peas	4·6 : 1	2·0	8
Potato Chips	5·0 : 1	1·5	8
Apple Dice	9·2 : 1	2·0	8½
Black Currants	5·7 : 1	1·5	8½
Plums	8·4 : 1	1·75	8
Raspberries	8·7 : 1	1·75	8½
Strawberries	15·0 : 1	1·75	9

*Figures will vary with fat content.

TABLE 4

Drying Conditions for Foodstuffs

Material	Thickness of Slices or Mat. *mm*	Loading Temperature *°C*	Plate Movement	Maximum Heating Water Temperature *°C*	Maximum Material Temperature (Final Heating Water Temperature) *°C*	Comments
Raw Meat Steaks and Cubes	15	−25	Initial closure with slight pressure, increasing gradually to a maximum 4 lb/in.²	120	60	With fatty slices of pork it is advisable to keep the material temperature to a maximum of 50° to avoid excessive rendering
Cooked Meat Minces (not Ham)	30	Frozen by evaporative cooling	Initial closure with slight pressure, increasing gradually to a maximum of 1½ lb/in.²	120	60	Material normally removed from the cabinet before the material temperatures reach 60°C. Material has normally been used for meat bar manufacture
Cooked Meat Slices (5 mm)	20 (2 layers, each of 2 slices thickness, with expanded metal sheet between the layers)	−25 (Can be frozen by evaporative freezing, but with somewhat inferior product)	Initial closure without contact (2 mm gap) Contact achieved after heating water is at maximum. Final pressure of 1½ lb/in.²	120	50	Final drying temperature is low to prevent ingress of rendered fat into lean tissue
Ham Mince	25	Frozen by evaporative cooling	As for cooked meat minces	80	40	Circulating water temperature low to prevent excessive rendering of fat

TABLE 4—continued

Material	Thickness of Slices or Mat. *mm*	Loading Temperature *°C*	Plate Movement	Maximum Heating Water Temperature *°C*	Maximum Material Temperature (Final Heating Water Temperature) *°C*	Comments
Raw Cod Steaks	15	−25	Initial closure with slight pressure, increasing gradually to a maximum of 6 lb/in.²	120	60	
Cooked Cod Flakes	25 (approx.)	Frozen by evaporative cooling	Initial closure with very slight pressure, increasing gradually to a maximum 4 lb/in.²	120	60	
Cooked Prawns	18 (approx.)	Frozen by evaporative cooling	As for cooked meat minces	100	60	Little experience with this material—figures based on one run only
Cheese Mince	20–25	−25 (Can be frozen by by evaporative cooling)	Initial closure without contact (2 mm gap). Slight contact (without pressure) achieved after heating water is at maximum. No pressure applied during run	70	40	Low temperature of circulating water throughout run, with absence of plate pressure to avoid rendering of fat
Broad Beans	15–20	−25	Initial closure without contact (5 mm gap). Plates closed at rate of 0·5 mm every 5 min until contact established. Maximum pressure about 1 lb/in.²	120	60	

TABLE 4—continued

Material	Thickness of Slices or Mat. *mm*	Loading Temperature °C	Plate Movement	Maximum Heating Water Temperature °C	Maximum Material Temperature (Final Heating Water Temperature) °C	Comments
French Beans	20 (approx.)	−25	No plate closure until the circulating water is at maximum temperature, then closure without contact (5 mm gap). Thereafter as for broad beans	120	60	
Brussels Sprouts	Max. 25	Frozen by evaporative cooling	Initial closure without firm contact (approx. 2 mm gap). Plates closed at rate of 0·5 mm every 5 min after circulating water reaches maximum temperature. Maximum pressure about 1½ lb/in.²	100	60	Little experience with this material—figures based on one run only
Cabbage	20 (approx.)	Frozen by evaporative cooling	As for Brussels sprouts	120	60	
Carrot Dice	20–25	−25	As for Brussels sprouts	120	60	High *sugar content* material vacuum destroyed with nitrogen
Cauliflower	15–20	Frozen by evaporative cooling	As for Brussels sprouts	120	60	

TABLE 4—continued

Material	Thickness of Slices or Mat. *mm*	Loading Temperature *°C*	Plate Movement	Maximum Heating Water Temperature *°C*	Maximum Material Temperature (Final Heating Water Temperature) *°C*	Comments
Peas	20–25	−25 (Can be evaporatively frozen but with inferior product)	As for Brussels sprouts	120	60	
Potato Chips	12·5	−25	Initial closure without contact (2 mm gap). Plates closed at rate of 0·5 mm every 5 min after heating water reaches maximum temperature. Maximum pressure about 4 lb/in.^{2}	120	60	
Apple Dice	20–25	−25	As for Brussels sprouts	120	60	High sugar content material
Black Currants	15–20	−25	As for French beans	120	60	Material abraded after blast-freezing to allow release of vapour during drying
Plums	15–20	−25	As for Brussels sprouts	120	60	
Raspberries	15–20	−25	As for Brussels sprouts	120	60	
Strawberries	15–20	−25	As for Brussels sprouts	120	60	Fruit cut longitudinally into halves (core out) before freezing

CHAPTER VI

Packaging

ANY food product requires packaging adequate to protect it from its environment according to its weaknesses to the factors of its environment, and it is evident that a fragile product needs a robust container to protect its physical form; but dehydrated foods, and perhaps particularly such foods prepared by the accelerated freeze-drying process, have packaging requirements additional to physical protection. The shelf-life of a dehydrated food depends like any other on a number of factors, some of which are influenced by the packaging and some not. This chapter is restricted to the effects on the stability of the food which should be achieved by efficient packaging.

PRINCIPAL CAUSES OF SPOILAGE

Those factors associated with the deterioration of dehydrated foods which can be influenced by packaging are: moisture uptake, oxygen uptake, flavour contamination, mechanical damage.

Other factors are dealt with in Chapter XI which gives a general description of the problems involved in the storage of dehydrated foods.

By examining each of the above factors in turn it is possible to arrive at a statement of the general requirements of a package suitable for a dehydrated food.

Moisture uptake

In the AFD process the final dry product is a rigid porous material, exposing a considerable surface area. The moisture content generally lies in the region of 2–4 per cent, which corresponds to a very low relative humidity (Table 5). Such foods are extremely hygroscopic and, in contact with an environment of greater relative humidity, will absorb moisture rapidly. Consequently they must be isolated as efficiently as possible from the atmosphere if moisture uptake is to be avoided.

The maintenance of the dry state is of twofold importance. Firstly, the major deteriorative chemical reactions in dehydrated foods (notably non-enzymic browning) are retarded at very low moisture levels (oxidation is an important exception, *q.v.*) and any increase in moisture content will increase the rate of these reactions, so shortening the shelf-life of the product. Secondly, the fundamental function of drying as a method of food preservation is to inhibit the growth of micro-organisms, and whether or not a micro-organism will grow is governed by the equilibrium relative humidity of the food product; bacteria in general requiring more than 90 per cent RH and moulds more than 70 per cent. Most dehydrated foods have an equilibrium relative humidity far below these limits but in an unsatisfactory package it is possible during long-term storage for the moisture content to increase to such an extent that the minimum level is exceeded.

TABLE 5

Relationship between Moisture Content and Equilibrium Relative Humidity at 20°C
g Water/100 g Solids (fat-free basis)

Equilibrium Relative Humidity	5	10	15	20	25	30	35	40	45	50	55	60	65	70	75	80	85
Raw Beef	3·5	4·2	4·9	5·7	6·4	7·3	8·2	9·3	10·5	11·8	13·2	14·8	16·5	18·4	20·9	24·3	29·8
Raw Cod	3·9	5·3	6·6	7·8	8·9	9·8	10·8	11·8	12·8	13·9	15·3	16·9					
Raw Pork	3·9	5·2	6·2	7·2	8·1	9·0	9·9	10·8	11·9	13·1	14·4	15·9	17·6	19·5	21·8	24·6	
Cooked Chicken	4·0	5·3	6·3	7·1	8·0	8·8	9·7	10·6	11·6	12·5	13·6	14·8	16·4	18·2	20·3	22·7	25·9
Raw Carrot	2·5	3·7	4·3	4·7	5·3	6·2	7·3	8·7	10·4	12·3	14·6	17·1	20·2	24·4			
Raw Peas	4·2	5·1	5·7	6·2	6·7	7·2	7·8	8·3	9·0	9·9	10·9	12·3	14·0	16·4	19·6	23·0	29·0
Raw Potato	4·3	6·2	7·5	8·4	9·2	9·9	10·4	11·1	12·0	13·1	14·3	15·7	17·2	18·9	20·6	22·7	25·4
Raw Cabbage	2·2	3·8	5·2	6·2	7·2	8·1	9·0	10·1	11·7	13·7	16·2	19·2	22·6	26·4	30·6		
Cooked Rice	4·8	6·7	8·0	9·0	9·8	10·6	11·6	12·6	13·7	14·8	16·0	17·2	18·4	19·6	20·8	22·1	23·6
Raw Black Currant	2·9	3·8	4·6	5·5	6·3	7·2	8·1	9·4	10·9	12·7	14·9	17·5	20·6	25·2	31·5	39·4	51·0
Raw Apple	2·1	3·1	3·8	4·4	5·0	5·9	6·1	8·8	10·8	13·4	16·8	21·2	27·0	34·2			
Raw Whole Egg	1·9	2·8	3·5	4·0	4·6	5·2	5·8	6·4	7·1	7·7	8·5	9·3	10·2	11·2	12·5	14·3	16·8

The relationship between equilibrium relative humidity and moisture content is peculiar to each product, and it is important for this relationship to be known, for it is significant in a number of ways. For example, it will enable a calculation to be made relating the moisture permeability of a packaging material to a safe shelf-life, i.e., predicting the time taken to reach a limit of acceptable ERH. It will also indicate the direction of transference of water vapour between components in packs containing a number of different items of dehydrated food, which could equilibrate in such a way that one or more might take up sufficient moisture from others to become susceptible to deteriorative change.

Oxygen uptake

Dehydrated foods are susceptible to deterioration when exposed to oxygen. Meats and other products containing fats develop rancidity; there is sufficient lipid material even in potatoes for strongly rancid flavours to develop when the dehydrated product suffers oxidation. Most green vegetables develop off flavours, frequently of a hay-like nature, and often some bleaching of the colour may occur; while carrots become bleached and develop a strong, unpleasant 'perfume'. Exceptions among vegetables seem to be peas, French beans and beetroot, which seem to deteriorate little when packed in air for periods up to a year. The effect of oxygen on dehydrated fruit has been little investigated; but in general they seem to be less sensitive than vegetables.

Because of the large surface area due to the porous nature of freeze-dried foods, it is necessary to avoid oxidation before removal from the drying cabinet, as previously described. For the same reason, calculations concerning the evacuation and inert-gas filling of containers of freeze-dried foods must take into account the fact that the headspace of the container actually penetrates the product, and is estimated to amount in most cases to some 80 per cent of the total package volume.

Associated with the effects of oxygen is the effect of light which, in some cases at least, catalyses oxidation and certainly adversely affects the colour of both meats and vegetables.

Flavour contamination

Experience has shown that this is not normally a problem, but trouble arose intermittently when meat bars, which contain a substantial amount of fat, were packed in some batches of a laminate consisting of rubber hydrochloride bonded to aluminium foil by an adhesive. The meat picked up a mineral oil flavour which was said to come from the adhesive. Development of more suitable adhesives has led to a marked reduction in the incidence of taints from this source, but in any laminate the adhesive must be fully evaluated and it would seem prudent to look towards extruded coatings for use where possible.

Mechanical damage

Two aspects of damage by mechanical means result from the particular structural properties of AFD products. Their light porous nature makes them rather fragile (some extremely so) and thus they may require protection against crushing not only during handling, but from atmospheric pressure after vacuum-packing in a flexible container. On the other hand, the tissues of which they are composed are often hard and sharp and may tend to damage a laminate by puncturing or tearing (notably the case with fish fibres). Both

these factors have to be taken into consideration when containers for AFD foods are being designed. Damage in transit may occur if the product can move in the container and some form of cushioning may be required if cans are used.

The Requirements for Protective Packaging

When a protective packing is being chosen for a dehydrated food product, several points as well as the fundamentals outlined above must be considered. Only when the following questions have been answered is it possible to choose the best package for the specific purpose:

What properties has the food product?
How long a shelf-life is required?
Under what conditions will it be transported and stored?
How much food is to be packed in each unit?
How much may be spent on packaging?
What packaging materials are available, and what are their properties?

Only when the answers to the first five questions have been established can the available materials be rationally considered, and the final solution will in all probability still be something of a compromise.

In general, deterioration increases with duration of storage and with temperature. Thus the protection required for 12 months' storage will be much greater than that for only 3 months, and for tropical greater than for temperate climates. The proposed shelf-life is, therefore, one of the basic factors in determining the packaging requirements. Others include the prevailing humidity and variations in temperature and humidity, warehousing facilities and conditions of transport and handling.

The size of the unit pack will have an important bearing on its design, a large-scale catering pack being essentially different from a domestic single-portion packet. Table 6 gives a collation of data relevant to the weights and volumes of eighteen representative dehydrated foods in cans.

Cost is, of course, also a factor which determines whether a particular package is feasible or not. If a plastic laminate or plastic and foil laminate is to compete favourably with tinplate as packaging material, it must also compete in cost, and in the present advanced state of development of tinplate containers and their closing machines this is unlikely. The non-rigid containers must therefore justify themselves by some other desirable characteristic, such as lightness.

There appears to be no universal solution to the problems of technical adequacy, consumer appeal and economic advantage. The necessary compromise must be to provide just the amount of protection to keep the product acceptable during its estimated shelf-life, protection in excess of this being unnecessary and economically wasteful. As has already been indicated it is necessary, therefore, to study the peculiar demands of the food product in question. The fragility of the product, its liability to damage the package and its tolerance levels of moisture and oxygen uptake are the most important considerations.

Fragility

Several products, especially fruit and vegetables, are so fragile that they require some protection against outside damage, but they may, in addition, be liable to damage by movement within the package. A can, which would

TABLE 6

Weight and Storage Space Data for AFD Products

Product	Approximate lb wet wt. [1] (edible portion) giving 1 lb dry	Bulk density (dry basis)		Packaging in A10 tall cans (603 × 904)[2]					Pounds edible product per lb packaging
				Net wt. per can		For 1 ton as eaten[3]			
		lb/cu. ft	g/ml	lb	oz	No. of cans	Net wt. lb	Gross wt. (lb)	Stowage space (cu. ft)	
1	2	3	4	5		6	7	8	9	10
Cooked Beef, slices[4]	2·9	19·8	0·32	—	—	—	772	—	—	—
Cooked Beef, minced	2·9	41·7	0·67	5	0	161	805	1,100	40·2	7·90
Cod Steaks, raw	4·8	8·3	0·13	1	0	477	477	1,352	119·2	2·56
Meat Chunks, raw	3·0	12·5	0·20	1	8	500	750	1,667	125	2·44
Beetroot Slices	7	10·4	0·17	1	4	256	320	789	64	4·78
Brussels Sprouts	8·5	10·4	0·17	1	4	210	263	648	52·5	5·82
Cabbage Shreds	13	8·3	0·13	1	0	172	172	487	43	7·11
Carrot Slices	10	10·4	0·17	1	4	179	224	507	44·8	7·91
Broad Beans	4·5	19·8	0·32	2	6	210	500	880	52·5	5·80
Green Beans	10·0	8·3	0·13	1	0	224	224	632	56	5·42
Peas	4·5	25·0	0·40	3	0	167	500	806	42	7·32
Potato Chips	5	16·7	0·27	2	0	224	448	859	56	5·45
Apple Dice	8	22·9	0·37	2	12	102	280	467	25·5	12·0
Apricots, skinned	6·5	8·3	0·13	1	0	345	345	978	86·2	3·54
Peaches, skinned	6·5	8·3	0·13	1	0	345	345	978	86·2	3·54
Plums	8·0	10·4	0·17	1	4	223	279	685	55·5	5·42
Raspberries	8·7	10·4	0·17	1	4	206	258	633	51·5	6·00
Beans in Tomato Sauce	4	25·0	0·40	3	0	187	560	903	46·8	6·53

NOTES

(1) Edible portion—i.e., after trimming; e.g., boned out meat, trimmed and cored cabbage, stoned plums, etc.
(2) Packing in 4 gal rectangular cans saves about 30 per cent space, 15–20 per cent weight, and 40 per cent steel, but uses 3 times as much tin, mainly in soldered seams.
(3) On basis of 100 per cent reconstitution; the figures are related to *one ton nett of the foodstuff, as eaten.*
(4) Not normally packed in cans.

afford protection from atmospheric pressure and external mechanical damage, could permit internal movement and abrasion, while a flexible container which does not afford the same protection against external damage, does at least reduce the freedom of movement of the contents. Mechanical protection can be improved greatly by placing a carton around the flexible package, and in this way lighter laminates can be employed without reducing the protection afforded to the contents.

For such products as would collapse under the pressure of the atmosphere, gas-packing at atmospheric or reduced pressure may be the solution; or support by a carton within the evacuated flexible pouch.

Rupture of Package

Many dehydrated products have corners and edges which are sharp enough to penetrate the package if enough pressure or rubbing is experienced. The surface abrasion brought about by such products is much greater if there is free movement within the package. As mentioned above, a flexible container adapts itself more readily to the shape of its contents than a rigid one, and this tends to restrict movement of the contents. The provision of an outer carton increases this restriction and lends support to the inner laminate. Vacuum packaging so accentuates this problem of rupture of the container that with certain products it is quite impossible to pack in this manner.

Moisture Tolerance

The amount of water absorption which can be tolerated by a product is determined by studying the relationship between its moisture content and its equilibrium relative humidity. In most cases excessive moisture uptake leads to serious non-enzymic browning before the critical level for microbiological growth is reached.

Oxygen Tolerance

This is a property which is peculiar to each food and has a very important bearing on the packaging required. It is essential, therefore, to know exactly how much oxygen can be absorbed by a food product without seriously impairing its acceptability over a long period. Only when this critical level is known can the minimum level of protective packaging be specified. Accelerated storage tests (i.e., for a short time at an abnormally high temperature) are always open to some doubt in interpretation and a more satisfactory method of discovering the oxygen tolerance of foods is to store them for their intended maximum storage life at different levels of oxygen concentration. Although taste-panel evaluation has weaknesses it is, nevertheless, a realistic assessment of quality changes in stored samples and can indicate the probable end of their acceptability.

Such a test is necessarily very long in duration and some acceleration might be desirable in spite of the danger of misinterpretation. In experiments of this kind at Aberdeen a period of 10 weeks storage at 25°C was used. Samples were packed at a number of oxygen levels between 0 per cent and 100 per cent oxygen in the head-space of the containers (8 oz cans). Each sample contained a standard weight of dehydrated food and, since its specific gravity was known, its volume could be calculated. The relationship between weight of oxygen and weight of food was calculated and the particular ratio at which

the product was just acceptable after 10 weeks at 25°C was regarded as the oxygen tolerance. It must be stressed that this was necessarily a short-term test and that it should be judged with caution.

The relationships for the seventeen representative food products examined are shown in Table 7.

TABLE 7

Food	Bulk Density *g/ml*	Specific Gravity	Oxygen Tolerance *mg* O_2*/g* dry food
Raw Beef Mince	0·25	1·33	1·0
Cooked Pork Cubes	0·20	1·37	0·1
Cooked Beef Slices	0·32	1·22	0·2
Minced Cook Ham	0·67	1·25	0·1
Minced Cooked Pork	0·67	1·24	0·1
Beef and Vegetable Stew	0·20	1·47	0·1
Spaghetti and Pork	0·20	1·66	0·1
Raw Cod Steaks	0·13	1·45	1·0
Cooked Cod Flakes	0·18	1·47	0·5
Potato Chips	0·27	1·78	5·0
Carrot Dice	0·15	1·21	0·2
Peas	0·40	1·48	1·0
French Beans	0·13	1·10	1·0
Broad Beans	0·32	1·65	0·7
Raspberries	0·17	1·73	0·2
Plums	0·17	1·39	0·3
Sago Pudding	0·13	1·89	0·4

Once the requirements of the package for a dehydrated product are known, packing materials which will fulfil these requirements may be sought. If the weight and bulk of a metal container can be tolerated there is little further concern since the protection afforded against oxygen and moisture penetration is as good as possible, and the greatest hazard is likely to be fragmentation within an only partly filled container. For large-scale (e.g., catering) packs the can is almost certainly the best container. If, however, a lightweight, close-fitting container is desired, it is necessary to determine the permeability of the various plastics and laminates which are available. Several methods are available for measuring moisture permeability, the simplest being the following:

(*a*) A specimen disc of the film, (approximately 50 sq. cm in area) is used to seal a cup containing a desiccant. The cup is then weighed periodically, giving weight increases from which the water vapour transmission rate (WVTR) is calculated. This is the standard test method described in the literature.[9]

(*b*) Sample packs of the plastic or laminate in question are filled with a desiccant and sealed. They are placed in a constant humidity chamber at 75 per cent RH and their increase in weight with storage time is noted. From this the WVTR is calculated.

The measurement of oxygen transmission is rather more difficult, but again many methods are available,[10] the one preferred in the work at Aberdeen being the pressure-increase technique.[11] All the methods using a virgin sample of the laminate or plastic merely provide an indication of the performance of the material, which is sufficient for screening a selection of possible

films. It is important to remember, however, that actual packages contain creases, folds and seals, which may considerably impair their performance. More practical tests of the made-up packages for oxygen transmission may be carried out by filling them with nitrogen, storing them in oxygen, and subsequently analysing the enclosed gas for oxygen. The results obtained even from such tests may not be universally applicable, and only when the exact size and shape of package is tested under closely simulated conditions of handling are the results likely to be relevant.

The results of tests on a variety of films and laminates (tested as sheets, not as packages) are listed in Table 8.

TABLE 8

Permeabilities of Typical Barrier Materials

Barrier	Permeability	
	Moisture[a]	Oxygen[b]
·001 in. MSAT/·003 in. Polythene	0·021	28
·001 in. MSAT/·001 in. Polythene	0·060	125
·0012 in. MXXT/·0017 in. Polythene	0·04	4
·0015 in. MXXT/·002 in. Polythene	0·010	3
·0015 in. MXXT/·003 in. Polythene	0·009	2
·0005 in. Melinex/·002 in. Polythene	0·205	55
·001 in. Melinex/·002 in. Polythene	0·180	39
27 lb GIP.[c] Paper/45 g[d] Saran	0·009	11
28 lb GIP. Paper/70 g Saran	0·002	2
28 lb GIP. Paper × 0·00035 in. Foil × 0·0008 in. Pliofilm	0·006	2
28 lb Paper × ·0005 in. Foil × ·0015 in. Pliofilm	0·001	1
·00035 in. Foil × 28 lb GIP. Paper × ·0015 in. Pliofilm	0·020	67
28 lb GIP. Paper × ·00035 in. Foil/·0015 in. Polythene	0·019	1
28 lb GIP. Paper/·0015 in. Polythene[e]/·00035 in. Foil/·0017 in. Polythene	0·003	2
·00035 in. Foil × 28 lb GIP. Paper/·002 in. Polythene	0·740	275
·001 in. Acetate × ·001 in. Foil/·0012 in. Polythene	0·005	1
·001 in. Acetate × ·001 in. Foil × ·0015 in. Pliofilm	0·005	4
28 lb GIP. Paper × ·00035 in. Foil/8·8 g H.S.C.	0·018	1
30 lb GIP. Paper × ·0005 in. Foil/10 g H.S.C.	0·001	1
30 lb GIP. Paper × ·00075 in. Foil/10 g H.S.C.	0·001	1
28 lb GIP. Paper/·0006 in. Polythene/·0005 in. Foil/4 g H.S.C.	0·008	1
·0009 in. Acetate × ·0005 in. Foil/4·5 g H.S.C.	0·001	2
·001 in. Acetate × ·001 in. Foil/8 g H.S.C.	0·001	3
·001 in. MSAT × ·00035 in. Foil/5·5 g H.S.C.	0·002	2
·0035 in. High Density Polythene	0·082	1,360[f]
·0007 in. Saran (Polyvinylidene Chloride Copolymer)	0·017	6[f]
·005 in. Polyvinyl Chloride (Plasticized) (Food Grade)	0·094	600[f]

Footnotes

(a) Permeability units are: g H_2O/100 sq. in. per day at 100 per cent RH and 25° C
(b) Permeability units are: c.c. oxygen/metre^2 per day at 1 atm. partial pressure difference and 75 per cent RH and 20° C
(c) Paper weight units are: lb/50 sheets (24 in. × 36 in.)
(d) Coating weight units are: g/m^2
(e) Polythene used as the adhesive between paper and foil
(f) Oxygen Permeability units corrected to ·001 in. thickness of film

NOTE

The measurements given in Table 8 were made at the Research Establishment on sheets of films and laminates, not on packages. It must be recognized that there may be considerable variation in the permeability characteristics of laminates made to nominally the same specification.

The gas-tight seals required for the closure of packages of dehydrated foods demand the use of thermoplastic films (such as polyethylene, rubber hydrochloride, polyvinylidene chloride and its co-polymers) or a heat seal coating on the inner ply, but in general plastic films do not have sufficiently good barrier properties to be used alone and another material must be added to improve their protective qualities. This barrier material may be aluminium foil, polyester, cellulose or coated cellulose films or other similar materials. The resulting laminate may now be an effective moisture and gas barrier but may not have sufficient mechanical strength to withstand handling. This applies particularly where aluminium foil is concerned since it is very susceptible to tearing and puncturing. The addition of a third layer of film to increase this strength may be advisable. Such a material might be paper or another plastic film; so we get a triple laminate.

Whatever laminate is used, it must be fabricated into the shape of the final container. This final shape is important; the familiar two-dimensional sachet (as used widely for dried soups) is ideal for small quantities of contents but is wasteful of storage space when the amount of contents is increased. The practicable limit of thickness of such packs is about $\frac{3}{8}$ in.; beyond this thickness a three-dimensional pack is more suitable since it is under less internal strain and the free space is reduced. A three-dimensional flexible pack is, however, more susceptible to mechanical damage and requires protection, which is most easily afforded by a paper-board carton with the flexible package forming a liner which can be sealed gas-tight. Some of the advantages of such a package have already been described; other advantages to a manufacturer producing a variety of dehydrated food are that a uniform outer carton could be used to identify and relate the contents while the liner could be varied in composition to be the most suitable for each particular product. With our present state of knowledge this type of pack seems to be the most suitable for small retail-size units.

Packaging Operations

It has already been pointed out that protection of the contents from oxidation is one of the most important functions of the package. It is therefore of prime importance to reduce the oxygen level in a container to the minimum possible. The three main packaging methods which are used to achieve this are: vacuum packing, inert gas-packing, compression.

Besides these, the addition of 'oxygen scavengers' which remove oxygen from within the pack has been practised and can be valuable when the maximum storage life is necessary.

Vacuum packing

AFD foods are apt to be fragile and the atmospheric pressure on an evacuated flexible package may be sufficient to crush the contents. In the case of foods which can be compressed or partly compressed without serious damage, blocks can be formed before packing and these have the mechanical strength to withstand the pressure differential. Compression also has the advantage of giving a regularly shaped product, so that the pressure on the wrap is absorbed uniformly across flat surfaces. This type of vacuum packing has been successfully used for military rations, but for most commercial uses compression

may not be suitable. Vacuum packing of rough irregular material places too great a strain on the package and contents to be of much practical value.

Vacuum packing has been used to some extent with powdered or small grain materials in which the particles are pressed together by the atmosphere to give a rigid mass of relatively high density. Such a package lacks mechanical strength and so is liable to damage.

Where vacuum packing is required it is important to have the pack fit the contents closely before evacuation, otherwise the excess packaging material suffers sharp and haphazard folding and is liable to damage. When rectangular blocks are to be packed in this way the problem has been overcome by neatly folding and shaping the pouch over a wooden form before inserting the contents as described below.

Considerable experience of this form of packaging was gained at the Experimental Factory, with $2\frac{1}{2}$ oz meat bars, which are typical compressed products, ideally suited to vacuum packing in flexible containers.

The blocks, measuring $5{\cdot}6 \times 4{\cdot}6 \times 2{\cdot}5$ cm, were vacuum packed in pouches of a laminate of ·0015 in. Pliofilm + ·018 mm aluminium foil + ·001 in. Melinex polyester. The laminate was converted to sachet-type pouches, $\frac{1}{4}$ in. fin-sealed on three sides and open on the fourth. The internal dimensions of the pouches were 110×90 mm.

Since it was important to have a closely fitting pack the pouch was formed on a mandrel of cross-section $5{\cdot}6 \times 2{\cdot}5$ cm. It was then easy to insert the meat bar into the pouch and fold the open end so that it was again smooth and flat, and ready for sealing. It was essential that the sealed area should be flat and smooth since the presence of wrinkles resulted in subsequent failure of the seal.

The vacuum sealer was specially constructed but was of a type which is readily available commercially. It consisted of a tilted rectangular metal cabinet hinged at the rear base edge so that the whole top and sides could be raised to expose the flat base, on which the packs were placed. A vacuum line was fitted to this cabinet and provision made for packing in either nitrogen or air (see Fig. 5). The jaws were located at the rear of the cabinet and were of the permanently heated type. The top jaw was fixed, and the lower one was raised for sealing by a piston operated by the difference in pressure between cabinet and atmosphere. An electronic timer and solenoid valve controlled the sealing operation so that the evacuation cycle could be varied in length as required.

The packed meat bars were laid four at a time on the platform with the flattened mouths of the pouches protruding between the sealing jaws. The lid with its rubber gasket was closed, and the cabinet exhausted to a pressure of approximately 1·0 mm Hg. At this level the jaws were closed for a period of approximately 3 seconds and the pouches sealed. The cabinet was now opened and the pouches removed from between the open jaws. The top of the pouch was folded to produce a neat rectangular block-shaped pack.

Testing. The pressure within a vacuum-packed container was measured by a simple non-destructive test. The package was placed in a small decompression chamber fitted with a heavy glass window and a vacuum gauge. The pressure was reduced and the point noted when the walls of the package just began to lift away from the contents. At this point the pressure in the chamber equalled that within the pack and could be read from the gauge.

It was found that in most cases the pressure inside a pack of dehydrated food tended to increase slightly over a period of 1–2 days, due to slow desorbtion of gases from the food. Nevertheless it was possible to get packs with internal pressures of 2 to 6 in. Hg using this technique.

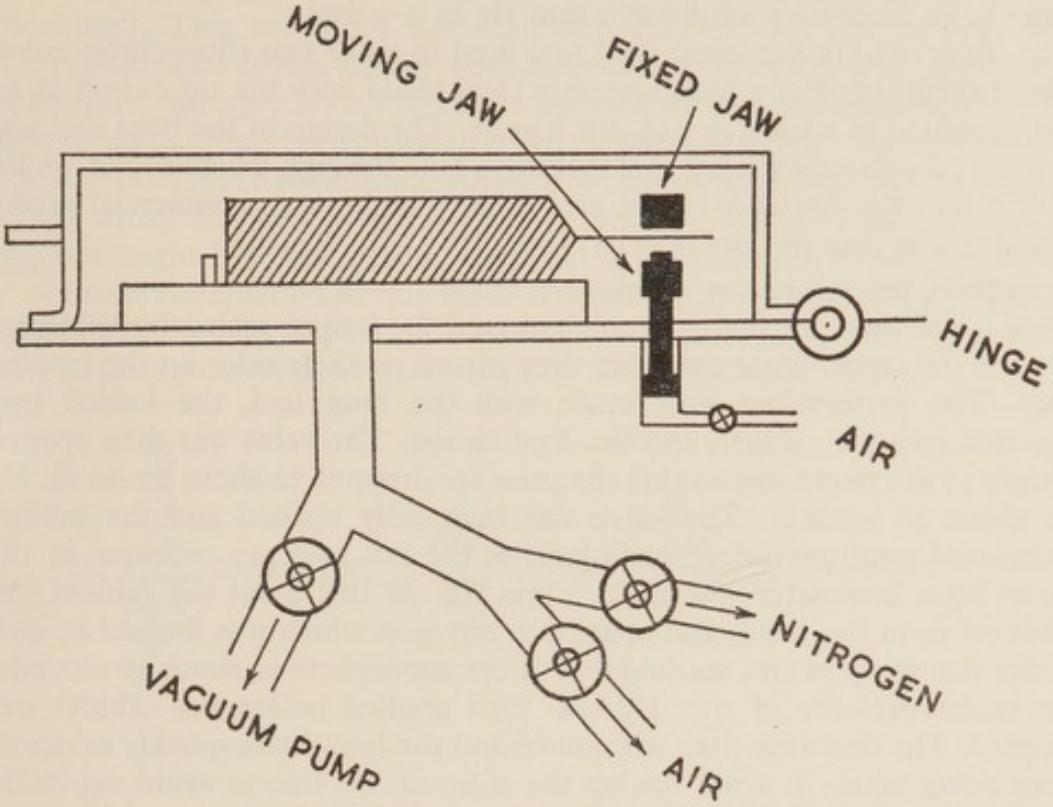


FIG. 5. *Systematic cross-section of vacuum sealer*

Inert gas packing

Air could be removed from the pack and replaced by an inert gas such as nitrogen or carbon dioxide. Since the pressure of inert gas within the pack could be adjusted to that of the outside atmosphere there was then no strain across the barrier walls as with vacuum packing. This permitted the use of either rigid metal cans or flexible packages as gas-packed containers.

Metal cans. The gas packing of metal cans is a well-established process, and in its simplest form consists of piercing a hole in a filled sealed can and placing it in a cabinet which is then evacuated. This removes the air from the can and, when a sufficiently low pressure has been reached, the inert gas is leaked in until atmospheric pressure is attained once more. The cabinet is then opened, the can removed, and the hole sealed by soldering.

Such a method was used at the Research Establishment for the gas packing of 4-gal rectangular tins of normal commercial type. These had circular single-friction lids fitted to the top end. Around the edge of these lids was a recess to permit the soldering-on of a tin tagger-plate to provide a gas-tight closure. The packing cabinet was a strong rectangular steel box, open at one end and of such a size that it held four 4-gal tins. The open end was closed by a door, hinged at the top and sealed with a rubber gasket. On one side wall of the cabinet was a flange bearing a lubricated 2-way vacuum tight cock, which was connected to a vacuum pump and a nitrogen cylinder. An adaptor

connected to a compound dial gauge, reading from 30 in. vacuum to 20 lb pressure, was let into the top of the cabinet. Also connected to this adaptor was a mercury-in-glass short-form barometer (calibrated in mm) for registering low pressures.

The pump was of a motor-driven rotary design capable of exhausting the cabinet to an absolute pressure of 1 mm Hg in 2–3 min.

The 'brog' tool (a hardened steel tool used to make two slits—'brog-holes' in the tin) consisted of a wedge-section steel blade with the tip cut off at an angle, mounted in a wooden pad-saw handle. The design of the brog tool was important; a correctly shaped tool making a hole through which solder would not drip into the contents of the can. The nitrogen was commercial grade, containing less than 0·5 per cent oxygen.

The packing procedure was as follows:

> Four 4-gal tins containing the dehydrated food were sealed by soldering around the tagger plate and were then placed on their sides on the loading tray. The perforations were made with the brog tool, the loaded tray inserted into the cabinet, and the door closed. The valve was then opened slowly to the pump line so that the pressure dropped to about 17–20 in. Hg in about 20 seconds. The valve was then fully opened and the cabinet exhausted until the difference in level of the two mercury columns in the short form barometer was about 4 mm Hg. At this point the cabinet was shut off from the pump and opened to nitrogen which was flushed at such a rate that the pressure reached that of the atmosphere in about 40 seconds; an excess pressure of 2 in. Hg was then applied before the cabinet was opened. The tins were then withdrawn and the brog-holes quickly soldered, care being taken to avoid flexing the sides of the tins to avoid expulsion of nitrogen and introduction of air.

A generally similar technique was used for cylindrical cans, except that these were stood upright on the tray and required only one brog-hole in the flat end of the can.

Testing for leaks. Cylindrical metal cans with seamed lids were usually found to be free from leaks but the large 4-gal tin had soldered side and end seams which were apt to develop faults, and it was therefore advisable to test these cans for leakage. This was done by inflating to about 2 lb/sq. in. above atmospheric pressure with nitrogen and immersing in a tank of water, when any leak was revealed by a stream of bubbles. Inflation was done by means of a special testing tool (which could, incidentally, also be used for extracting samples of the internal gas).

This tool consisted of a rubber-mounted probe for piercing the tin and a device for holding the probe in position (Fig. 6). After the test the probe perforation and any other leaks were sealed by soldering. As a final precaution, for long-term storage, the soldered seams and any other exposed tinplate were painted with a bituminous emulsion in order to prevent corrosion and to seal any small leaks that might have developed during subsequent handling of the cans.

Flexible packages

Gas-packing of flexible containers could be carried out quite easily by a number of methods. An obvious one was to use equipment and procedures similar to those used for vacuum packing, except that, at the end of the ex-

haustion cycle, the vacuum was broken with the inert gas before the seal was made. This entailed an operation time about twice that of vacuum packing, owing to the slow infusion of the inert gas. Another method was simpler and consisted of blowing a jet of inert gas into the bottom of the filled package and displacing the air surrounding the product before sealing (so-called 'flushing'). This technique does not remove much of the air held within the product and as has already been pointed out, in AFD foods this is a very substantial volume. If, however, at the end of the dehydration process the drying cabinet had been released to atmospheric pressure with the inert gas, and if subsequent handling and packing of the food had been rapid, the amount of air which diffused into the product was not too great and this flushing method might have been quite adequate for normal commercial purposes.

A further adaptation of this method might be to use a pressure of inert gas a little less than atmospheric, so getting the compacting or close-fitting effect of a flexible pack without overdue strain on the laminate.

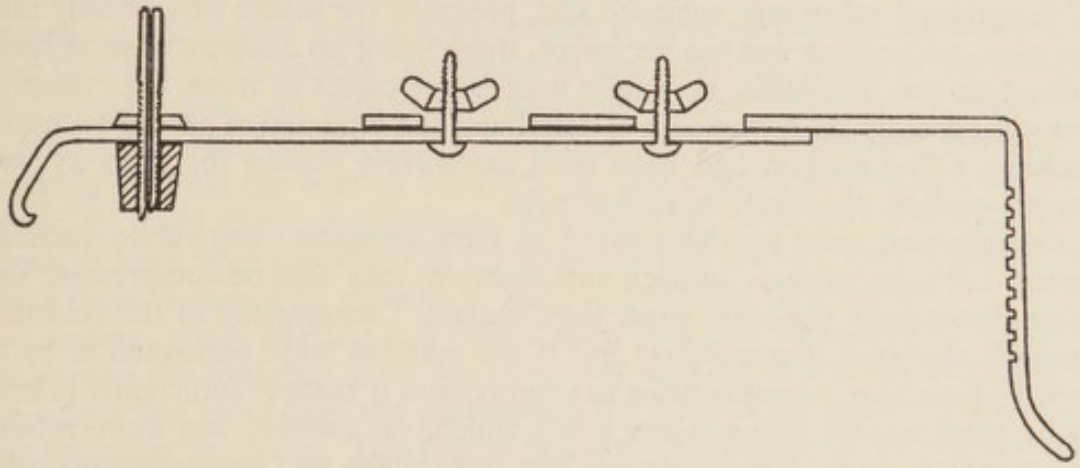


FIG. 6. *Testing and sampling tool*

Testing. When a leak occurs in a vacuum pack it is easily noticed since the tightness of the package walls is relaxed, but if a leak occurs in a gas pack there is no easy way of detecting it. The most effective method of control was to sample some of the packs and examine each of these for oxygen content by gas analysis. Sampling from the flexible container was carried out by sticking a soft rubber patch (a bicycle patch) to a wall of the pack and withdrawing a sample of gas via a hypodermic needle thrust through this and connected to a gas analysis apparatus. If the pack was under partial-vacuum the same general method could be employed after an initial injection of a known amount of nitrogen to dilute the gas within the pack before extraction.

A simpler test for leaks in a flexible container was to fasten a rubber patch to one of the walls of the pack, and inject nitrogen or carbon dioxide by means of a hypodermic needle so that the pack was inflated to a little over atmospheric pressure. If the pack were now immersed in a tank of water, a stream of gas bubbles revealed any leaks. It may be noted that in this test the pack was under positive pressure so that the product was not in contact with the water and could be repacked if required.

Experience has shown that very small leaks were sometimes to be found around the final seal at the top of the pack, too small to be detected by this

simple test. Confirmation of this could be made by removing the contents and pouring a small quantity of an ether solution of dye into the pack and allowing to stand for a few minutes. This solution of low viscosity quickly penetrated any slight leak and revealed it by staining.

Compression. The ratio of free space to product volume in a container of dehydrated food is often extremely high; thus in air packs the proportion of oxygen to food is very considerable. Removal of the oxygen from this space has already been discussed as one solution of the problem. Another approach is to increase the volume of food in the container and so reduce the available free space. This can be done by compressing the food to a higher density so that the free space is small enough to make gas packing unnecessary.

Certain meat products with relatively high fat contents were found to compress fairly easily if they were pre-cooled to about -5°C (to avoid expression of oil), and pressures of up to 1,500 lb/sq. in. were employed either to produce blocks for insertion into cans or to press directly into cans held in a reinforced clamp mechanism. In these ways reduction in bulk to about one-third of the original value was achieved and, provided the blocks fitted neatly into the cans, gas packing was not necessary, there being no headspace; some fragmentation was inevitable, especially with larger pieces of meat, e.g., chunks, but was offset by the advantages of debulking and omission of gas- or vacuum-packing. (This method had been used extensively during the 1939–45 war for pre-cooked, air-dried, minced meats.)

Compression need not be limited to fatty products. Vegetables such as carrot, cabbage, turnips, spinach and beetroot, may also be compressed, the more successfully the more sugar they contain. Compression of the cold dry material shattered it completely but if the material were preheated at 77°C (170°F) for about 1 minute prior to compression it became sufficiently plastic to be compressible into a dense block which, on cooling, was quite robust and handled easily. Fragmentation was only slight and reconstitution only slightly retarded, these being minor considerations against the advantages of bulk reduction and absence of gas packing. For institutional use and large-scale catering vegetable compression is attractive if only for considerations of saving of storage space; e.g., 300 lb of uncompressed dehydrated cabbage gas-packed in 4-gal cans required 60 cans and occupied 29·6 cu. ft; when the cabbage was compressed and packed as $\frac{1}{2}$ lb circular blocks in A10 tall cans, 40 of these much smaller cans were required, occupying only 7·5 cu. ft of storage space and requiring no gas packing. (This quantity of cabbage would provide something like ten thousand 3 oz servings.)

CHAPTER VII

Economics of the process

UNTIL recently the high cost of freeze-drying has been regarded as prohibiting its application to foodstuffs, and it is only with the acceleration of the process brought about by the developments here reported that it has appeared to be economically feasible. Even so freeze-drying remains, and is likely to continue to be, a costly procedure, whose economic prospects must depend on a demand for the specific advantages of the products—convenience in handling, high culinary quality, light weight, some reduction in bulk, and no necessity for refrigerated storage. Freeze-dried foods will naturally be compared with quick frozen foods which require special storage equipment and facilities at the factory, in transit, at the receiving depot, at the retailers and in the home; and with canned foods which are bulky for the amount of actual food value contained and which require a considerable amount of packing material per unit of foodstuff. In this chapter no attempt has been made to study these comparisons, but essential basic data for such AFD foods as have been processed in the Experimental Factory are presented, and from these it should be possible to make appropriate calculations.

Canning, quick freezing and dehydration (whether by hot-air or by AFD) all require the foodstuff to be pre-processed to more or less the same degree. In only a few cases does the preparation for AFD differ markedly from that for the other processes. The special features of AFD lie in the actual drying process and the subsequent packaging and storage. The basic data to be considered here will therefore be confined to these aspects and will include the following: capital cost of drying units; operating cost of drying units (steam and power and cooling water); throughput of drying units; cost of packaging equipment; cost of packing materials; throughput of packaging equipment; storage requirements; labour. It must be emphasized, however, that the limited data presented in this chapter have been obtained from operation of the Mark I AFD plant and refer *only* to this type of equipment; developments in design are rapid and by the time this Report appears it may well be that freeze-drying plants will have appeared with greater shelf area per unit of initial cost, or with the ability to carry heavier tray loading (but still with an economic drying time) or with more economical vapour extraction.

COST OF DRYING UNITS

It is doubtful whether a single unit of the capacity of the Mark I cabinet could be operated as an economic proposition except as a Market Research Unit; an installation of four such units, although still small by normal food processing standards, has been thought reasonable as giving a better balance between throughput and overheads. For simplicity, however, and also because

all the relevant data have been obtained from a single unit, the following discussion of costs will be in terms of a single cabinet which is assumed to be one member of a 4-unit installation.

The Mark I cabinet at Aberdeen, with 240 sq. ft of tray area, cost £9,000 and the steam ejector vapour extraction equipment £4,500. Part cost of a general purpose boiler (suitable for a 4-unit installation) might be as much as £3,000, and total installation cost £2,000. The capital investment for the drying stage is therefore about £18,500. In a multi-unit system a number of cabinets could be arranged to share a common extraction system, somewhat reducing the capital cost of this item, but for the present purpose the figures above will be used, any such reduction being a bonus.

Depreciation

A figure of 10 per cent per annum is used in the calculations—this is thought to be reasonable as the machinery described above has few moving parts and will not wear out quickly—obsolescence of design will be a more important factor than wear and tear. On this basis, then, depreciation will be £1,850 per annum.

Interest on Capital Invested

Practice varies from one firm to another; for the present purpose interest is taken as 7 per cent on half the capital invested, i.e., £650 per annum.

Operating Cost

The steam consumption averaged about 1,100 lb per hr, and a cost of 10*s*. per 1,000 lb of steam is assumed, but it should be pointed out that this figure is applicable only to an installation where condensate is returned to the boiler. With direct mixing of steam and cooling water as is employed in a steam jet system, some loss of overall thermal efficiency is unavoidable but the use of exhaust steam discharge from the fourth stage ejector for feed-water heating would partially compensate for this. It should also be noted that for about 10 min at an early stage in the AFD cycle there was a maximum demand per cabinet at the rate of about 4,000 lb per hr; this has been taken into account in the calculation of the average steam consumption, and in the calculation of the size of boiler required, since no accumulator is assumed.

The consumption of electricity was about 26·5 kW per hr with water being pumped from a river. If no pumping up of water is required, but only circulation, the figure is reduced to 19 kW. In the latter case, however, it may be necessary to purchase the cooling water, some 11,500 gal per hr, though the consumption of water would be very greatly reduced by the inclusion of a recirculatory cooling system, incorporating, for example, a cooling tower. The estimated cost of the latter for a plant with four cabinets is about £3,000. For the calculation which follows it has been assumed that there will be river water available, and that consumption of electricity will be 26·5 kW per hr at $1\frac{3}{4}d$. per kW-hr.

For many commodities blast-freezing before drying is necessary. The cost of this is estimated at approximately $\frac{1}{2}d$. per pound fresh: this has *not* been included in any of the tables.

Throughput of Drying Units

Each cabinet held 30 trays measuring 3 ft $3\frac{1}{8}$ in. × 2 ft $6\frac{3}{8}$ in. (8·2 sq. ft). This gave a loading area of approximately 240 sq. ft. Different commodities require different densities of loading on the trays and different drying times. Thus the rate of input was not the same for all foods and experimental results for a range of foods (but by no means an exhaustive list) indicated that tray loads, loads per charge and drying cycles (i.e., actual drying time plus $\frac{1}{2}$ hr for removing trays of dried material from the plant and inserting a fresh charge) were of the order indicated in columns 6, 7 and 10 of Table 9, and these are the figures that have been used when the drying cost per pound of prepared foodstuff was calculated. It must be emphasized, however, that many of these figures may be subject to considerable revision, and any change is likely to be towards greater throughput; improvements in the design of plant should increase the capacity and preparation and drying techniques could be further developed.

In Table 9, column 2 indicates the approximate overall drying ratios for a number of foods, i.e., the number of pounds of raw, unprepared foodstuff (vegetables or fruit as received from the farm, meat in the form indicated—guns, sides, etc.), which yielded 1 lb of dehydrated product. There could be a considerable range in these figures, as the raw materials vary greatly in dry matter content (e.g., at the extreme, cabbage may vary in dry matter content from 6 per cent to 14 per cent), and the weight required to give 1 lb of dehydrated product differed correspondingly; the range that was encountered in practice is shown in column 3. The ratios of prepared foodstuff entering the drying cabinet to the weight of dehydrated product are shown in column 4; again there is a wide range in these figures, as indicated in column 5. Column 8 in Table 9 is related to the foregoing data, as it shows the average output expected from one charge of the dryer; the range is shown in column 9.

The information given may be used in calculating the cost in terms of prepared material entering the plant or in terms of the dehydrated product, but it is sometimes desired to relate costs to the foods as served on the plate. The dehydrated product contains all the food which was present before drying, e.g., in Table 9, column 4, it is shown that 1 lb of dehydrated Brussels sprouts is obtained from 8·3 lb of fresh (prepared) sprouts. After reconstitution and cooking, 1 lb of dehydrated sprouts yields material equivalent to 8·3 lb of fresh, cooked sprouts. A complication does arise, however, because in most cases dehydrated foods do not, when reconstituted, take up all the water they lost during drying. In this particular case 1 lb of dehydrated sprouts produces only about 6 lb of reconstituted material, yet this 6 lb contains the same food as the original 8·3 lb, but less water. It is left to choice whether costs should be related to the true food material present, i.e., on the fresh equivalent, as if 100 per cent reconstitution were assumed, or on the *actual* weight of the reconstituted portion. Both these quantities can be derived from Table 9: the fresh equivalent from column 4 and the actual reconstitution ratios from column 11.

Packaging

So little experience was gained in the packaging of AFD products in small consumer units that all that can be offered is information on the costs of packing in large cans for institutional feeding. It is assumed that these would

TABLE 9

Basic Data for Accelerated Freeze-drying

Commodity	Overall drying ratio (lb fresh giving 1 lb dehydrated product)		Prepared/dry ratio (lb prepared material giving 1 lb dehydrated product)		Plant load[1]		Output dehydrated product per charge (lb)		Approx. drying cycle[2] (hr)	Reconstitution ratio (lb reconstituted from 1 lb dehydrated)
1	2 Normal	3 Range	4 Normal	5 Range	6 lb/sq. ft	7 Total charge	8 Normal	9 Range	10	11
Broad Beans	15·4[3]	—	4·5	4·0–5·0	1·6	370	82	75–90	9¼	4·5
French Beans	11·7	10·0–12·5	10·0	9·1–10·5	2	480	48	45–53	9½	8·5
Brussels Sprouts	10·0	9·2–14·7	8·3	8·0–10·6	2	480	58	45–60	8½	6
Cabbage: Shreds	18·2	15·6–26·4	13·2	8·1–20·8	2	480	26	23–59	8	8–10
Carrots: Dice	13·3	13·1–16·4	10·0	8·6–11·1	2·5	600	60	54–70	10	7·5
Cauliflowers: Curds	33·3[4]	33·3–41·7	11·0	10·3–11·1	2	480	44	43–47	10	7·5–8·0
Peas	5·5[5]	5·0–6·2	4·5[6]	4·2–5·0	2	480	106	96–114	9½	4·5
Potatoes: Chips ½ × ½	8·0	6·6–9·9	5·0	4·4–6·4	2·5	600	120	94–136	9½	4·5–5·0
Swedes: Dice	16·6	—	10·5	—	2·5	600	57	—	10	6·5
Apples: Citrus and sugar	—	—	3·0	—	2·25	540	180	—	8½	3·0
Apple: Dice	12·5	11·2–12·6	8·0	7·6–9·7	2·0	480	60	62–63	9½	6·0
Apricots	10·0	—	6·7	5·9–7·9	2·0	480	80	68–91	8½	6·0[7]
Black Currants	6·1	5·7–6·5	5·7	5·0–6·0	1·5	360	63·5	55–70	8½	6·5[7]
Peaches	10·0	—	6·7	5·7–7·9	2·0	480	80	68–95	8¼	6·0[7]
Plums	10·0	—	8·0	6·7–8·5	1·1	270	34	—	7	6·0[7]
Raspberries	8·7	7·7–10·0	8·5	7·7–10·0	1·6	390	47	48–62	9½	6·5[7]
Strawberries	6·5	—	15·0	—	1·6	390	25	—	10	7·5[7]
Beef, sliced, cooked (guns)	7·7[8]	—	2·9	—	3·4	800	276	—	7½	2·25
Beef, sliced or cubed, raw (guns)	5·3	—	3·6	—	2·25	540	150	—	6½	2·25
Lamb, sliced, cooked (whole carcasses)	8·0	—	2·9	—	3·4	800	276	—	7½	2·25
Lamb, sliced or cubed, raw (whole carcasses)	6·7	—	2·9	—	2·25	540	186	—	8	2·25

TABLE 9—continued

Commodity	Overall drying ratio (lb fresh giving 1 lb dehydrated product)		Prepared/dry ratio (lb prepared material giving 1 lb dehydrated product)		Plant load[1]		Output dehydrated product per charge (lb)		Approx. drying cycle[2] (hr)	Reconstitution ratio (lb reconstituted from 1 lb deyhdrated)
1	2 Normal	3 Range	4 Normal	5 Range	6 lb/sq. ft	7 Total charge	8 Normal	9 Range	10	11
Pork, sliced, cooked (sides)	11·6	—	2·7	—	2·25	540	200	—	6½	2·25
Pork, sliced or cubed, raw (sides)	8·4	8·3–8·6	3·2	3·2–3·3	3·0	720	225	218–225	9½	2·25
Meats, minced, cooked	4·4[9]	—	2·7[10]	—	3·0	720	266[10]	—	7½	2·5
Cod steaks (bone in)	9·1[11]	—	5·1	—	2·5	600	118	—	8	3·25

NOTES:

1. The Mark I plant has 30 trays each approximately 8 sq. ft in area, giving total tray area approx. 240 sq. ft.
2. Actual drying time + ½ hr for loading and unloading cabinet.
3. In the pod.
4. Preparation losses may mount to as much as 75 per cent of the material received at the factory. In commercial practice presumably payment would be made on basis of heads only.
5. After vining.
6. Cooked: Dry ratios. There is a substantial loss in weight during the period of steam cooking before dehydration.
7. This weight includes the weight of syrup in which the fruit is served.
8. Very little fat is left on meat prepared for the AFD process.
9. Bone in weight (guns) giving 1 lb adjusted product (see note 10).
10. When adjusted to 40 per cent fat and 7·5 per cent moisture content (as required for meat bar) prepared to dry ratio becomes 2 : 1 and equivalent output per charge becomes 360 lb.
11. Head on, bone-in.

be A10 tall round open-top cans ($6\frac{3}{16} \times 9\frac{1}{4}$ in.). Each drying unit would provide enough to fill from 30 to 130 cans per batch, according to commodity, or, if a 4-unit plant is considered, 120 to 520 cans per 8 hr shift. Packing would be direct from the trays into the cans and seaming would follow at once. A single seamer will easily handle these quantities, and is normally hired at about £10 per annum. On the assumption that packing in nitrogen would follow immediately (as would be the best practice), this output would be handled by one inert gas unit holding 12 cans per cycle. These units are also normally hired, and would cost about £29 per annum. The cans cost approximately 1*s.* 3*d.* each, labels $\frac{1}{2}$*d.* each and cartons for 6 cans 1*s.* 11*d.*, giving a total cost per 6 cans of 9*s.* 5*d.*

Packing densities

Closely relevant to the above is the packing density of the various products. Packing density is also important in calculating the cost of storage and transport of the finished material. Packaging data are given in Chapter VI, Table 6, which indicated the weight of fresh food, as trimmed for eating, giving 1 lb of dehydrated food (column 2) the bulk density of the dehydrated foods in pounds per cubic foot (column 3), and the net weight of dehydrated food per A10 tall can (column 5). From these figures the weight of dehydrated food giving 1 ton of edible foodstuff has been calculated, on the basis of 100 per cent reconstitution (column 7) and the number of cans required to hold this quantity of food (column 6). In columns 8 and 9 the gross weight and volume of this amount of packed food are shown, and in column 10 an indication is given of the weight of food contained in 1 lb of packing material—a figure useful for comparison with the requirements of packaging materials for normal canned foods (as distinct from dehydrated foods packed in cans).

Labour

Assuming a plant with 4 cabinets operating on a cycle of 8 hr, i.e., $7\frac{1}{2}$ hr actual drying and 30 min loading and unloading: this would give one batch of dried product every 2 hr. Labour requirements are *estimated* as the following (based on the Experimental Factory operation, with logical extensions):

(a) Actual plant operation: 1 man continuously plus 1 man for 10 min at the start of each drying period.

(b) Loading and unloading trays into and from the plant: 2 men for 30 min—the same 2 men as (a).

(c) Loading trays with the prepared food: 2 women. They would load at the rate of 900 lb per hour for 1 shift, which is three times the plant capacity. Loaded trays in excess of the plant's requirements would go into the chill room for use in the other two shifts.

(d) Unloading the dehydrated product from the trays: 3 operators, 2 actually putting the food into cans and the third seaming the cans. This operation should be carried out as quickly as possible and should not require more than 15–30 min per batch. The number of cans would be between 30 and 130 according to commodity.

(e) Gas packing: 1 man—the second plant operator who is available for 1 hr 20 min in each 2 hr. period. In an 8 hr shift he could probably be available for only three such periods owing to meal breaks, etc.

(f) The 3 operatives from (d) will therefore be available for other duties for about $1\frac{1}{2}$ hr in each 8 hr shift. These would include labelling and cartoning cans, porterage, washing trays, etc.

(g) The total labour force per shift for drying and packaging in this manner would therefore be 5 men for 4 cabinets plus 2 women loading the trays at the end of the pre-processing line.

Use of the basic data

From the information presented above it is possible to estimate the costs of drying a number of foods, and the costs involved in packaging. An example of the type of calculation, as applied to peas, is given below.

Capital costs	£
Cabinet	9,000
Extraction equipment (steam ejector type)	4,500
Boiler (proportion)	3,000
Installation, including water supply	2,000
	18,500

Running costs (per day, assuming 250 days operation per year)

	£	*s.*
Depreciation at 10 per cent p.a.	7	8
Interest at 7 per cent on ½ capital	2	12
Electricity 27 kW for 23 hr per day including water pumping at 1¾*d.* per kWH	4	11
Steam 1,100 lb/hr for 23 hr per day at 10*s.* per 1000 lb	12	13
Operator's wages (assuming he looks after 4 cabinets)	2	2
Maintenance (approximately)	3	15
	33	1

From Table 9 it is seen that the tray load for peas is 480 lb and the drying cycle 9½ hr.

Thus the input of prepared peas per drying cabinet per day is $(480 \times 24)/9{\cdot}5$ = 1,210 lb, and since the drying ratio for peas is 4·5 to 1 the 1,210 lb of peas entering the cabinet will yield 1,210/4·5 = 269 lb of AFD peas (approximately).

Relating the input and output figures to the running costs of the cabinet, we have:

Cost of drying stage
= 6·5*d.* per pound of prepared peas entering the drying cabinet, *or*
29·5*d.* per pound dehydrated.

Similar calculations may be made for other foodstuffs using the data in Table 9; approximate figures for certain items are shown in Table 10.

Comparison of Cost of Hot-air Drying and AFD

The most obvious comparison which will be made is between the new freeze-dried products and their already established air-dried counterparts, and it will be pointed out that the cost of the drying stage of accelerated freeze-drying very greatly exceeds that of hot-air drying, by a factor ranging from six- to ten-fold. It must be borne in mind, however, that the drying process is only one stage in a series of operations, and that increasing the cost of this stage will not increase the price of the finished product in anything like the same proportion.

Consider the case of peas. The selling price of the best grade of air-dried peas in catering size packs is 96*d.* per pound. The cost of the drying stage alone is approximately 5*d.* per pound, and it is shown above that the corresponding cost for AFD peas is about 29½*d.* Using this figure brings the potential selling price to 120*d.* This does not allow for the additional cost of packaging, which is estimated at 7½*d.* per pound, giving a total of 128*d.* per pound. This is a substantial increase on the 96*d.* per pound for air-dried peas, but since 1 lb of AFD peas will give approximately 4½ lb on the plate, the cost per

pound as eaten is only 28½*d.*; and the difference between, for example, 4 oz portions on the plate might only be the difference between 6*d.* and 7*d.*

TABLE 10

Approximate costs of the drying stage for AFD foods

Commodity	Approximate cost of AFD drying stage (*d. per lb*)	
	Prepared weight entering dryer	AFD product
Broad Beans	8·3	37·5
French Beans	6·5	65·0
Brussels Sprouts	5·8	48·5
Cabbage: Shreds	5·5	72·5
Carrots: Dice	5·5	55·0
Cauliflower Curds	6·8	75·0
Peas	6·5	29·5
Potato Chips (French fries)	5·3	24·0
Swedes: Dice	5·5	58·0
Apples: Citrus and sugar	5·1	15·0
Apples: Dice	6·5	52·0
Apricots	5·9	59·0
Black Currants	7·8	48·0
Peaches	5·9	59·0
Plums	8·5	85·0
Raspberries	8·0	68·0
Strawberries	8·5	55·0
Beef, sliced, cooked	3·1	8·5
Beef, sliced or cubed (raw)	4·0	14·5
Lamb, sliced, cooked	3·1	8·5
Lamb, sliced or cubed (raw)	4·9	14·0
Pork, sliced, cooked	4·0	11·0
Pork, sliced or cubed (raw)	4·9	15·5
Meats, minced, cooked	3·5	9·5
Cod Steaks	5·5	26·5

DIRECT COSTING

Since there is no commercial production of AFD foods at the time of writing, there is no information available about costs of any stage other than that of drying. It has already been pointed out, however, that the processes prior to dehydration are basically similar to those before quick freezing, and an estimate can be made of the total cost of production of an AFD product using available figures for this process. The following costs have been estimated in such a manner for raspberries:

	FRESH BASIS *d. per lb*	DRY BASIS *d. per lb*
Raw material cost	15·8	134·0
Transport of raw material	0·5	4·3
Preparation	2·0	17·0
Other labour	2·0	17·0
Overheads (management, buildings)	2·0	17·0
Drying (including labour)	8·0	68·0
Packing materials	2·3	19·0
TOTAL	32·6	276·3
Distribution, advertising, sales	1·0	8·5
TOTAL	33·6	284·8
Profit margins (total at 50 per cent)	16·8	142·4
TOTAL	50·4	427·2

The reconstitution ratio (drained weight) of dehydrated raspberries is 5 to 1, and the cost per pound of reconstituted product on the plate will therefore be approximately 85½*d.*

It is of interest to note how this compares with other forms of preservation. The A1 (14 oz) can of raspberries contains approximately 8 oz drained weight and sells for 32*d.* to 45*d.*, which makes the price of the raspberries 64*d.* to 90*d.* per pound.

A leading brand of quick frozen raspberries in 11 oz consumer packs contains 8½ oz raspberries and sells for 45*d.*, the price of the raspberries thus being 85*d.* per pound.

It should be noted, however, that the canned and frozen fruits contain sugar, which is included in the cost; this is not the case with the AFD product.

General Conclusions

It appears from the above that the cost of the accelerated freeze-drying stage alone will range from about 3*d.* to 8½*d.* per pound of prepared foodstuff entering the dryer; or from 8½*d.* to 85*d.* per pound of dry product, according to the commodity. Although these costs are a fraction of those regarded as inevitable for freeze-drying only a few years ago, they are still high, especially for certain vegetables and fruit. Where the initial cost of the raw material is low these high drying costs appear particularly severe, and the convenience factor would have to be high to enable AFD potatoes, for example, or cabbage, to find a ready market. On the other hand relatively costly raw material such as meat could presumably carry the processing costs more easily, and it so happens that the drying costs for meats are much lower than for vegetables and fruit. Even with drying costs of the order of those indicated in this chapter, however, many commodities should be economically feasible. The cost to the consumer should be little different from that for quick frozen foods, for essentially the products are quick frozen goods, carrying an extra cost at manufacture of 3*d.* to 8½*d.* per pound. After manufacture, however, they require no refrigerated storage or transport, and in general, substantially less storage space (Table 6). The longer they are stored, or the further they have to be transported, the more is this difference in production price minimized, and in many cases the final selling price of the AFD product could be less than that of its quick frozen counterpart.

The Future

It has been emphasized that all the foregoing calculations were based on the comparatively small scale operations of the Mark I AFD unit employed in the experimental work at Aberdeen. Processing on a larger scale might well reduce costs and on both sides of the Atlantic designers have been publishing estimates.

Vickers-Armstrong[12] has estimated a cost for the drying stage of approximately 2½*d.* per pound of foodstuff entering the cabinet. This estimate is particularly interesting, as calculations have been made both for systems with steam ejectors and for systems using mechanical pumping and refrigerated condensers, the comparisons being summarized as follows in pence per pound of foodstuff entering the driers:

	STEAM EJECTOR SYSTEMS	MECHANICAL PUMPING WITH REFRIGERATED CONDENSERS
	d. per lb	*d. per lb*
Capital and interest	0·78	0·86
Labour, maintenance and overheads	0·46	0·84
Vacuum and heating	1·30	0·80
	2·54	2·50

These figures apply to plant which will process 20 tons of prepared food per day, the capital cost being approximately £250,000 for that using steam ejectors, and about £280,000 for that using refrigerated condensers.

CHAPTER VIII

Possible future developments

It is almost certain that no further great reduction in drying time will be achieved in commercial scale plants within the next few years, but undoubtedly strenuous efforts will be made to reduce the processing costs while maintaining or improving quality. This will entail extensive research by the equipment manufacturers to develop more efficient plant, and in addition food processors already active in the field will be undertaking product development and market research.

The various features of the process which would probably merit further study are:

Evacuating equipment
Heating methods
Cabinet type and installation arrangement
Process control
Product development
Pre-processing methods
Packaging

Evacuating Equipment

Approximately 60 per cent of the cost of the dehydration stage on the Mark I plant was attributable to the evacuating equipment,[7] and it therefore seems likely that the greatest immediate saving in operating costs could be achieved in this direction. Steam ejectors are already highly specialized and well designed pumping units, so any improvement in efficiency using such equipment will probably result more from their economic application to multi-unit plants, and possibly their combination with other forms of pump, e.g., liquid ring pumps, in the final stages, than to radical changes in design. However, the main weakness in the steam ejector system is the large quantity of relatively cold water required in the condenser. This precludes its use on many sites, where alternative vacuum equipment is undoubtedly required.

The only alternative at present commercially available is the rotary pump and refrigerated condenser system, and while this entails much lower operating costs than the equivalent steam ejector installation, the capital cost is substantially higher.[12] Comparison between the two systems[13] indicates that as the capacity of the cabinet is increased, or alternatively as the absolute pressure in the cabinet is lowered, the rotary pump/refrigerated condenser system may be more economical. A major disadvantage of existing plant of this type is that it is common practice to use a fixed tube condenser capable of condensing out all the vapour evolved from the batch load. This results in a large vessel taking up a considerable amount of floor space, and increasing the volume of air to be removed before the operating pressure is reached. In

addition, the subsequent defrosting and re-cooling cycle can take as long as 2 hr. Should it prove more economical to use a refrigerated condenser system this problem can be mitigated by using twin parallel units, one of which is in operation while the other is defrosting. Not only would this reduce the size of the equipment but, in addition, it would eliminate excessive delay between batches.

A further point which should be considered is that operation of high-vacuum equipment requires a higher degree of technical skill than is necessary for most food processing plant. This is because the slightest damage to door seals, or incorrect operation of valves, etc., will introduce air leaks; and rotary pump systems are extremely limited in the quantity of leakage air they can handle. Steam ejectors on the other hand are not so sensitive in this respect, since the smallest ejector will handle about 10 lb/hr of air. Consequently, mechanical damage due to faulty plant operation is less critical with steam ejectors, though it should not be assumed that air leakage will not affect the operation of such a system at all, since this does in fact introduce a supplementary load on the augmentors, so limiting the quantity of water vapour that can be pumped.

Some work has been carried out in the U.S.A. on the possibility of utilizing liquid absorbents for removing the water vapour from the system.[14] These can be concentrated solutions of a number of hygroscopic chemicals, which at the temperatures and concentrations used have extremely low vapour pressures. Such a technique has much to commend it, since the solution containing the condensed water vapour could readily be pumped from the cabinet for subsequent concentration in a multiple-effect evaporator, and eventual return to the cabinet. It is to be preferred to the other form of continuous condenser, namely, the scraped surface condenser, since ice which forms under the conditions of AFD is extremely hard and difficult to remove by scraping. In addition, the scraper drive shaft has to pass through a gland seal, which is difficult to maintain free of leaks.

Another possibility is the use of these liquid absorbents in multi-stage water jet pumps. All of these methods, or combinations of them, are theoretically and practically possible; but careful study of the many alternatives will be necessary to determine which is economically preferable.

Heating Methods

The study of the AFD process has indicated that in the slow classical freeze-drying process the rate of heat input was the controlling factor. But the new process has adequately solved that problem and, except perhaps for small pieces of meat and fish, higher heating rates would simply aggravate the existing problem of vapour transport.

However, any method which relies on heat transfer through the layer of dry tissue is inherently bad, since some thermal damage is invariably caused, and for this reason alone some other method would be preferable. Recent work in U.H.F. heating at a frequency of 2,450 Mc/sec. has shown some promise,[15] but although this method selectively heats the ice in the product, it has so far been used only on a pilot scale and has a number of disadvantages. These are mainly caused by lack of uniformity in the application of the energy throughout the mass, but in addition the efficiency of conversion of energy is poor, and control of the process is difficult. This form of heating

would be of greatest benefit towards the end of the process, and while it would be impractical to utilize two different methods when using a batch system, the possibility of economic operation in a continuous drier should not be overlooked.

Of the methods which do employ heat conduction through the outer dry layer to the ice front, it has been shown that the double contact system gives the highest rate of heat transfer. (Experiments which purport to demonstrate an advantage in raising the upper heating plate are really only demonstrating that they are operating under conditions where vapour removal, not heat input, is the limiting factor.) Much is made of the cost of contact plate equipment but a study of the economics of the process will show that the capital cost must be reduced very substantially before the cost of the products is significantly changed. However, the system does not lend itself to adaptation to a continuous drier, and it will probably prove to be preferable to utilize some other form of heating for this purpose. A further disadvantage of the contact plate system is the necessity for having extremely uniform and level tray loads; although irregular loading is undesirable with any system the degree of uniformity is critical when contact plates are used and this intensifies the problems of preparation (e.g., cutting of slices of meat or frozen blocks) and of tray loading.

Work is currently proceeding in Canada,[16] where a commercial scale plant is being constructed, to study the suitability of purely radiant heating, with the product carried on mesh trays. The reported drying rates appear to be lower than AFD, but are probably sufficiently high to be suitable for a continuous drier. Similarly, simple shelf drying prolongs the drying time as compared with AFD, but is being proposed by at least one commercial undertaking for a continuous drier.

It is not suggested that the use of expanded metal is the ultimate in the AFD process, since it is recognized that it complicates the loading of the foodstuffs on the trays, results in a sandwich which requires careful handling and, in addition, it is fairly easily damaged if accidentally dropped or otherwise mishandled. Tests have been carried out on other forms of serrated heat transfer surfaces formed from dimpled aluminium sheets and aluminium sheets with machined grooves. These gave results which, although encouraging, were not as good as those obtainable with expanded metal; but if a suitable surface could be devised for a heating plate it might be a notable advance.

There appears to be no special reason for preferring any particular source of heat energy, although electrical heating tends to be rather expensive since the maximum demand on the plant is high. Another difficulty with electrical heating is that the plates must be fairly heavy to withstand the rather high plate pressures used, and consequently they have a high thermal capacity. This can introduce control problems where some cooling is required during the process, but in particular with regard to the need to cool the plates between batches.

Type of Cabinet and Installation Arrangement

As indicated in Chapter IV the Mark I cabinet was cylindrical, giving large clearances between the side of the plate stack and the shell of the cabinet. This proved to be extremely useful from the point of view of accessibility

for cleaning, but it did result in an unnecessarily large volume to be evacuated. On the other hand a cylindrical shell is the cheapest form of cabinet to make. The sub-division of the plate stack into three banks was not necessary, and in fact the Mark II AFD cabinet at present being designed by Atlas will not have this feature (nor does the Vickers-Armstrong model), but instead will have about double the active plate area in a shell of much the same dimensions.

It is generally agreed that one cabinet of the Mark I type does not constitute a viable freeze-drying installation, and it is suggested that four should be installed together, with a common evacuating system. This would allow for more economic utilization of the vacuum equipment, since the cabinets could be used serially and there would be an evening-out of the load on the vapour-removal system. Furthermore, the capital cost for a common evacuating system would be less than for separate units on each cabinet, and the utilization of the ancillary pre-processing and packaging equipment would be more complete.

The eventual aim in the development of most processes is to make them continuous, since steady-state conditions are more conducive to economic operation, a more uniform product is obtained, and labour requirements are reduced. It is probable that the AFD process will be no exception, but it is felt that it will be many years before continuous AFD driers are in common use for the types of product so far handled, and even then they will be, at best, only semi-continuous. It will be extremely difficult to devise a method of continuously feeding discrete pieces of solid foodstuffs into the evacuated space and spreading it uniformly on the drying surface; and thus, in the foreseeable future, loading and unloading will still be by batch operation through air-locks. However, the industry is still in its infancy and a great deal of knowledge is still required, not only as regards the process itself, but also with respect to consumer acceptance, before continuous high-capacity plants could be justified.

Process Control

The Mark I cabinet was completely manually controlled, but automatic control could readily have been devised. The evacuating system is inherently automatic in reducing the pressure in the cabinet towards the end of the run, but the heating cycle could well benefit from suitable automation of the heat input rate. This could take the form of a cam-type controller operating a diaphragm valve on the steam supply to the heater. The shape of the cam could be determined from previous experience with the particular product being dried, and it is possible that if sufficient experience were gained the use of thermocouples would not be required to indicate the end of the process. However, the surface temperature of the food is the variable which should be held at a safe maximum, and it therefore seems possible that a suitable control mechanism could be built round this feature. This would take the form of a number of surface thermocouples (say six distributed through the load), connected in parallel to indicate the mean temperature of the product surface. The resulting signal could then be used to control the rate of heat input to the plates, preferably with a rate-of-change sensing device, since the rate of change of temperature is rather high at the beginning of the cycle.

For recording purposes this mean surface temperature could be recorded with the plate temperature and an icicle; and a simple alarm system, actuated

by the icicle, could warn the operator of any excessive rise in the absolute pressure within the cabinet.

Product Development

It will be obvious that it has not been possible to devote a great deal of time to new product development in the Experimental Factory, and most of the experimental work has been concentrated on the drying of selected individual foodstuffs. Many of these products appeared to be saleable commodities since they compared favourably with similiar material preserved by other means.

It seems reasonable to assume that the main asset of freeze-dried materials is their convenience value, and that for many products they can be superior to their counterparts preserved in other ways. However, the ultimate convenience as far as the housewife is concerned would be the production of a complete dish which required the minimum of preparation, say, simply the addition of hot water. This is in fact possible with a number of the AFD products, and further investigation in this direction would probably be rewarding. Those that are dried from the raw state are somewhat less 'instantly' prepared but permit a choice of cooking methods; whereas those that are dried in a cooked state are ready to eat on reconstitution but are possibly less attractive. Chapter X gives an indication of a number of the products which were developed on the experimental plant.

Pre-processing Methods

It is generally true to say that most of the preparation techniques for the AFD process are similar to those employed in the quick freezing and canning industries. However, it has been demonstrated that improvement in the quality of the product and its storage behaviour can be achieved by the introduction of new methods, e.g., steam scalding of vegetables suits the AFD process better than water scalding.

The preparation of meat and fish present particular problems especially in that they have to be cut so that the pieces are flat and of uniform thickness, and also the muscle fibres should be at approximately right angles to the heating surfaces. This has involved a considerable amount of handling and the technique used experimentally at Aberdeen would scarcely be acceptable on the commercial scale. Simplification and mechanization are most desirable.

Similarly the experimental handling of composite food items containing liquids, such as meat and vegetable stew, rice pudding, etc., which were first frozen and then band-sawn into slices, was far from satisfactory. It is desirable to finish with a product which is regular in shape to simplify packaging, and a possible solution is to freeze the slurry in perfectly flat moulds loaded to a depth which will give the required thickness of piece. This would reduce handling requirements and losses of 'saw-dust' which can amount to as much as 10–15 per cent of the throughput. For the same reason the band-sawing technique used was equally unattractive with meat products. This can be avoided by using a bacon slicing machine or a guillotine, but the capacity of a bacon slicer is limited and experience with a guillotine was not very encouraging since the cut slices curled up.

The pre-freezing stage requires further study since the rate of freezing as well as the final temperature can have a marked effect on the product. Where

the product is to be frozen in moulds these should be as small as possible in section to accelerate the cooling cycle, and the use of some form of immersion freezing might possibly be beneficial. The use of a plate freezer to lower the temperature of beds of material (discrete particles or slices) would have the advantage of giving substantially flat surfaces, and an extension of this idea is to use the drier itself as the freezer by circulating a refrigerant through the plates. Although this would ensure that any corrugations on the heating plates would be duplicated in the tray of material, so giving 100 per cent contact, it is felt that the complication of the circulation for both cooling and heating would not be justified.

Packaging

Packaging has probably received the least attention in the development of the AFD process and a very considerable amount of research and development work is required in this field. There is obviously no point in turning out a sound product if it is impossible to ensure that it reaches the consumer in good condition. As indicated in Chapter VI, the requirements are many and varied, and quite obviously for retail consumer packs the tin-plate container is not the answer, although it may be suitable for institutional purposes.

SECTION B

Food Technology

CHAPTER IX

Choice of raw materials

It is not the function of this Report to deal with the suitability of raw materials for food processing in general, although freeze-drying has demands which are obviously common to other forms of food preservation. What is presented here is an account of those considerations which were found to be particularly relevant to the AFD process.

FRUIT AND VEGETABLES

Agricultural Considerations

It may go without saying that this relatively expensive process calls for the utmost economy in supply, but specifically the need for regularity in tray loading (so as to avoid uneven drying) and the influence of reducing sugars on storage life make special demands in terms of uniformity of size and maturity in vegetable crops.

A special point with reference to plant disease is that some foods are not cut up before freeze-drying, and Brussels sprouts, for example, may suffer from an internal necrosis which is not apparent but which produces an internal browning in the dried product.

Suitability for Mechanical Handling

The fruit or vegetable should be of a type suitable for mechanical handling in the factory; for example, stone fruit should be free-stone rather than cling-stone; soft fruit should not be over-mature; strawberries and raspberries should be varieties that are easily husked or plugged. Carrots should be stump-rooted, of uniform taper, preferably cylindrical, red-cored, free from woodiness, not too small (as this would lead to heavy preparation losses) but, if transverse slices are to be cut, small enough to be of good appearance, i.e., not more than $1\frac{1}{4}$ in. diameter. Cabbage should be firmly hearted as loose leaves do not handle well in the cutting machinery.

Potatoes should be uniform in shape, with shallow eyes and preferably thin skins, so that excessive preparation losses and handling may be avoided. It has been found that surprisingly large losses can be incurred if the degree of peeling is heavy, Table 11 (page 82) showing that when a spherical potato of 3 in. diameter is peeled to a depth of $\frac{1}{8}$ in. the resulting loss is not less than 23 per cent.

Experience on a commercial dehydration plant working under war-time conditions underlined the importance of this factor—a change from King Edward VII, a thin-skinned, shallow-eyed variety, to Kerr's Pink, reduced the output of dehydrated product from over 30 tons to 27 tons per week, the reduction being due to a combination of higher peeling losses and slower trimming rates.

TABLE 11

Losses suffered by potatoes when peeled to different depths

Diameter of potato	Percentage of volume in layer of thickness	
in.	$\frac{1}{16}$ *in.*	$\frac{1}{8}$ *in.*
2	17·6	33·0
$2\frac{1}{2}$	14·3	27·1
3	12·0	23·0

FLAVOUR

Natural flavours are largely contributed by volatile substances in the plant tissue, and even under conditions of accelerated freeze-drying there is some loss of volatiles by distillation. There is also some loss during the scalding that usually precedes drying. Processing techniques have not yet progressed far enough to eliminate these losses. It is therefore most desirable to start with a raw material that is strongly flavoured. This has been recognized in the U.S.A., where onions have been specially bred for dehydration, the flavour of the fresh onion being so strong that it is virtually inedible. After dehydration it has moderated to the extent of giving a product of full onion flavour.

Wild cranberries and blackberries have stronger flavours than cultivated, and this difference persists after freeze-drying.

Although there is no systematic knowledge about the effect of freeze-drying on flavour, observations at Aberdeen have shown rather unexpected changes in certain fruit. Apricots, for example, should be fully ripe; unripe fruit were found to develop oily or fishy taints after dehydration. Unripe greengages yielded a product with hay-like aroma, but ripe greengages gave satisfactory products. In general, it seems that red and blue plums gave better flavoured products than the green varieties. White-fleshed peaches, after freeze-drying, were considered to have better flavour than the yellow varieties, though the latter were still regarded as satisfactory. In the case of black currants the varieties Cotswold Cross, Mendip Cross and Malvern Cross were considered to have flavours less suited to dehydration than most other British varieties.

It has been stated that carrots sown late and grown quickly have a superior flavour to early sown carrots,[17] but there is little precise information. Stringless French beans have been bred, and although these are weaker in flavour than stringed varieties, the advantages of a stringless bean are paramount.

Although off-flavours in the product may be caused by processing or storage faults, they may also originate in the raw material itself, and it is important for the processer to be satisfied that his raw material is free of such characteristics. For example, carrots kept in cold storage may develop a strong bitter flavour.[18] Certain insecticides, such as benzenehexachloride (BHC) applied to the soil may confer taints on root vegetables grown in that soil; similarly TVO, frequently used as a herbicide, is liable to confer taints if the growing season is dry. Certain sprout depressants, e.g., tetrachloronitrobenzene (TCNB), applied to potatoes in store may cause flavour taints which persist into the finished product.

Texture

In fruits and vegetables there are often characteristic varietal differences in texture, and this is also affected by cultural conditions—the type of soil, manuring, amount of rainfall, etc. A striking case is that of potatoes, in which different varieties grown under similar conditions show wide and characteristic differences in texture; but the same variety grown under different conditions may show an even greater range of textural difference. In other words, the effect of cultural conditions is greater than the effect of variety.

The stage of maturity is of great importance. In fruit, ripening leads to changes in the type of pectin and in the juiciness of the tissue. In apples, for example, these changes mean that the fruit approaching the ripe condition is able to dry more rapidly and, after dehydration, to reconstitute more rapidly. In general it is true to say that the riper the fruit the more easily is it dehydrated; on the other hand the softness of the tissue makes it more easily damaged by handling and processing, and thus fruit for dehydration should be harvested just at the point of ripeness or just before ripeness. Over-ripeness is to be avoided. Peas, broad beans and French beans should be harvested at the same stage as that at which they are normally taken for freezing (not canning), i.e., the two former with tenderometer reading about 95, and stringless French beans before the swelling of the seeds causes visible swelling of the pods.

Colour

Material of the appropriate colour must be selected; red-cored carrots are desirable on grounds of appearance and also because of the higher carotene content; peas should be of dark green varieties (the type of peas grown for quick freezing rather than those grown for canning). Most varieties of broad beans contain leuco-anthocyanins which are liable to darken during dehydration, giving irregular purplish patches and thereby detracting from the appearance of the product. Variety Threefold White is free from leuco-anthocyanins, and from the point of view of appearance was considered suitable, though the flavour was regarded by some as inferior.

Potatoes with slightly lemon-coloured flesh gave a more attractive product than dead-white varieties. (Some varieties of potato are also liable to greying —probably stem end blackening—during processing.) King Edward VII was better than Majestic on both scores and had all the desirable characteristics except that it was usually a little more expensive.

Black currants should be dark coloured rather than the lighter coloured varieties; Seabrooks Black was particularly good in this respect. Strawberries should be well coloured all over. White patches gave rise to unpleasant grey blotches in the processed fruit and the seeds tended to go black. Over-ripe tomatoes tended to give a product with an almost purplish tinge. It was essential for plums which changed from green-and-red to red, or green-and-blue to blue, to be ripened until the green disappeared, for on drying the green areas became an unpleasant brown. In general red and blue plums were preferred to green plums, but more on account of flavour than colour.

It was sometimes difficult with unripe apples (i.e., early in the season) to control slight discoloration of the dice. This trouble did not appear to be due to enzymic browning and might perhaps have been associated with leuco-anthocyanins.

Nutritive Value

Chapter XIII deals with this subject in respect of the dehydrated product, and of course every attempt should be made to start with material of high nutritive content. There is, for example, virtually no loss of β-carotene during dehydration,[19] but if a high β-carotene content is required in the product, material with initially high β-carotene content must be processed; and in general therefore red-cored carrots would be preferred to pale-cored ones.[20]

The ascorbic acid content is well maintained in dehydrated peas and cabbage, and reflects closely the content of the original material. In the case of cabbage this is a function of the season rather than the variety, there being a very marked and steady drop in the ascorbic acid content of fresh cabbage from about May on to the end of the Savoy and winter cabbage season. At any time of the season the several varieties have much the same ascorbic acid content, ranging from about 1,000 mg/100 g dry matter in the spring down to about 200 mg/100 g dry matter in the winter (Fig 7).

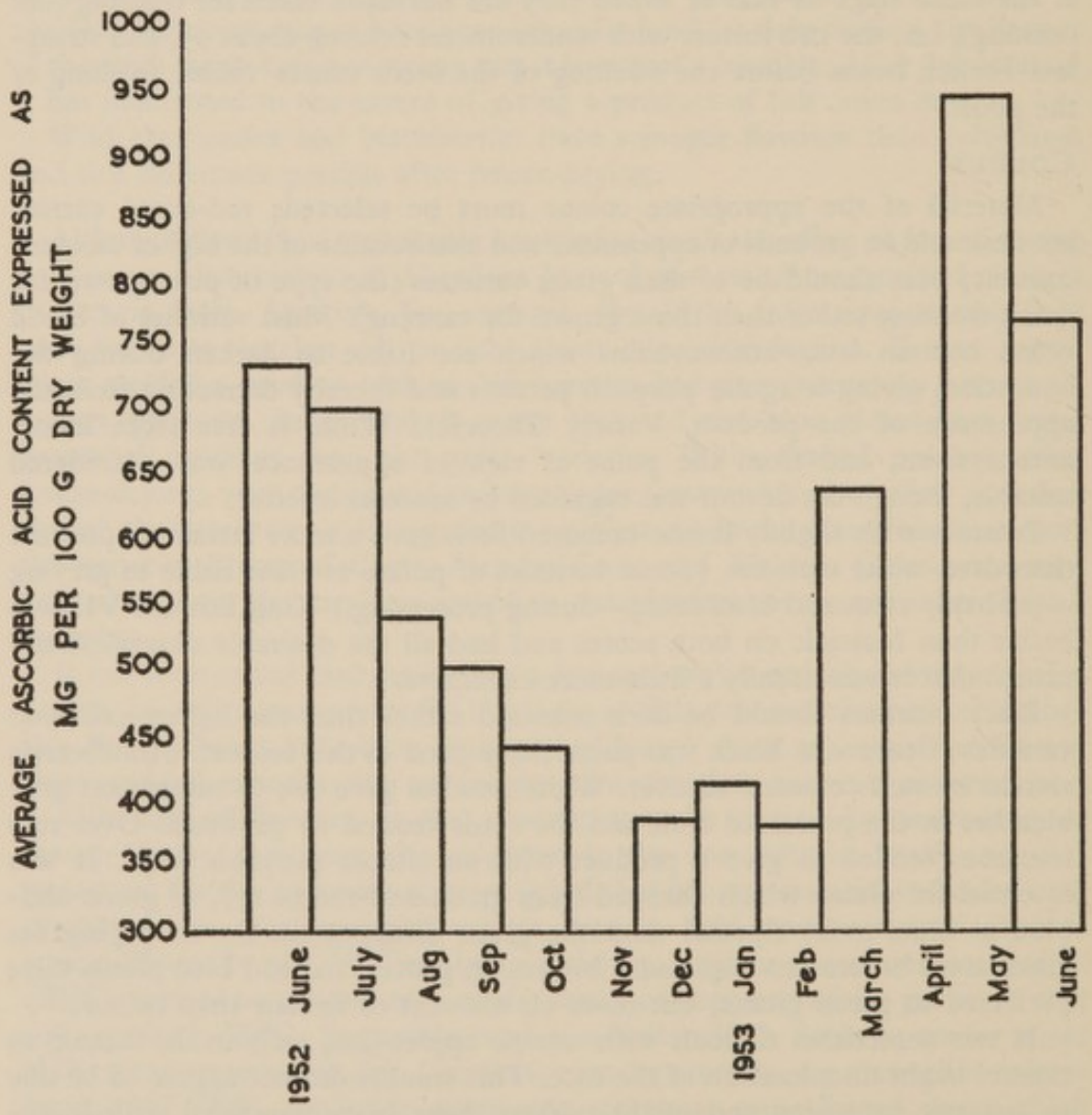


FIG. 7. *Monthly averages of ascorbic acid in fresh cabbages (from all sources)*

Black currants are also valuable providers of ascorbic acid and it was found that both large and small fruits contained less ascorbic acid per unit weight than those of medium size.

Chemical Composition

The storage behaviour of dehydrated foods, and to some extent their behaviour during processing, may be affected by the chemical composition of the raw material. The browning reaction has been examined in some detail

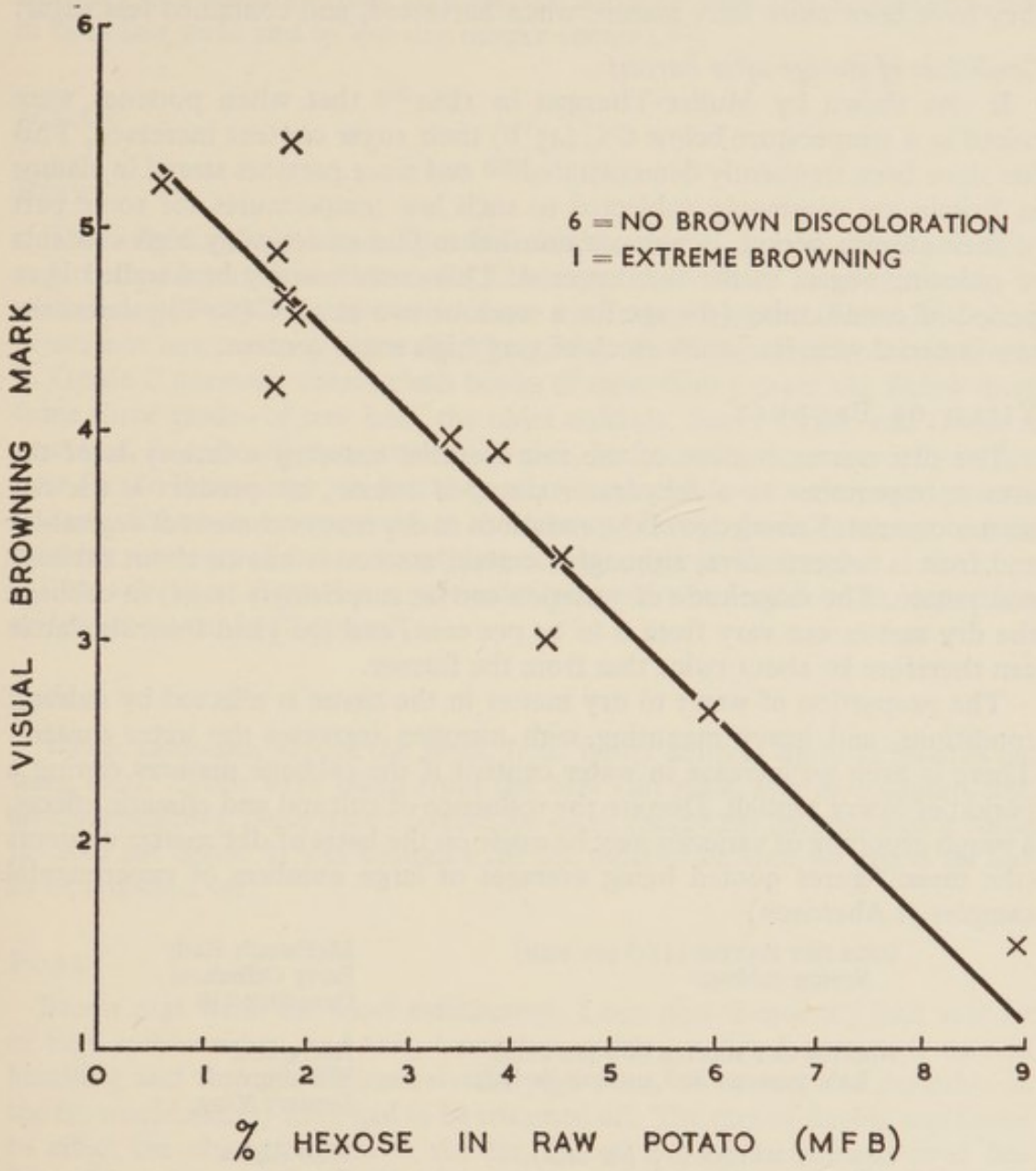


Fig. 8. *Reducing sugar content of raw potatoes and visual browning mark scored by their dehydrated products after* 3 *days storage at* 55°C

in the case of potatoes and, as might be expected, the extent of browning developed under particular conditions of storage is closely related to the reducing sugar content of the dried product, which in turn is governed by the reducing sugar content of the raw tubers (Fig. 8). It was thus necessary to

consider the factors which affected the reducing sugar content of the tubers. These are as follows:

The maturity of the potatoes at harvest

It has been known for a long time[21] that the reducing sugar content diminished rapidly during the last few weeks of maturing of the tuber—maturity being defined as the time when the haulms turned yellow and died back naturally. In years with high summer rainfall (and consequently low sunshine) potatoes have tended to be physiologically immature and with high sugar content. By contrast, in years with low summer rainfall (and much sunshine) they have been more fully mature when harvested, and contained less sugar.

Conditions of storage after harvest

It was shown by Muller-Thurgau in 1882[22] that when potatoes were stored at a temperature below 6°C (43°F) their sugar content increased. This has since been frequently demonstrated[23] and since potatoes stored in clamps in Britain are commonly subjected to such low temperatures for some part of their storage period, it was not unusual to find undesirably high contents of reducing sugars in the raw material. This could usually be handled by a period of conditioning (storage for a week or two at 21°C (70°F)), unless the raw material were immature stock of very high sugar content.

Yield of Product

The dry matter content of the raw material entering a factory is of the utmost importance to a dehydrator since, of course, his product *is* the dry matter content. Knowledge of the variations in dry matter content of vegetables and fruit is not extensive, although a certain amount is known about cabbage and potato. The magnitude of variation can be surprisingly large; in cabbage the dry matter can vary from 6 to 14 per cent, and the yield from the latter can therefore be about twice that from the former.

The proportion of water to dry matter in the tissue is affected by cultural conditions, and heavy manuring with nitrogen increases the water content. There is even an increase in water content if the cabbage matures during a period of heavy rainfall. Despite the influence of cultural and climatic effects, a rough grouping of varieties may be made on the basis of dry matter contents (the mean figures quoted being averages of large numbers of experimental samples at Aberdeen)

HIGH DRY MATTER (11·5 per cent)	
Spring cabbage	McEwan's Early
	Early Offenham
	Ormskirk 218
MEDIUM DRY MATTER (8·6 per cent)	
Late summer and autumn varieties	Rearguard
	Winningstadt
	January King
LOW DRY MATTER (6·8 per cent)	
Early summer varieties	New Alpha
	Premier
	Primo
	Golden Acre
	Utility

Although the edible portion of varieties with high dry matter appears to be no more fibrous or coarse than that of varieties with lower dry matter content, there is some indication that in the former the outer leaves are

coarser and that heavier trimming losses are therefore involved; the improvement in yield from a given weight of raw cabbage of high dry matter content is seldom, therefore, as great as might have been anticipated.

The dry matter content in potato is again partly a function of cultural conditions and partly of variety. Golden Wonder, Arran Chief and Doon Star, for example, normally have high dry matter contents (22–23 per cent); Majestic, King Edward VII and Kerr's Pink have medium contents (20–22 per cent); and Arran Banner is low. The effect of cultural conditions is greater than that of variety, and in Scotland there has been ample confirmation of Burton's generalization that high rainfall between May and September leads to both low yield and to low dry matter content.[21]

MEAT

Beef

The following is the broad general classification of carcasses, in descending order of price, used in Scotland. (The term 'clean beast' is used to refer to castrated males and females which have not been allowed to breed.) Of clean beasts not more than 3 years old, the grades are Super, Grade A and Grade B. Grade C normally covers clean beasts of more than 3 years old. Below these come three grades of cow beef, the older animals. Super Grade and Grade A carcasses, in spite of their attractive finish, had no advantages for dehydration over Grade B carcasses; in fact they were less suitable since they carried more fat, which had to be removed in the factory. Lower grade carcasses had increasing amounts of connective tissue and were generally tougher.

The selection of cut for a particular purpose was found to be important, and is discussed in Chapter X.

Lamb

Whole carcasses, except the flank, from fleshy animals up to 12 months old were used. Chops were taken from the best end neck, loin and chump, and all the remaining parts were suitable for slices except the shanks, which could be used for cubes. It was desirable for the meat to be lean, as excess fat had to be trimmed off.

Pork

Bacon pigs were the most satisfactory. Lean pigs frequently had soft fat of high iodine value, and this, being less stable, required particular care in handling and storage. Excessively fat pigs were, of course, not desirable as the fat would merely have had to be trimmed off. The diet of the pig was found to affect the characteristics of the fat, and if it contained unsaturated fats (e.g., from fish meal) the pigs laid down unsaturated fats in their depots. These characteristics are perhaps less important in accelerated freeze-drying than in other forms of dehydration, since there is little exposure of the meat to oxygen, but even so the greater the stability of the fat the better.

As with lamb the whole loin from the fifth rib was used for chops, and all the rest of the carcasses was suitable for slices, except the shanks, which could be used for cubes or mince. The flanks were not used.

FISH

The characteristics and distinguishing flavours and textures of the various species of fish are only obtained after the fish has come out of rigor, and although the general statement is made that fish for freeze-drying should be as fresh as possible, it is made with the above reservation.

In practice it was found that fish kept in ice for periods of up to 10 days was satisfactory; this being because the di- and tri-methylamines which developed during storage and gave the characteristic and increasingly fishy flavour, were in large measure distilled off during dehydration.

Although fatty fish, such as herrings, are unsuitable for hot-air drying, owing to the unsaturated and unstable nature of their oils, they could be successfully handled by accelerated freeze-drying provided their fat content was not exceptionally high. If there was a very high content of fat, although drying might be satisfactory, the presence of the fat impeded rehydration.

A summary of the qualities required by twenty-seven representative raw materials for freeze-drying is given in Table 12.

TABLE 12

Summary of Qualities of Raw Material for Freeze-drying

Commodity	Varieties known to be suitable for AFD	Remarks
ANIMAL PRODUCTS		
Beef	Grade B carcasses	Guns for fillet and rump steaks, cubes, high grade mince. Fores for low grade cubes and mince.
Lamb	Whole carcasses (without flank)	Ribs for chops. Remainder for slices or cubes except shanks.
Pork	Bacon Fat Pigs	Ribs for chops. Remainder for slices or cubes except shanks.
Fish	Post rigor: up to 10 days in ice	
VEGETABLES		
Broad Beans	Threefold White	Tenderometer 95.
Brussels Sprouts	Cambridge Special Sanda	To pass 1¼ in. riddle.
Cabbage	Most varieties	Preferably firmly hearted, hearts not too large otherwise there is excess of white material and less dry matter content. Ascorbic acid is high early in the season, falling off through the summer and autumn.
Carrot	Red-cored Chantenay James' Intermediate Stump-rooted Intermediate	Red-cored, stump rooted varieties, with strong carrot flavour, medium sized if required for slices. Large (free from woodiness) if required for dice.
Green Beans	Saxa Konserva II Record	Harvested just before swelling of seeds causes visible swelling of pods.
Peas	Laxton's Exquisite Perfection Freezer Lincoln Kelvedon Wonder Witham Wonder	Tenderometer 95. To be processed as quickly as possible after vining.

TABLE 12—continued

Commodity	Varieties known to be suitable for AFD	Remarks
WILD FRUITS		
Cranberries	Wild Scotch and Howe (late, cultivated)	Ripe, well coloured, free from leaves and grouse droppings. Dried whole, with apple or as pulp.
Rose Hips	Wild Scotch	Well coloured, deep red berries. Dried as halves or as pulp.
Brambles or Blackberries	Wild Scotch	Dried separately or with apple. Sound, free from moulds and shrivelled berries.
Rowanberries	Wild Scotch	Sound, ripe, free from maggots, moulds and brown and shrivelled fruit; dried whole or as pulp.
IMPORTED FRUITS		
Apricots	Not Known	Spanish apricots have been used throughout. *It is absolutely essential* for the fruit to be fully ripe and well coloured.
Peaches	Not Known	Italian, French and South African peaches have been employed. French white-fleshed dessert peaches have been found to give the best flavour, although texture inclined to be soft. Peregrine and Duke of York only two named varieties used—both suitable.
Bananas	Not Known	Best used when completely yellow before they become speckled with brown.
Pineapples	Not Known	Material found most suitable was that imported from the Azores. Must be well coloured and sound.
Dates	Not Known	Iraq dates dried whole or as pulp. Product powdered and used for flavouring, e.g., milk shake.
Raisins	Californian Blue	Dried after milling to pulp, alone or with apple.
Citrus Fruits	Lemons, Grapefruits and Oranges	Used in conjunction with apple as a pulp composed of flesh and outer layer of rind. Rind must be well coloured and free from blemishes.

TABLE 12—continued

Commodity	Varieties known to be suitable for AFD	Remarks
HOME GROWN CULTIVATED FRUITS		
Gooseberries		All commercially available varieties are suitable. Should not be too ripe.
Strawberries	Auchincruive Climax Royal Sovereign Red Gauntlet Talisman	Berries should be ripe and evenly coloured and plug removed. Huxley is not satisfactory because of the difficulty of removing the plug.
Raspberries	Malling Promise Malling Exploit Malling Jewel Malling Enterprise Lloyd George Norfolk Giant	Berries should be well coloured, without plug and firm.
Black Currants		Most commercially grown varieties are suitable. They should be ripe and well coloured. The following varieties have been found to be both slightly better in flavour and also high in ascorbic acid—Westwick Choice, Wellington XXX, Baldwin, Seabrooks Black and Rosenthals.
Apples	Grenadier Lord Derby Bramley Newton Wonder Sturmer Pippin	These five varieties have been more frequently used than any other. Most cooking varieties would be suitable. The best stage of ripeness would appear to be when the green colour in the skin is changing to yellow with the flesh still firm and juicy.
Plums	Various	The most suitable plums are the blue and red varieties. They should be used when all green colour in the skin is absent. Damsons and Zwetsche plums with strong flavours have been found to give excellent products. All commercially grown. Blue and red varieties can be successfully dehydrated. If green plums and gages are used they must be ripe.

CHAPTER X

Preparation of foodstuffs for drying

For all food preservation processes the foodstuff has to be suitably prepared, and indeed one of the advantages of the AFD process is that the preparation is done in the factory so that all that is stored, transported and purchased is edible material without waste. A further advantage is that foods may be freeze-dried in the raw state, so that after reconstitution they may be cooked to suit the consumer; or they may be freeze-dried after cooking, when water alone will render them ready for consumption.

The preparation of foods for accelerated freeze-drying is similar in many respects to that demanded by other processes, though there are certain special requirements which are dealt with below, by commodity.

MEAT AND MEAT PRODUCTS

Meat and meat products may be dehydrated either raw or after cooking.

Raw Beef Steaks, Cubes and Mince

The most suitable material was found to be Grade B beef, and it was usual to use 'guns' (i.e., the hindquarter cut between the fifth and sixth rib, with the flank removed) and to discard the shank. Forequarters were not generally used owing to the low percentage of steak cuts and the high proportion of connective tissue. After slaughter the beef was hung for 7 days at 4°C (40°F) to bring about a reasonable degree of tenderness. (It was necessary to have forced circulation of air in the conditioning room to avoid mould growth.)

After hanging, the meat was boned and trimmed. All lymph nodes and obvious pieces of gristle were removed. True steaks, suitable for grilling, were taken from the rump and fillet and the remainder normally diverted to the preparation of pre-cooked products, although good quality raw beef cubes and raw beef mince were made from the hindquarters when required.

The trimmed meat, in the form of joints, was frozen in a cold room at −7°C (20°F). If required, it could be frozen in moulds to yield a regular shaped mass. After 24–30 hr the frozen meat could be sliced to the required thickness on a bacon slicer.

It was essential at this stage to cut the steaks from the joint in such a way that, as far as possible, the cut surface was at right angles to the direction of the muscle fibres. This is normal practice for cutting steaks, but is particularly important in this process if rapid drying and good reconstitution are to be achieved.

The second point, of paramount importance, was to ensure that no thawing took place, even on the surface. Steaks which were not totally freeze-dried, as a result of partial thawing before drying, were difficult to dry and impossible to reconstitute completely.

The cut pieces were laid on the drying trays between two sheets of expanded metal, and the tray load kept hard frozen until required for insertion into the drying cabinet.

If the joints were allowed to freeze to temperatures below about −7°C (20°F) they became too hard to cut on a bacon slicer and band-sawing was necessary, with resultant heavy sawing losses. It was thought that a guillotine might be developed to slice such hard frozen material without such wastage.

In order to give reasonably short dehydration times, steaks were commonly sliced to a thickness of 15 mm and loaded at about 2½ lb/sq. ft. With this material moisture contents of less than 2 per cent were achieved in a drying time of less than 6½ hr. Thicker slices could be dried satisfactorily with longer drying times, but there was evidence that longer drying times had an adverse effect on quality, and 15 mm seemed a fair compromise.

Cubes cut from frozen joints to a standard 15 mm edge were dried, with a loading of some 2 lb/sq. ft, to 2 per cent moisture in 5½ hr. With cubes it was not essential to have the fibres lying at right angles to the heating plates.

Raw beef mince could be dried after freezing by evaporative cooling in the cabinet, provided it was ground with a cutter plate with holes not exceeding $\frac{5}{32}$ in. in diameter. If raw mince of larger particle size was required it was necessary to pre-freeze before loading into the cabinet. When raw minces were dried care was taken to provide an even bed of material on the expanded metal sheets, and a bed thickness of 20 mm could be dried in about 8 hr. This thickness of bed represented a loading of 3 lb/sq. ft.

Minced, Cooked Beef

After the carcass meat had been hung in a conditioning room for 7 days at 4°C (40°F) (with forced circulation of air), it was boned out and trimmed. The trimmed meat was cut into cubes of about 1½ in. edge, during which operation all tendons, lymph nodes and blood clots were removed. The trimming was such as to yield approximately 35 per cent of fat in the dried product.

One hundred pound quantities of trimmed cubes were cooked in each batch, using 11 gal of water in an open steam-jacketted pan. Simmering was continued for 40 min and then the meat removed as quickly as possible from the pan. Serial cooking was used for a maximum of 3 cooks, the volume of water being made up each time to 11 gal with fresh water. The meat was transferred to cooling bins and cooled with a through-blast of filtered air. When cool (a minimum of 20 min), the meat was placed in covered metal containers and held at 2°C (35°F).

After each set of 3 cooks, the liquor was drawn off from the cooking pan, strained through a fine stainless steel mesh, and passed through a centrifugal separator to remove solid particles and fat. The clarified gravy was returned to the steam-jacketted pan and concentrated by evaporation until the weight of gravy had been reduced to about one-tenth of the weight of the cooked meat. Immediately before drying the concentrate was added to the cooked meat, and the gravy-coated chunks minced through a ⅜ in. cutter plate directly on to the drying trays.

Pre-freezing was not necessary as cooked minces freeze satisfactorily by evaporative cooling. Loadings up to 3 lb/sq. ft were dried in 6½ hr.

Cooked Beef Slices

The trimmed meat was cut as necessary to fit into the rectangular cooking moulds and packed so that, as far as possible, the muscle fibre direction in the pieces of meat lay parallel to each other and to the long axis of the mould. Spring-loaded lids were attached, and the moulds placed in a steam chest where cooking took place at 82°C (180°F). The material was considered cooked when the temperature at the centre of the meat reached 66°C (150°F). After cooking, the moulds were drained of their liquor, from which a 10 per cent gelatine solution was made. This was returned to the moulds and the lids replaced with mild pressure. They were held at 4°C (40°F) until the gelatine set, and then the blocks of cooked meat were removed and sliced on a bacon slicer.

Slices of 5 mm thickness were found to be robust enough when dried to stand reasonable handling when packing. Thinner slices were too fragile, and thicker slices were slow to reconstitute. The slices were placed directly on to expanded metal sheets on the drying trays.

A double layer, each consisting of 2 slices, the layers being separated by a third sheet of expanded metal, amounted to a loading of over 3 lb/sq. ft and could be dried to less than 2 per cent moisture in 7 hr.

Pre-freezing cooked beef slices before drying is not absolutely essential, as they can be frozen by evaporative cooling in the cabinet. Pre-freezing will, however, allow the material to be dried at a lower temperature under the same vacuum conditions, and the product is lighter in colour and more attractive in appearance.

Raw Pork Chops, Steaks and Cubes

The most suitable material for raw pork products was bacon-quality pigs with a dressed weight of 7½ score (150 lb). There was no necessity to condition the carcasses and it was usual to bone-out as soon as convenient after the effects of rigor had worn off.

Chops of acceptable quality were cut from the fifth rib down to the last lumbar vertebra. The flank was not used. Thus when the side of pork was boned and trimmed the main part of the back (the middle, between fore and gammon) was rolled into chops.

Freezing was carried out in the cold store at −7°C (20°F) and after 24–30 hr the material was sliced on a bacon slicer. As with beef every effort was made to cut at right angles to the muscle grain; and the material was not allowed to thaw. The remainder of the carcass (i.e., those parts not used for chops) could be treated in the same way and dried as steaks or cubes, depending on quality.

Material cut to a thickness of 15 mm could be dried to a moisture content of 2 per cent in 7–8 hr, with a tray loading of 2½ lb/sq. ft.

Cooked Pork Mince and Slices

The best material for this purpose was found to be the fores and gammons of bacon pigs. With this material there was very little trimming loss, especially in the case of mince, since sufficient fat to give 35 per cent in the dry product is intentionally left in the cubes before cooking. The processing of the pork for mince and cooked slices is similar to that described above for beef.

Lamb

Lamb, for the production of raw chops, steaks and cubes can be treated in similar fashion to pork. The raw material giving the best yields as well as the best product was year-old fleshy lambs. Hanging the carcasses to condition before boning was more important than with pork but not as important as with beef. It was the practice at the Experimental Factory to hang carcasses of lamb for 3–4 days at 4°C (40°F) and carcasses of older animals (mutton) for up to 7 days at 4°C (40°F). Drying times in the region of 7–8 hr for material cut 15 mm thick were normal for lamb chops and steaks.

Lamb can also be dried satisfactorily in the form of cooked mince or slices, using the methods described above for beef and pork; and the whole carcasses but for flank and shanks, of year-old lambs, were used.

Ham

Cooked fores or hams were used to prepare ham mince, cubes and slices. The mince was passed straight on to the drying trays from a mincer with $\frac{3}{8}$ in. cutter plate.

Although ham mince stored well after dehydration, this was not the case with cubes or slices. Using the same techniques the latter seemed excellent on removal from the drying cabinet, with good reconstitution and high acceptability to a taste panel. After a few months, even when gas-packed or vacuum-packed they became poor in colour and deteriorated in flavour and texture. The reason is as yet unknown.

Veal

Cooked veal mince was made in similar manner to cooked beef mince, starting either with calves a few days old or with imported frozen boneless veal. It was used to make a veal-and-ham mixture.

Chicken

Cooked chicken pieces and mince have been dried by the AFD process. Dressed carcasses were cooked by immersion in boiling water. When cool enough to handle the flesh was removed by hand and divided into light and dark meat. The best product was composed entirely of light flesh, the dark being rather slow to reconstitute. It is better minced. It is possible to concentrate the cooking liquor, as with beef mince, and add it to the chicken flesh before drying, but it tends to discolour the product.

A loading of $2\frac{1}{2}$ lb/sq. ft could be dried in less than $7\frac{1}{2}$ hr.

Offals

Materials such as kidney, liver, pancreas and brain from cattle have been dried with reasonable success; but the work on these has been limited and practically nothing is known of their storage properties.

FISH

Work on the dehydration of fish has been confined to cod (*Gadus callarius* L.). The fish was received in the gutted state, the heads removed and the tail taken off in front of the last dorsal fin. (The tail portions were filleted and the fish used for cooked products.)

After thorough cleaning, the fish were blast-frozen to about −12°C (10°F). In some cases they were frozen in moulds to make a more regular-sized product, but in any case the belly flaps were folded in to make a neater form.

The best products were in the form of steaks (i.e., transverse sections about 15 mm thick), and these were transferred directly from the band-saw to the drying trays, using expanded metal inserts, and held refrigerated until loaded into the drying cabinet.

It was usual to leave the backbone in the material, which made it very robust for handling. On the other hand, more rapid drying was achieved if the bone was removed, at the risk of breaking up the steak either on packing or, most likely, on reconstitution.

Drying times for 15 mm steaks loaded at 2½ lb/sq. ft were about 8½ hr.

Cooked fish was dried as flakes. Cooking was carried out in an atmosphere of steam, the skinned fillets being laid out on mesh trays. After cooking, the material was broken up in a dough-mixer, and then rapidly cooled to prevent bacterial growth. The cooled material was laid on expanded metal sheets on the drying trays. Pre-freezing was not necessary.

Loadings of 2½ lb/sq. ft gave a moisture content of less than 2 per cent in 9 hr.

Smoked fish (for a smoked fish-cake mix) was dried in a similar manner.

Shellfish

Sample quantities only of pre-cooked prawn, shrimp, crab and lobster meat were dried. The products reconstituted rapidly and their culinary quality was very good.

CHEESE

Several small batches of minced cheese were dried, ground through a cutter plate with $\frac{5}{16}$ in. holes. There was no evident difference in the products dried with or without expanded metal sheets, and either pre-freezing or evaporative freezing techniques could be used, the former being preferred.

The drying time was 8 hr for a loading of 2¼ lb/sq. ft.

VEGETABLES

The essential details for the preparation of vegetables are summarized in Table 13.

Washing and Inspecting

Transfer of vegetables from store to the processing line was by fluming, which was economical in floor space, reduced airborne dust and effected some washing of the raw material.

Root vegetables were elevated from the flume sump by a lifting wheel to the pre-washer, through which a continuous flow of water was maintained. Next came the inspection belt, where diseased material, stones, etc., were removed by hand and vegetables which by their size or shape might cause difficulty in peeling were cut into suitable pieces.

Peeling and Trimming

Potatoes and carrots were fed at an even rate to continuous carborundum roller peelers. Uniformity of feed was essential, for once the machines had been set to handle a given throughput they under- or over-peeled if the rate

varied. Trimming of the material by hand to remove blemishes such as worm holes, eyes and diseased patches took place immediately after peeling.

Potatoes

Trimmed potatoes were fed into the appropriate machine for chipping or dicing (success was achieved with chips up to $\frac{1}{2}$ in. by $\frac{1}{2}$ in. cross-section) and the cut material passed through a water spray to remove starch from the cut surfaces. If this was not done the surface starch gelatinized during scalding and eventually formed a skin which had an adverse effect on drying and reconstitution.

The washed material was fed into a steam-scalder, the time taken for passage through being determined by a peroxidase test for enzyme inactivation (see Chapter XIV).

The scalded material was placed on expanded metal sheets on the drying trays, blast-frozen and then inserted into the drying cabinet. Loading at $1\frac{1}{2}$ lb/sq. ft gave a drying time of about 8 hr.

Carrots

Processing of carrots was similar to that for potatoes, but the crowns had to be removed during trimming and there was no necessity to wash after cutting as no problems arose from surface starch. The smallest dimension of the cut pieces should not exceed $\frac{3}{16}$ in.

Cabbage

Nets of cabbage were emptied on to wooden tables at the end of the trimming belts. The cabbages were hand-trimmed and cored on wooden blocks at the side of the belts. The method involved four strokes of the knife—the first at right angles to the stalk, cutting off the base of the stalk and removing the outer coarse leaves: the second through the heart, parallel to the stalk but just off-centre, leaving the whole core in one side; the third and fourth obliquely on each side of the core, so as to remove it as a triangular pyramid. This was followed by shredding (maximum thickness of shreds $\frac{3}{16}$ in.), washing and scalding.

The time required for scalding different batches varied both by variety and by source of supply. Sometimes there was a very narrow margin between peroxidase inactivation and over-scalding, the latter leading to sogginess on the drying tray, difficulties in drying and a mushy product. The correct scalding time was between 2 and $2\frac{3}{4}$ min, but good results depended to a great extent on the skill and judgment of the operator.

The addition of sulphur dioxide, in the form of sulphite or metabisulphite, is normal practice with air-drying, for it reduces loss of ascorbic acid, inhibits non-enzymic browning and in the case of green vegetables gives products of brighter colour. With freeze-drying the conditions of dehydration and the low final moisture content of the foodstuff render sulphiting unnecessary so far as protection against loss of ascorbic acid and development of browning are concerned, but it still seems to confer some slight advantage in colour to such vegetables as cabbage, Brussels sprouts and French beans.

The sulphite was applied as a spray to the vegetable emerging from the steam scalder; about 1,000 p.p.m. being required in the dry product, and this

requiring about 0·5 lb spray per pound of cabbage, the spray consisting of a solution of 0·3 per cent sodium sulphite and 0·05 per cent sodium carbonate (the latter to neutralize organic acids liberated by scalding, the presence of which converts the bright green chlorophyll to olive green phaeophytin).

After scalding the cabbage was spread on the drying trays, using expanded metal, and dried after evaporative freezing in about 9½ hr for 2 lb/sq. ft loading.

Brussels Sprouts

The varieties Cambridge Special and Sanda were mostly used, and the size limited to 1¼ in. diameter. During trimming the outer leaves were removed and a cross-cut made in the base of the sprout. This assisted scalding, drying and reconstitution and did not seriously impair the appearance of the product.

Washing, scalding and sulphiting were carried out as for cabbage; and when testing for peroxidase activity the samples were cut through to expose the centres. Expanded metal inserts were used and with a loading rate of 2 lb/sq. ft the drying time was 9 hr. Pre-freezing was not necessary.

Cauliflowers

Small quantities of cauliflower were dried, the curds ('flowers') being removed by hand and cut into small florets, carefully washed in water and steam-scalded until the peroxidase test was negative. The material was then loaded, with expanded metal sheets at the rate of 1½ lb/sq. ft, frozen by evaporative cooling and dried in 8½ hr.

French Beans

Both cross-cut and shoe-string pieces were used, with washing, scalding and sulphiting as for cabbage. Blast-freezing before drying was used, but it is not known how essential this step is. Loading at 2 lb/sq. ft on expanded metal gave a drying time of 9 hr.

Broad Beans

Broad beans with a tenderometer reading of 95 were used. After podding, the beans were pricked to pierce the tough testa, unpricked beans being difficult to dry and slow and irregular in reconstitution. After scalding the beans were loaded at 1½ lb/sq. ft on to expanded metal and dried in 8½ hr.

Peas

Peas with a tenderometer reading of 95 were shelled mechanically and sorted into three size grades by riddling through two meshes ($\frac{12}{32}$ and $\frac{14}{32}$ in.) to give 'small', 'medium' and 'large' peas. After grading they were washed and scalded in each size grade with no dosing solution. Limited tests with pricked and unpricked peas did not yield conclusive evidence as to the better product.

Peas could be dried either cooked or uncooked. In the former case they were held in the steam scalder for a period of about 60 seconds less than was required for a complete cook, as determined by a laboratory test. (Actual times were about 4 min for small grade, 7 min for medium grade and 9 min for large grade peas.)

Uncooked peas were also scalded before drying, for 1½ min. This product took 5–7 min to cook after reconstitution, but had the advantage of a slightly brighter colour than the instant pre-cooked peas.

No expanded metal was used, and after loading with 2 lb/sq. ft and evaporatively freezing, the drying time was 8 hr.

FRUIT

Little systematic work was done on fruit, but experimental batches of strawberries, raspberries, black currants, plums and apples were dried. Colour and flavour retention was excellent, but (as with frozen fruit) the texture was markedly affected. The product was quite satisfactory for use as a stewed fruit or as part of a made-up dish. A summary of the methods of preparation for fruits is given in Table 14.

Strawberries

The skin of the strawberry is relatively impermeable to water-vapour and successful freeze-drying was only achieved with cut strawberries. The clean fruit, with stobb removed, was cut longitudinally and laid on the drying trays, using expanded metal inserts, with the cut surface uppermost. After blast-freezing to −25°C the material was loaded into the drying cabinet; with a load of 1¾ lb/sq. ft, the drying time was 9 hr.

Raspberries

Raspberries do not appear to have as impermeable a skin as strawberries. Whole raspberries, with plug and calyx removed, were blast-frozen and with loadings of 1¾ lb/sq. ft the drying time was 8½ hr on expanded metal.

Black Currants

Black currants have a relatively impermeable skin, so after removal of the strigg they were hard frozen and then abraded in a domestic potato peeler with carborundum rollers. After loading on to expanded metal inserts on the drying trays they were re-frozen and at 1½ lb/sq. ft they were dried in 8½ hr.

Plums

Ripe fruit, after halving and stoning were laid, cut surface uppermost, on the drying trays and exposed to the fumes of burning sulphur for an hour before freezing. With loads of 1¾ lb/sq. ft on expanded metal the drying time was 8 hr.

Apples

It was found difficult to prevent liquid-phase drying if the smallest dimension of the cut apple was over $\frac{3}{16}$ in. Apple diced to $\frac{3}{16}$ by $\frac{5}{16}$ by $\frac{3}{8}$ in. was dried successfully. The cored, peeled apples were dipped in a 1 per cent solution of sodium sulphite for 15 seconds before dicing. The dice were loaded on to expanded metal at 2 lb/sq. ft and blast-frozen before drying in 9½ hr.

TABLE 13

Preparation of Vegetables for AFD Drying

Vegetable	Preliminary Washing	Peeling	Trimming	Cutting	Washing	Scalding	Cooking
Potatoes	Yes	Yes	Yes	Yes	Yes	Yes	No
Carrots, Parsnips, Turnips, Swedes	Yes	Yes	Yes	Yes	No	Yes	No
Beetroot	No	After cooking	Yes	Yes	No	No	Yes (First operation)
Celeriac	Yes	Yes	Yes	Yes	No	Yes	No
Cabbage	No	No	Yes	Yes	Yes	Yes	No
Spinach	No	No	Yes	Yes	Yes (before cutting)	Yes	No
Brussels Sprouts	No	No	Yes	Stems split	Yes	Yes	No
Leaf Celery	No	No	Yes	No	Yes	Yes	No
Celery Stalks	No	No	Yes	Yes	Yes (before cutting)	Yes	No
Cauliflower, Broccoli	No	No	Yes	Yes	Yes	Yes	No
Onions	No	Yes	Yes	Yes	Yes (before cutting)	No	No
Leeks	No	Cut into lengths	Yes	Yes	Yes (before cutting)	No	No
Peas	Yes	From viner	Culling	No	Grading	Yes	No
Green Beans	Yes	No	Yes	Yes	No	Yes	No
Broad Beans	No	Pods removed	Culling	No	No	Yes	No

Composite Food Items

The composite food items in the Experimental Factory were either dehydrated as individual foods and then mixed in the dried state or were dehydrated as composite items. For the first class of products use was made of non-AFD ingredients, such as spray-dried egg and mashed potato powder. It may well be that commercial development will produce numerous 'instant meals' and other 'convenience foods', and the examples that follow are merely reported as demonstrating the variety possible.

Meat Bars

Of products compounded in the dry state the first was the meat bar, which was conceived as a dual purpose item, primarily for use by the Services or by explorers, which could be eaten dry or, with a minimum of preparation, as a hot stew. It has been an outstandingly successful and popular product.

Minced cooked beef and minced cooked pork were prepared and dehydrated as described earlier. The fat and moisture contents of the minces were determined and then adjusted to 40 per cent and 7½ per cent respectively by addition of premier jus to the beef mince and flare fat to the pork, and water. The adjusted minces were blended in equal proportions and salt and monosodium glutamate added at the rate of 0·75 and 0·5 per cent respectively.

The blend was chilled to $-5°C$ to avoid loss of fat during compression and then pressed into blocks under a pressure of 1,800 lb/sq. in., with a dwell time of 10 seconds. The blocks had a density of 1 g/ml and were packed into triple laminates before they relaxed.

Variations on the basic formula gave the following bars:

Beef and pork (50:50)
Beef only
Pork only
Curried beef and rice (AFD cooked rice)
Steak and kidney (powdered AFD cooked kidney)
Veal and ham
Beef and vegetable (superseded by the stew described below)

Fish-cake Mixture

Either plain or smoked fish was dried and compounded as follows:

	Parts
AFD cooked cod	48
Mashed potato powder (commercial)	36
Onion flakes (commercial)	1
Parsley (commercial)	0·4
Salt	1·5
Pepper	0·1
Hydrogenated fat	13

After reconstitution the mixture was formed into patties and fried.

Sausage Meat

Attempts to dry sausages by the AFD process were unsuccessful. The reasons are not fully understood, but it is suspected that the high fat content enrobes the finely-divided constituents with a waterproof layer. It was possible,

TABL

Preparation of Frui

Fruit	Source	Preparation and nature of the portion used	Whether dehydrated by itself or mixed with other ingredients
Apricots	Spain South Africa	Ripe, stoned halves	By itself
		Dice	By itself, with sugar, with apple, with apple and sugar
		Pulp	Apple, sugar and pineapple with sugar, with sugar and apple, by itself
Gooseberries	England Scotland	Whole	Whole
Strawberries	England Scotland	Plugged whole Longitudinal halves	By itself
Raspberries	Scotland	Plugged whole	By itself
		Pulp	With sugar, with pectin, with guar bean gum, with cider pomace, with sulphured apple pomace
Black Currants	England Scotland	Whole strigged	By itself With sugar
Plums	England Scotland	Stoned, halves	By itself
		Dice	With sugar
Peaches	England Italy and France	Stoned, halves	By itself
		Dice	With sugar With sugar and apple With sugar, apple and ginger With sugar and ginger

14
for AFD Drying

Individual pretreatments	Combined pretreatments	Comments on drying conditions, etc.	Uses	Packaging and storage life
Blast-frozen, sulphured, steam scalded raw dried, ascorbic acid dip, or sulphite under vacuum	Sulphured and blast-frozen. Steam scalded and sulphured and blast-frozen	Removal of skin after scalding can assist drying	As stewed fruit	At least 3 years at 20°C with moisture content less than 4 per cent. Nitrogen pack not essential
Sulphured, ascorbic acid treated. Sulphite dip	Ascorbic acid and sulphite under vacuum	Essential for fruit to be ripe	As stewed fruit	At least 3 years at 20°C with moisture content less than 4 per cent. Nitrogen not essential
Raw dried. Steam scalded. Blast-frozen	Blast-frozen and abraded		As stewed fruit as for pie or tart. As stewed fruit, and for jam	Ditto
Raw dried. Steam scalded. Blast-frozen. Sulphured	Raw dried and sulphured. Sulphured and blast-frozen. Steam scalded and blast-frozen			
Raw dried. Steam scalded. Blast-frozen. Sulphured	Sulphured and blast-frozen	All shelf dried	As stewed fruit, for jam, as flavouring. As flavouring for sweet centres, jellies, drinks, etc.	
Raw dried, steam scalded, sulphured, blast-frozen, abraded	Blast-frozen and abraded		As stewed fruit and as a flavouring	At least 3 years at 20°C with a moisture content of less than 4 per cent nitrogen
Steam scalding, Sulphuring	Sulphuring and steam scalding	Dried by two-plate contact, etc.	As stewed fruit and as a flavouring	At least 3 years at 20°C in nitrogen and provided either a ripe gage or red or blue plum
Blast-freezing	Sulphuring and blast-freezing, steam scalding and sulphuring and blast-freezing			
Sulphuring, blast-freezing, steam scalding, skins removed (only if steam scalded).	Steam scalding and sulphuring and skin removal. With or without blast-freezing	Two-plate contact	As stewed fruit and for dry eating	At least 2 years at 20°C moisture content less than 4 per cent

TABLE I.

Fruit	Source	Preparation and nature of the portion used	Whether dehydrated by itself or mixed with other ingredients
Apples	England Scotland Australia	Peeled and cored Thick slices and segments	By itself By themselves
		Dice	By itself Plus brambles Plus apricots Plus pineapples Plus citrus pulp Plus citrus pulp and bananas All above with sugar Plus raisins Plus dates Plus bananas
Brambles	Scotland	Sorted	By itself Plus apple dice
		Juice Concentrate	By itself
Cranberries	Scotland U.S.A.	Sorted	By itself With apple As pulp
Rowanberries	Scotland	Sorted	As pulp
Rosehips	Scotland	Sorted	Halves Pulp
Chestnuts	Spanish	Cooked Peeled	With milk and butter and seasoning as a sauce. Plus sugar
Oranges	South Africa	Outer layer of rind and flesh as pulp	
	Jaffa	Outer layer of rind and flesh as pulp	Plus apple pomace Plus apple dice Plus apple dice and sugar
Lemons			Plus sugar Plus apple dice Plus apple dice and sugar
Grapefruit		Skinned, whole	Plus apple dice
Bananas		Skinned, whole Horizontal slices and apple, see under Apple	With apple and apricot With apple and citrus pulp
Dates	Iraq	Stoned. Whole Pulp	By itself. With roast ground peanuts With apple
Raisins	California Blue	As pulp	With apple By itself

—continued

Individual pretreatments	Combined pretreatments	Comments on drying conditions, etc.	Uses	Packaging and storage life
Steam scalding Sulphite dip Blast-freezing Ascorbic acid	Sulphite dip and blast-freezing Ascorbic acid and sulphite dip, with or without blast-freezing	Two-plate contact and shelf drying	As stewed fruit, pie filling, meringue pie filling and citrus pulp. With icing sugar followed by after drying as a fruit bar	
Blast-freezing Raw drying None		Shelf dried	As stewed fruit and pie filling	
Raw dry, Blast-freeze. Steam scald. Cooked		Shelf dried	As stewed fruit and pie filling and sauce	
Cooked. Blast-frozen. Sulphured. Cooked	Sulphured and blast-frozen	Shelf dried	For sauce. For making syrup	
None	None	Shelf dried		Nitrogen pack absolutely essential
None	None		As a drink Use as for apple mixtures	
None	None		Use as for apple mixture	
None	None		Use as for apple mixture	
Sulphured. Raw dried. Blast-frozen Steam scalded. Ascorbic acid dip. Sulphite dip, both under vacuum	Sulphured and blast-frozen. Ascorbic acid. Sulphite dip		Dry eating Dry eating	
None	None	Two-plate contact	As fruit bar and can be stewed	
None	None	Two-plate contact	As fruit bar and can be stewed	
		Two-plate contact	As fruit bar and can be stewed	

however, to prepare a sausage meat blend after dehydration. The beef sausage mixture was:

		Parts
Raw beef mince	} in suitable proportions	
Shredded suet	} to give 60 per cent fat	64
Rusk		32·5
Seasoning		3·5

The finished product had a fat content of about 40 per cent, which is lower than normal sausage meat, but more fat led to difficulties in reconstitution.

Cheese Omelette

An acceptable cheese omelette mix was made by grinding AFD minced cheese and then mixing it with an equal weight of spray-dried whole egg powder and 1 per cent salt.

Ham Omelette

Replacing cheese by ham in a similar formulation gave a ham omelette mix.

Beef and Vegetable Stew

This was prepared on a large scale from a stew dehydrated after preparation from the following ingredients:

Boned and trimmed beef in 2 in. by 1 in. by 1 in. cubes	225 lb
Carrot slices	110 lb
Potato dice	100 lb
Onion slices	70 lb
Salt	3¼ lb
Onion salt	15 oz
Pepper	2 oz
Monosodium glutamate	2 oz
Cornflour	4¾ lb
Water	as required

Cooking was carried out in steam-jacketted pans. The times at which the ingredients were added was, of course, of great importance so that all should be cooked at the end of the period. The meat required 2¾ hr, while the potato only required 15 min.

The quantities above were sufficient for a batch of 570 lb cooked stew, which was blast-frozen in moulds, and then sawn into 15 mm thick slabs, the yield being 480 lb. After dehydration this became 96 lb, a drying ratio of 5 : 1.

The sawn product was held frozen and then treated in a similar manner to raw beef steaks. Although the cabinet load (480 lb, i.e., 2 lb/sq. ft) was on the low side it took 11 hr to reduce the moisture content to 2 per cent. Thinner slices (10 mm) with the same tray loading dried in 7 hr, but this involved the use of smaller pieces of meat and vegetable, to the detriment of the attractiveness of the product.

Spaghetti in Tomato Sauce with Pork

The inclusion of a high percentage of pork in this item was intended to make the product a more substantial meal and to raise the protein content. The formulation was:

	lb
Spaghetti	37
Cooked pork cubes	100
Tomato sauce (commercial)	216
Tomato purée	20
Cheese (Cheddar)	2¼
Onion pulp (raw)	3¼
Butter	2¼
Salt	6½
Cornflour	2¼

The pork, after boning and trimming, was cut into cubes of about 1 in. edge, which were cooked in water with 1¾ lb of the salt, for about 40 min. They were then drained and cooled. The spaghetti was cooked for about 20 min in a minimum of water with the remainder of the salt, and then drained. The sauce was made from the remaining ingredients except the cornflour, the latter being made into a paste with a minimum quantity of the sauce. The final mixing took place thus:

The sauce was added to the strained spaghetti,

The pork was added to the spaghetti and sauce,

While simmering, the cornflour paste was added.

When thickened, the mixture was removed from the pan, poured into moulds and blast-frozen. The frozen blocks were sawn into slabs 15 mm thick and dehydrated.

The quantities quoted gave about 420 lb cooked material, which after sawing became 355 lb, yielding 72 lb of dried product (drying ratio 5 : 1).

The drying time was about 10 hr. As with the meat and vegetable stew the drying time could be reduced by sawing to 10 mm. This would dry inside 7 hr but again at the expense of particle size. With careful arrangement of the material in the moulds (notably the spaghetti, which because it lies along the long axis of the moulds gets sawn into very short lengths) it should be possible to deal with this problem. Another possible solution would be to freeze in multiple moulds of portion size and avoid the sawing operation (with its 16 per cent losses) altogether.

Cooked Rice Pudding

This material (and cooked sago pudding) was prepared to make a dessert course for a dehydrated pre-cooked meal. The formula used was:

Rice (Siam, Patna)	56 lb
Fresh milk	20 gal
Evaporated milk	56 lb
Sugar	21 lb
Salt	6 oz
Water	18 gal

The rice was washed in cold water and allowed to soak overnight. The soaked rice was then heated in a steam-jacketted pan with the salt and half the water. As the mixture thickened the milk and the remainder of the salt were added. When fully cooked (a period of about 90 min) the sugar was added and the pudding thoroughly mixed.

These quantities yielded about 445 lb rice pudding, which after pouring into moulds, blast-freezing and sawing into 12·5 mm slabs became 395 lb. The yield of dehydrated product was 93 lb, a ratio of 4·3 : 1, and the drying time was 8 hr.

Chicken Suprême

The more expensive luxury foods can presumably carry additional processing charges more easily than cheaper foods, and it may well be that they will be included among the first freeze-dried foods to be put on the market. It is almost inevitable that they will include chicken dishes. Several of these were tried out on a pilot scale in the Experimental Factory, and for Chicken Suprême the following recipe was used:

Four medium size chickens	white flesh only
Butter	4 oz
Flour	4 oz
Stock (from cooking the chickens)	2½ pints
Pepper	1 g
Salt	¾ oz
Parsley (chopped)	trace

The chickens were cooked in water until the flesh was loose on the bones. The white meat was picked off and the liquor from the cooking was separated from the fat by centrifuging. The sauce was made from the separated liquor, butter and flour, with the addition of seasoning. The chicken pieces were now added to the sauce, and finally the parsley as a garnish.

The prepared dish was now poured into a mould, blast-frozen, and sawn into 15 mm slabs, which dried in 10½ hr. The drying ratio was 3·6 : 1.

Curried Chicken

Like all curries this product is difficult to prepare to suit all palates. However, a recipe acceptable to a high percentage of people who liked curry dishes consisted of:

Cooked chicken pieces (white)	2½ lb
Stock (from cooking chickens)	2 pints
Butter	3 oz
Flour	3 oz
Onions (sliced)	3
Apples (cooking, sliced)	2
Tomatoes (skinned and sliced)	4
Dates (chopped ¼ by ¼ in.)	2 oz
Desiccated coconut	½ oz
Mango chutney (chopped ¼ by ¼ in.)	½ oz
Curry powder	2½ oz
Salt	¾ oz
Lemon juice	5 ml

The sauce was made by cooking the onions in the butter, along with the curry powder and the flour; the stock was added carefully with stirring and the salt, dates, coconut and tomatoes added before cooking. When cooking was almost complete the chutney and apples were added. It was important not to over-cook the apple as the texture of the reconstituted material was better retained after reconstitution if it was somewhat under-cooked. The cooked chicken and the lemon juice were added just prior to removal of the product for blast-freezing. Fifteen mm slabs dried in 9½ hr with a drying ratio of 3·75 : 1.

CHAPTER XI

Storage behaviour of dehydrated foods

PROBABLY the main reason why dehydrated foods gained so poor a reputation in the Second World War was because in general there was insufficient knowledge about the factors which led to their deterioration during storage and, where such knowledge existed, factory techniques were insufficiently developed to take advantage of it. During the past decade, however, there has been a considerable increase in our basic knowledge and there have been corresponding advances in processing, notably in the development of the accelerated freeze-drying system, which can bring foodstuffs to extremely low moisture contents—a matter of great importance, as will be shown below.

The first requirement for the adequate packaging of dehydrated foods for storage is, obviously, to keep them dry and thereby prevent microbial spoilage, but even when moisture is barred from access to the food, deterioration can take place by chemical reactions between the constituents of the food itself or between constituents of the food and oxygen, if it be present. The oxidative reactions may be considered first.

OXIDATIVE DETERIORATION

Oxidative rancidity of fats is produced by the autoxidation of the unsaturated compounds, probably by the addition of molecular oxygen to the double bonds of the unsaturated acids, with the production of labile peroxides and ultimately aldehydes, ketones and short-chain acids. Unsaturated fats ('soft' fats) such as those of lean pigs or marine animals, are more readily oxidized than are more highly saturated fats.

The degree of oxidation which has occurred in the fat may be expressed in terms of the peroxide value, which is defined as the number of milliequivalents of peroxide per 1,000 g of fat.[24] In the early stages, the higher the peroxide value the more rancid the fat and experience with several meat products suggests that rancidity is virtually undetectable if the peroxide value of the fat is below 4, and that few people can detect it below the value of 8. Meat products, of course, vary widely in their fat contents but the above seems to hold good over a wide range.

As a general rule dehydrated meats will become noticeably rancid within a week or two of exposure to the air; and if cured, e.g., ham or bacon, even more rapidly. With the development of severe rancidity in meat fats there is alteration in colour to yellow or yellowish green. Oxidation also leads to bleaching of the muscle pigments, the pink oxymyoglobin of freshly freeze-dried beef being first rapidly oxidized by atmospheric oxygen to the brown metmyoglobin. (Even when the meat is stored in nitrogen, however, metmyoglobin develops from oxymyoglobin, and it appears that the ferrous iron is losing an

electron to some electron acceptor present in the freeze-dried meat itself.)[25 26]

When fish is stored in air it develops pungent odours, which are probably from volatile products of the oxidized fish oils. Recent studies at the Hormel Institute[27] have shown that among such volatile compounds are short-chain aldehydes, such as formaldehyde, acetaldehyde and propionaldehyde; ketones, such as acetone, butanone and pentanone; and alpha and beta unsaturated monocarbonyls, glyoxal, and low molecular weight alpha-ketonaldehydes and diketones.

In certain plant materials there are sufficient fats for rancidity to develop. Potatoes, for example, contain only about 0·001 per cent of fat,[28] but this is thought to be responsible for the staling (a definitely rancid odour and flavour) of dehydrated potatoes which have been stored in air.

The carotenoid pigments readily suffer oxidation and this is particularly noticeable in the case of dehydrated carrot, in which the pigment responsible for the colour of the tissue (β-carotene) suffers degradation, ultimately becoming bleached; and it has been stated that β-ionone is the major product of oxidation.[29] Coincident with the oxidation of β-carotene there develop perfume-like odours and flavours, but it has not yet been definitely established that there is a causal connection. β-carotene is provitamin-A and oxidative destruction of this pigment therefore implies loss of nutritive value of the carrot as a source of vitamin A.

Most green vegetables develop a hay-like odour when stored in air, though the substances and reactions involved are not known. There is also rapid loss of ascorbic acid (vitamin C).

Some, at any rate, of the oxidative reactions are more rapid at lower moisture contents. Dehydrated potato of 10 per cent moisture content may be stored in air for upwards of 6 months without developing rancidity, while potato of 2 or 3 per cent moisture content will become rancid in 2 to 3 weeks. Carrot of 8 or 9 per cent moisture content will not develop oxidized flavours nearly so quickly as materials of low moisture contents. On the other hand these high moisture contents accelerate the development of the browning reaction which will be described below.

The reason why low moisture contents favour oxidation is not fully understood. Uri[30] has recently suggested that it may be associated with the catalytic effect of heavy metals on the free-radical chain reaction that is an essential part of the development of oxidative rancidity. He has shown that in dehydrated potato trace metals are present in sufficient quantity to catalyse such reactions, and has put forward the hypothesis that there is a correlation between the ease of electron transfer leading to free radical formation and the polarity of the environment. At a certain stage of the dehydration process, trace metals would be denuded of their hydration shells and their catalytic activity in the initiation of oxidative rancidity would then set in abruptly.

There are a few dehydrated vegetables which do not suffer rapid loss of culinary quality when stored in air, even when their moisture contents are low. These are peas, French beans, beetroot and onions. As already mentioned, however, where ascorbic acid is present it is lost rapidly.

The incorporation of antioxidants into vegetable and fruit tissues is difficult because most antioxidants have exceedingly low solubilities in water; it is, therefore, virtually impossible to get them in contact with the minute quantity of fatty constituents in the plant cells. Partial success has been achieved in

the case of mashed potato powder and potato flakes, in which the antioxidant may be incorporated during the mashing process which precedes dehydration.

Even with meat the addition of antioxidants has been attended with only limited success. The first problem is to introduce the antioxidant into the tissue, which is virtually impossible except in the case of cooked minces, where it can be added to the cooking water.

Less is known about the liability of dehydrated fruit to oxidation. Wherever ascorbic acid is present it will rapidly diminish, but many fruits seem to retain their culinary quality for 6 months or more when packed in air, though systematic knowledge is lacking.

The only really practicable protection against oxidation is packing in the absence of oxygen; this may be achieved by packing in vacuum, in nitrogen (or other inert gas), or by filling the container so completely with the compressed dehydrated product that there is virtually no headspace remaining. This latter technique can only be applied in exceptional cases, e.g., compressed blocks of dehydrated carrot. The essential feature here is that the amount of oxidation that does take place is insufficient for the resulting taint to be detected by taste panel assessment. On this basis it is possible for oxygen-tolerances to be determined for various foodstuffs but, in fact, there is very little information on this, though a maximum figure of 0·14 mg oxygen per gram of dehydrated carrot has been found at Aberdeen, and Sharp[31] has indicated a figure of 0·07 mg oxygen per gram dehydrated meat (cf. Table 7 for more approximate figures for a range of foodstuffs).

It was mentioned above that certain vegetables could be stored in air with little deterioration of culinary quality; there may be others, and there may be fruits which also are not seriously affected by oxidation, but it is a wise general principle to regard all dehydrated foods as liable to oxidative deterioration, and to pack them accordingly, unless there is ample proof to the contrary.

In some senses a more serious problem than the rancidity of fat is the influence of oxygen on the class of chemical reactions generically known as browning reactions. In the case of well packaged dehydrated meats these may produce changes which in the early stages are far from unpleasant (resembling the flavour produced by roasting meat) although they lead eventually to quite unacceptable bitterness and staleness. If oxygen is present, on the other hand, the reactions are quite different and extremely unpleasant flavours are produced in remarkably short time. This phenomenon, which takes place only in the presence of both protein and fat, is not inhibited by the usual range of antioxidants.

NON-OXIDATIVE DETERIORATION

During prolonged storage even at relatively low temperature there is slow deterioration in the culinary quality of most dehydrated foods; the tissue becomes tougher, the capacity to take up water on reconstitution diminishes, particularly in the case of certain types of meat, and the flavour becomes stale. The nature of these changes is not well understood. At temperatures below 0°C the changes are extremely slow, and vegetables have been held virtually unchanged for periods of about 10 years at −10°C. It cannot be

said, however, that dehydrated foods can be kept indefinitely, but all the recent work on accelerated freeze-dried foods (and for that matter on air-dried foods of low moisture content) shows that nearly all will keep for periods well in excess of 2 years, with virtually no detectable change in culinary quality, and the indications are that most will remain acceptable for periods of up to 5 years or more, when they are stored under temperate conditions, i.e., about 18°C (65°F) provided always that they are in moisture-proof containers and in the absence of oxygen. (It should also be noted that light has a detrimental effect on many dehydrated foods, causing bleaching of chlorophyll in green vegetables and of the pigments of meats. The container should, therefore, be opaque.)

With increasing temperatures the storage life of dehydrated foods is reduced, being determined by the development of a brown discoloration, which is largely the result of a complex series of chemical reactions initiated by the condensation of reducing sugars and amino acids or proteins, the initial products being colourless, but later products being brown. This series of reactions is commonly termed the Maillard or the browning reaction. The development of a bitter flavour is associated with this brown discoloration; in meat this is not unpleasant in the early stage of browning and is like that of a meat extract or gravy, but in vegetables and fruit it is not appreciated.

There is a vast amount of literature on the chemistry of the browning reaction: some has been summarized by Ross[32] and various authors (e.g.,[33]) have described possible pathways of reaction, notably Hodge.[34] The first steps in the Maillard reaction appear to be the condensation of aldoses with amino-groups, and recent work by Anet and Reynolds[35,36,37] in Australia confirm the earlier suggestions that the glycosylamines so formed undergo the Amadori rearrangement to give ketose amines, which react further with aldoses to give di-ketose amines that in turn decompose, rapidly regenerating ketose amines and giving a reactive carbonyl compound. The steps by which dicarbonyl compounds produce the so-called melanoid pigments are still being studied.

Along with the browning reaction there occur other forms of deterioration. In vegetables there is loss of vitamin C, and structural changes leading to incomplete reconstitution and toughness in the cooked products. In meats a number of changes have been observed in detail; reducing compounds and fluorescence increase; free amino groups, soluble protein and non-protein nitrogen decrease, as do free glucose and mannose; papain digestibility decreases; haematin pigments become less soluble, and the colour changes from light pink to brown.[38] In addition there is reduced uptake of water on reconstitution. All of these changes are good criteria for carbonyl-amine browning, and it has been suggested that carbonyl-amine browning is the main and probably the only significant non-oxidative deteriorative reaction. There is also rapid loss of thiamine (vitamin B_1), but all the other B vitamins remain unchanged, and there is virtually no loss of biological value of the protein. Thus, in meat products, except for diminution in the content of thiamine, there is little, if any, loss of nutritive value.

In all foodstuffs, generally speaking, conditions that favour browning favour the other forms of degradation and vice versa. The extent of browning is relatively easy to study and the factors that affect the rate of development of browning have, therefore, received the greatest attention. The assumption

has been made that the other forms of degradation are affected in the same manner. The first of these conditions is:

Temperature

The reduction in storage life (i.e., acceleration of browning) with increasing temperatures of storage is striking. A dehydrated food which will keep for

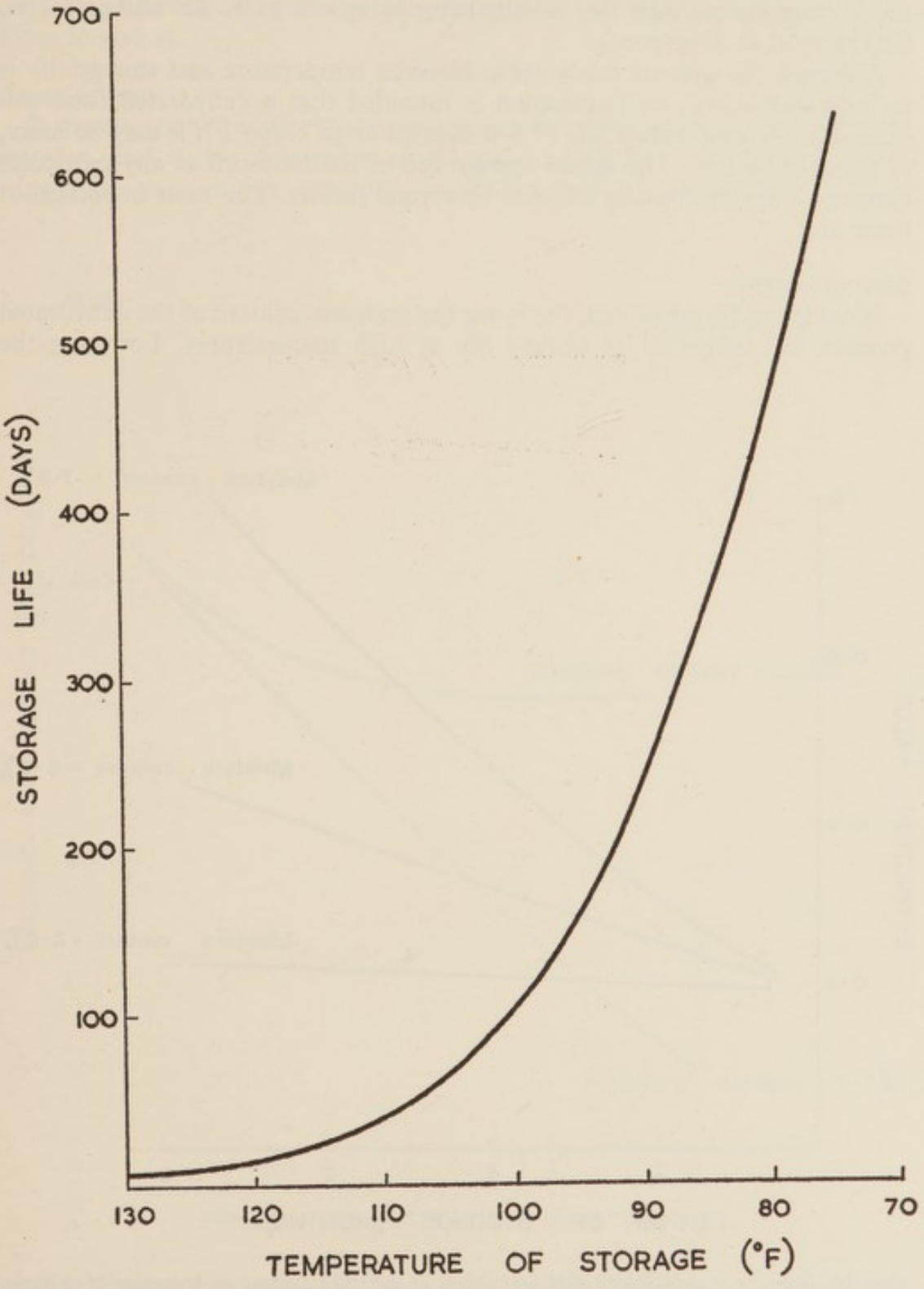


FIG. 9. *Dehydrated vegetables: Effect of temperature on storage life*

about 2 years at 16–21°C (60–70°F) may be expected to last for a year or 18 months at a steady temperature of 27°C (80°F) before becoming so brown and bitter as to be inedible. At 32°C (90°F) this stage would be reached in 6–8 months, at 38°C (100°F) in about 3 months, and at 49°C (120°F) in about 2 weeks. The decrease in storage life at temperatures above 21°C (70°F) is very rapid and storage life at 49°C (120°F) is liable to be very short indeed (Fig. 9, page 113). Fortunately even tropical temperatures are rarely of this order; the average temperature lies usually between 27 and 32°C (80° and 90°F) (as, for example, at Singapore).

Although the general relationship between temperature and storage life is as indicated above, no implication is intended that a dehydrated food will necessarily have a storage life of 6–8 months at 32°C (90°F); it may be more, or it could be less. The actual storage life of the foodstuff at any particular temperature is profoundly affected by several factors. The most important of these are:

Moisture content

If oxidation be prevented, the lower the moisture content of the dehydrated product the longer is its storage life at high temperatures. Lowering the

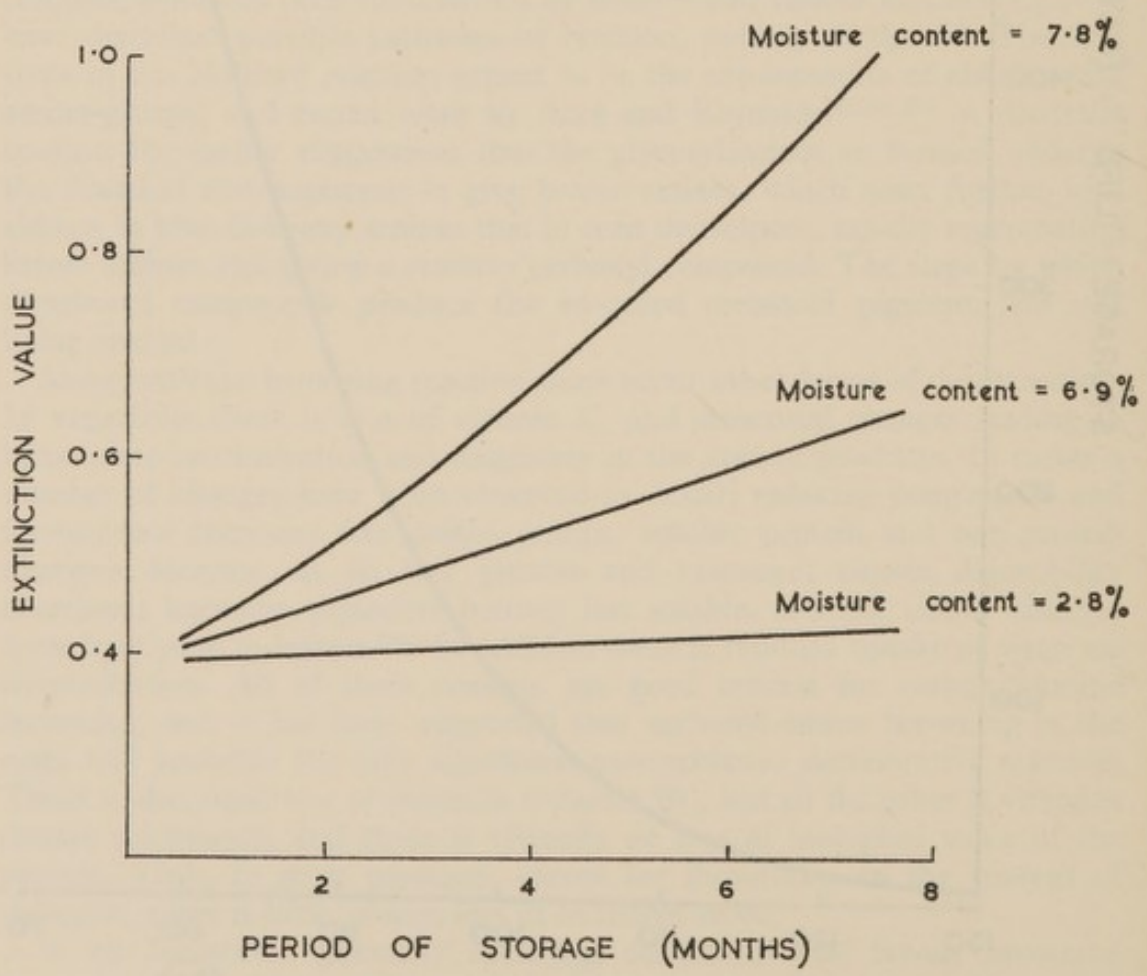


FIG. 10. *Storage of dehydrated cabbage: Effect of moisture content on browning of material stored in cans in nitrogen at* 37°C

moisture content of cabbage, for example, from 5 per cent to 3 per cent will double its storage life at 37°C (98·6°F). Accelerated freeze-drying gives considerably lower moisture contents than were thought possible a few years ago, and most foods can now be packed with moisture contents of 1 per cent or 2 per cent, with a three- or four-fold increase in tropical storage life compared with air dried foods of 5 per cent or 6 per cent moisture content. Some actual experimental results for cabbage indicating the effect of moisture content on browning and on the retention of ascorbic acid are shown in Figs. 10 and 11.

Sugar content

It was mentioned earlier that reducing sugars were involved in the browning reaction, and it therefore seems obvious that in those cases where reducing sugars are the limiting reactants, browning would be prevented by their

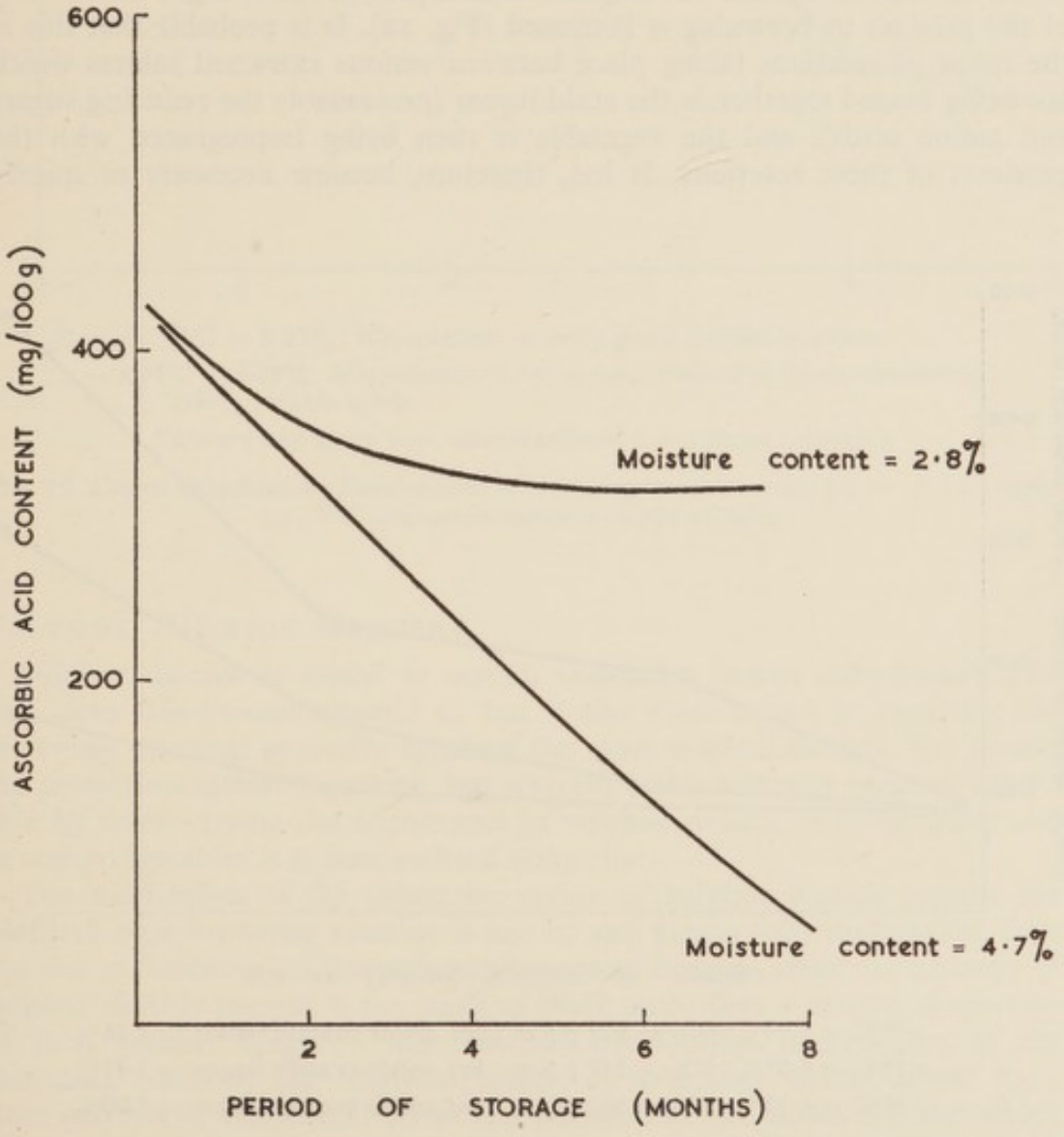


FIG. 11. *Storage of dehydrated cabbage: Loss of ascorbic acid (vitamin C) from material of 2·8 per cent and 4·7 per cent moisture content, packed in nitrogen and held at* 37°C

removal (Fig. 8). In the case of potatoes considerable progress has been made in this direction. Potatoes harvested mature have lower sugar contents than immature potatoes.[21] During subsequent storage the reducing sugar contents of the potatoes will rise if the tubers are exposed to temperatures below about 6°C (43°F) so that for minimal reducing sugar contents it is therefore essential for potatoes to be harvested mature, and then stored indoors, with minimum temperatures not less than 6°C (43°F) (with the use of a sprout depressant to inhibit the sprouting, which is likely to occur at this temperature) [39,40]. Eggs may be stabilized by treatment with glucose oxidase before dehydration; and similar treatment has been applied experimentally to minced cooked meats, but since even without this treatment their storage life is so satisfactory, this is generally regarded as an unnecessary complication.

Methods of scalding

In vegetables which are scalded in water in continuous or serial scalds before dehydration, it has been found that if the concentration of the solutes which have been leached out into the scald liquor becomes high, the liability of the product to browning is increased (Fig. 12). It is probable that this is the result of reactions taking place between various extracted solutes which are being heated together in the scald liquor (presumably the reducing sugars and amino acids), and the vegetable is then being impregnated with the products of these reactions. It has, therefore, become necessary to specify

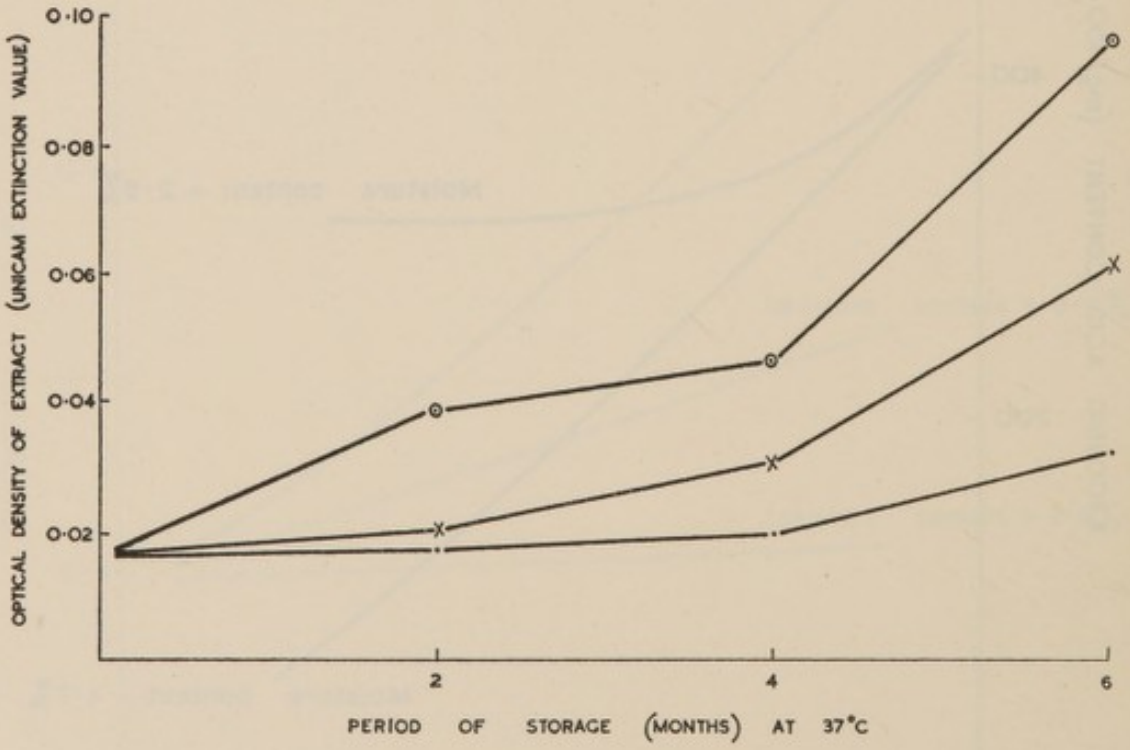
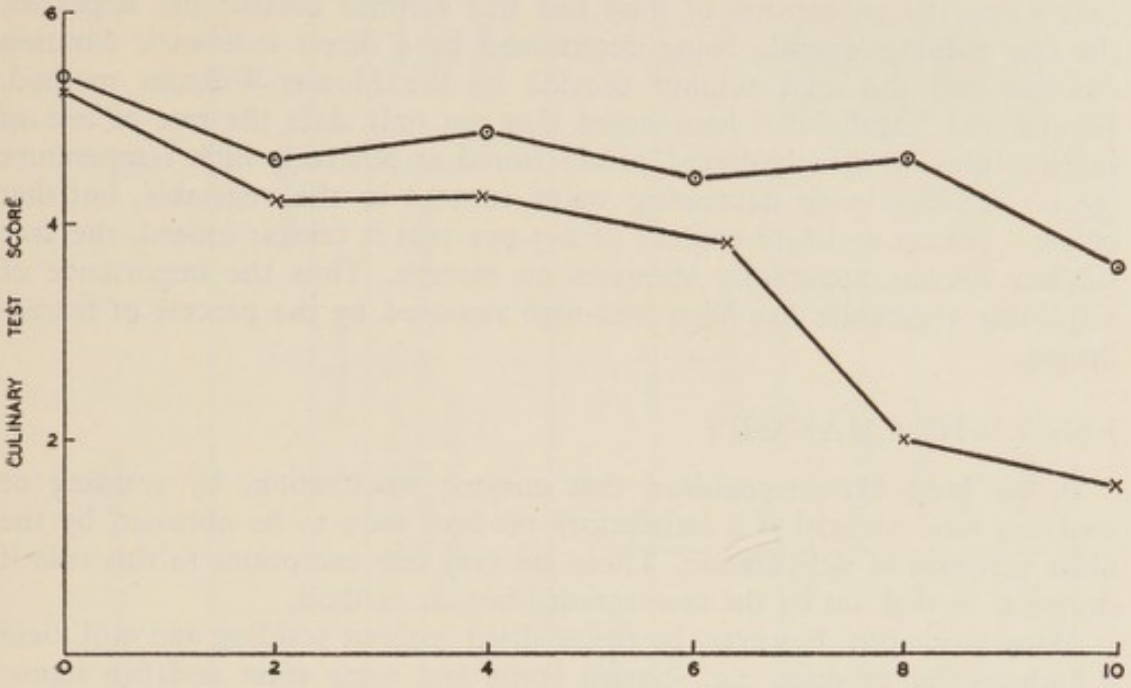


FIG. 12. *Carrot dehydration: Development of browning in dehydrated carrot strips* ($\frac{3}{16} \times \frac{3}{16}$ in.) *stored at 37°C; carrot scalded in low, medium and high concentrations of soluble solids in the scald liquor*

the maximum levels of soluble solids concentration to be allowed in the scald liquor. This effect is particularly important in the case of carrot and a soluble solids concentration of about 3 per cent is recommended. The problem does not arise when steam scalding is used[41] (Fig. 13).



⊙ — ⊙ M/C = 5·33%: SO_2 content = 544 p.p.m.: scalded in steam
x — x M/C = 5·29%: SO_2 content = 441 p.p.m.: scalded in liquor containing 2·41% soluble solids
Culinary test score 6 = natural colour: 0 = intense browning

FIG. 13. *Carrot dehydration: Development of browning in carrot strips* ($\frac{3}{16}$ = $\frac{5}{16}$ in.) *stored at 37°C after either steam or water scalding*

SULPHUR DIOXIDE CONTENT

Sulphur dioxide is added to certain vegetables before dehydration. Not only does this protect vitamin C, but it also plays a part in retarding the browning reaction: generally speaking the more sulphur dioxide, the greater the protection against browning, but with the lower moisture contents attainable by freeze-drying the importance of sulphur dioxide is diminishing and in many vegetables it is now omitted altogether.

The mechanism of the protective action of sulphur dioxide against the Maillard type browning reaction is not by any means fully understood. It is not due to 'blocking' of the reducing groups of hexoses, since the quantity of sulphur dioxide present is too small to block more than a minute proportion of such groups. Wager[42] has obtained a colourless intermediate of the browning reaction, (an isoglycosylamine of molecular weight about 235), from extracts of dehydrated carrot and potato, and has shown that this yields brown pigments when heated at 52°C and 30 per cent R.H. Further chromatographic studies have suggested that when sulphite was present a bisulphite compound was formed in relatively large amounts. The carbonyl compound

derived from it browned less readily than the isoglycosylamine isolated previously and gave rise to a number of fragments some of which reduced silver nitrate and some of which reacted with ninhydrin.

During storage sulphite disappears from dehydrated vegetables, i.e., it can no longer be detected by the usual analytical techniques. A considerable amount of literature (e.g.,[43]) has been devoted to this phenomenon. In this connection the conception of total and free sulphur dioxide has appeared, the free sulphur dioxide being determined by a direct iodimetric titration method, and the total sulphur dioxide by the Monier-Williams method. Hearne and Tapsfield[44] have noted that not only does the rate of loss of sulphur dioxide in dehydrated potato stored at relatively high temperature (38°C) diminish with decreasing water content in the vegetable, but that below a critical moisture content of 2–3 per cent it ceases; indeed, the free sulphur dioxide apparently increases on storage. Thus the importance of sulphiting vegetables has been well-nigh removed by the process of freeze-drying.

ENZYMIC CHANGES

It has long been recognized that enzyme inactivation, by scalding or cooking, was essential if a satisfactory product were to be obtained by the older methods of dehydration. There are very few exceptions to this rule if drying is carried out by the conventional hot-air method.

Many foods can, however, be freeze-dried without scalding and still yield a high-quality product, e.g., certain fruits and some meat and fish items. There is evidence that enzymes are not destroyed by the accelerated freeze-drying process, although they are largely inactivated in the absence of water. However, even at the low final moisture contents of these foods (1–3 per cent) some enzymic reactions may take place. Precisely what effects these reactions have on the storage behaviour of the foods is not known for certain, but freeze-dried raw meats, for example, have a substantially shorter storage life than their cooked counterparts, particularly at elevated temperatures, when their liability to browning is also increased, possibly owing to enzymic liberation of glucose or other reactant which is then able to take part in a Maillard reaction.

STORAGE TESTS

A discussion of the storage behaviour of dehydrated foods would be inadequate without some mention of the techniques used for storage tests. This is a considerable subject and only the briefest outline can be given here. The approach most commonly used at the Experimental Factory followed that of the pioneer work of Tomkins[29] and his colleagues, though refinements were introduced into the methods of assessing the reliability of panels and the validity of their results.[45] Samples of the foodstuffs to be compared, differing only in the characteristic under investigation, were packed in whatever manner was most appropriate (usually in nitrogen in sealed cans unless it were the packaging itself that was under investigation) and placed at various temperatures of storage. Commonly used temperatures were −50°C (−20°C for meats) for the control samples, 18°C or 25°C to represent temperate storage, and 37°C to simulate rather severe tropical conditions. At intervals

TABLE 15

Quality Assessment of Dehydrated Cod Fillets

APPEARANCE

1 Odour		2 Colour		3 Opacity		4 Structure			
						Flakiness		Texture	
Fresh boiled cod	0	Curdy white	0	Opaque	0	Flaky, as fresh fish	0	Hard	0
Odourless	2	Appreciable off-colour	4	Translucent	2	Separates partially into flakes	2	Firm, as fresh fish	2
Slightly off-odour	4	Very marked off-colour	8	Transparent	4	Does not flake	4	Soft	4
Appreciable	6							Mushy	6
Marked	8								

EATING QUALITIES

5 Intensity of Fish Flavour		6 Taints		7 Tenderness		8 Juiciness		9 Acceptability	
Full	0	Nil	0	Tender	0	Juicy	0	Dislike extremely	1
								Dislike very much	2
Weak	2	Slight	2	Neither tender nor tough	2	Neither dry nor juicy	2	Dislike moderately	3
								Dislike slightly	4
Completely masked	4	Appreciable	4	Slightly tough	4	Slightly dry	4	Neither like nor dislike	5
								Like slightly	6
or flavourless		Marked	6	Tough	6	Dry	6	Like moderately	7
								Like very much	8
		Inedible	8	Very tough	8	Very dry	8	Like extremely	9

samples were taken from each type of storage and examined in the laboratory and by a trained taste panel, who recorded their observations in the form of a score on a sheet especially designed for the particular commodity. A very considerable amount of information was obtained from tests of this kind, and it was usually possible to graph results in such a way that trends of changes could be observed over long periods. However, when several variables were being investigated the number of samples involved, and the amount of time spent in repeated examinations of the samples during the period of storage were a considerable strain on resources. For many purposes it was possible to devise simple tests which were less time consuming. For example, it was not always necessary to follow trends and a single examination after, say, 3 months was sufficient to establish a point.

It is important to bear in mind that a small analytical taste panel, as used in the Experimental Factory, cannot judge the acceptability of a product to any prospective consumer. Their task is to assess the extent of changes independently of preference. Even so, it is sometimes necessary to be able to give some idea of the stage at which a product would become unacceptable, and it is necessary to use the judgment of the taste panel in such cases, with reservations. They may be asked to use a hedonic scale with answers ranging from 'like very much' to 'dislike intensely' (see Table 15), or they may, in discussion of a particular score sheet, agree that a rating below a certain figure for particular characteristics would make them as individuals reject a sample (examples of score sheets are given in Tables 15, 16, 17 and 18).

TABLE 16

Score Sheet for Dehydrated Carrot

APPEARANCE (GENERAL)

1. Strips more than 1 in. long
2. Strips somewhat fragmented ½ in. to 1 in. long
3. Strips less than ½ in. long
4. Strips fully reconstituted (surfaces not concave)
5. Strips not fully reconstituted (surfaces concave)
6. Surface slimy
7. Surface not slimy

COLOUR

8. Reddish orange
9. Yellow orange
10. Yellow
11. Bright
12. Dull
13. General 'washed out' appearance
14. Grey discoloration (No. of strips in tenths)
15. Brownish discoloration (No. of strips in tenths)
16. Green discoloration (No. of strips in tenths)
17. Pale
18. Medium } Intensity of colour
19. Dark

NATURAL FLAVOUR

20. Full carrot flavour
21. Weak carrot flavour
22. Flavourless
23. Slight sweet flavour
24. Strong sweet flavour
25. Slight earthy flavour
26. Strong earthy flavour

FOREIGN OR 'OFF' FLAVOUR

27. 'Perfume'
28. Bitter
29. Scorched
30. Sulphite
31. Other foreign flavour (specify)
32. Weak
33. Moderate } Intensity of foreign flavour
34. Strong

TEXTURE

35. Firm, tender and slightly crisp
36. Firm and tender but not crisp
37. Some strips soft but not mushy
38. Generally soft but not mushy
39. Some strips mushy
40. Generally mushy
41. Some strips hard
42. Generally hard
43. Some strips rubbery
44. Generally rubbery
45. Texture gritty
46. Texture slimy

SPECIFICATIONS

The practical application of the knowledge gained about the storage behaviour of dehydrated foods is, of course, applied in the devising of specifications for the products and their packaging, and in the preparation of recommendations for their storage. Ideally, these foods should be prepared to the lowest possible moisture content and packed in sealed containers in inert gas; and exposure to high temperatures of storage should be avoided. The ideal is, of course, usually impracticable and a manufacturer has to decide his specifications in accordance with the use to which the food has to be put, and the conditions in which it is likely to be stored. Specifications had been drawn up for the air-dried foods which were supplied to the Services, but there were no specifications available for freeze-dried foods. The general principles were, however, clear; products to be used in the tropics must be of the lowest possible moisture content and exposed to a minimum of oxygen, i.e., usually in nitrogen or vacuum; where reducing sugars can be controlled these should be low; due attention should be paid to scalding conditions. If

TABLE 17

CARROT

Score Sheet for Storage Tests 34 *and* 35

1. ODOUR
   - 6 Pronounced natural odour
   - 5 Weak natural odour
   - 4 No odour
   - 3 *Off-odour just detectable
   - 2 Marked off-odour*
   - 1 Unacceptable on grounds of odour

   *Foreign odour must be described

2. COLOUR
   - 6 Natural bright reddish orange
   - 5 Slight dulling of natural colour
   - 4 Slight discoloration of paler parts
   - 3 Marked discoloration of paler parts
   - 2 Overall discoloration
   - 1 Excess discoloration—unacceptable

3. FLAVOUR
   - 6 Natural carrot flavour
   - 5 Slight loss of natural flavour: sweet
   - 4 Sweet but otherwise flavourless
   - 3 Slight caramel flavour or perfumed
   - 2 Marked caramel flavour or perfumed
   - 1 Inedible

4. TEXTURE
   - 6 Firm and tender
   - 5 Rather soft
   - 4 Slightly hard but not rubbery
   - 3 Slightly rubbery
   - 2 Definitely rubbery
   - 1 Extremely rubbery

5. RECONSTITUTION
   - 3 Complete surfaces (surfaces not concave)
   - 2 Surfaces slightly concave
   - 1 Surfaces markedly concave, strips wrinkled

a storage life of more than 6 months or so in a temperate climate is required, moisture contents should still be relatively low (in the region of 5 per cent), and packing in an inert gas or vacuum is required. For certain dehydrated vegetables and fruits with a quick turnover in a temperate climate it may be possible to pack in air without serious deterioration, if the moisture content of the foodstuff is relatively high. If the food is to be used within a very few weeks after manufacture it may be possible in a few instances (e.g., potato) to pack material of this kind in multi-wall or plastic lined paper sacks, which will give reasonable protection from uptake of moisture vapour for a period of a few weeks. It must always be remembered, however, that dehydrated foods are liable to oxidative and high temperature deterioration, and these dangers should be recognized and avoided on the basis of the knowledge outlined above.

TABLE 18

Score Card for Dehydrated Meat Panel

TEXTURE

Size of particle		Toughness		Juiciness		Grittiness	
Coarse	0	Tender	0	Juicy	0	No grit	0
Medium	2	Slightly tough	2	Slightly dry	2	Slightly gritty	2
Fine	4	Tough	4	Dry	4	Appreciably gritty	4
Mushy	6	Very tough	6	Very dry	6	Very gritty	6

FLAVOUR

Intensity of Meat Flavour		Rancidity		Other taints		Comments
				Intensity	Description	
Full	0	Nil	0	Nil	0	
Medium	2	Slight	2	Slight	2	
Weak	4	Appreciable	4	Appreciable	4	
Very weak or entirely masked by other flavours	6	Very strong (Inedible)	6	Very strong (Inedible)	6	

RECOGNITION OF DETERIORATION

Most of the signs of deterioration have already been mentioned, but it will be useful to recapitulate. How can one tell whether a sample of dehydrated food has gone off and is not suitable for human consumption? With regard to meat there are three extreme cases when the meat should be rejected. One is when the meat has gone rancid due to a leaky can or pouch. Rancidity is easily recognized by the smell and by the yellow-green colour of the fat and meat. The second case arises when meat has been kept well past its normal storage life and has developed an orange-brown colour and a very unpleasant burnt smell. Such meat is not harmful, but is certainly unpleasant to eat. The third possibility is that of actual mould or bacterial growth, which would only happen when the meat has been exposed for a long time in a humid atmosphere. The off smell associated with deterioration

of this kind is probably the most reliable guide: such meat should on no account be eaten. It is potentially as dangerous as fresh meat in a similar state of decay.

In the case of vegetables, as has already been mentioned, the smell is a useful guide. Rancid, strongly perfumed, or strong hay-like odours show that oxidation has taken place, and a caramel-like odour is associated with severe browning due to long exposure to high temperatures. Bleaching of carrots indicates oxidation; browning of any vegetable indicates heat damage. In none of these cases is the vegetable harmful to eat, but it will be unpalatable and it will have lost some of its nutritive value. In extreme cases of exposure to damp air vegetables may become mouldy or develop a fetid odour and they are then quite unfit for use.

Briefly, then, freeze-dried foods are safe to eat even beyond the stage when they cease to be palatable and attractive. Once they have been reconstituted, however, they are as perishable and vulnerable as the original raw materials.

CHAPTER XII

Reconstitution and cooking

In preceding chapters the production of AFD foods has been described, and it has been emphasized that the objective is for the food, when served, to be as nearly as possible indistinguishable from the fresh, after the processes of reconstitution and cooking.

The terms 'reconstitution' and 'rehydration' are sometimes regarded as synonymous but the former really implies rather more than the latter, embracing recovery of form and texture and not merely the uptake of water. For this reason it is the term most generally used when referring to the preparation of dehydrated foods for eating. Adequate reconstitution is, of course, essential if the foodstuff is to have satisfactory eating quality and one of the essential features of routine quantity control should be an organoleptic test in which this is assessed. Less essential in routine testing, but imperative during research and development, are measurements of the extent of rehydration—the quantity of water taken up by a foodstuff during reconstitution.

The quality of the cooked product is the final measure of the success or otherwise of preceding operations: the rehydration of the foodstuff is only one aspect of this. The appearance of the foodstuff, its flavour and its texture must all be up to the highest standards, and a full evaluation of these qualities is an essential routine for factory control. In this chapter the operation of such tests is described in some detail.

LABORATORY MEASUREMENT OF REHYDRATION

For most AFD products the measurement of rehydration is a relatively simple matter, though for some items, e.g., mashed vegetables or composite items such as spaghetti and tomato sauce, accurate determination of rehydration is more difficult. The general principle is that the samples are immersed in water (if necessary held under the surface by light pressure, e.g., with a watch glass) at the required temperature for varying periods of time and are then drained, blotted free of surplus water and weighed. The techniques used in the Experimental Factory were varied slightly according to the commodity being examined.

Meat and fish

Samples of 10 or 20 g of the AFD product were covered with cold water in a flat dish. It was necessary to hold them under water as the displacement of gas from the tissues was slow if they were allowed to float on the surface. At 5-min intervals the material was taken out and surplus water removed by blotting carefully with coarse filter paper. Then it was quickly weighed and returned to the water. The procedure was repeated at intervals until constant weight was reached.

Rather greater accuracy was obtained if instead of using the same sample repeatedly, several samples were put to soak at the same time, and the first blotted and weighed after 5 min, the second after 10, the third after 15 and so on. This removed any effect due to disturbance and possible squeezing of the food during blotting which might perhaps affect its rate of rehydration during subsequent immersion.

By such methods a rehydration curve could be obtained in which the weight of the material was plotted against time.

It was sometimes of value to examine the rate of rehydration first in cold water and subsequently during a period of cooking, and this could of course be carried out by suitable modification of the technique described above.

The rehydration of minced meat was measured in a fine mesh wire basket which was immersed in water for appropriate periods. Surplus water was removed by shaking the basket and contents for a standard time.

Vegetables

Whereas AFD meat and fish products were normally reconstituted in cold water (because the addition of hot water to dry protein material generally caused coagulation and toughening), AFD vegetables were usually rehydrated by immersion in hot or boiling water. In the laboratory therefore 10 or 20 g samples were treated by pouring on boiling water and allowing to stand for 5 min. The liquor was then poured off through a strainer and the vegetables lightly blotted and weighed. The procedure was repeated at 5-min intervals and, as with fish and meat, a rehydration curve obtained. If the vegetable was one which could be reconstituted by direct cooking without any prior period of soaking, the water was kept simmering during the period of the test.

In all cases the figure quoted as the 'rehydration ratio' was calculated from the curve when constant weight had been achieved.

Fruit

In only a few cases could rehydration ratios be measured satisfactorily since many fruits lost their texture on rehydration. Where the physical structure of the fruit made it possible, the procedure was generally similar to that for vegetables except that blotting was usually impossible and draining alone had to suffice. With soft fruits all that could be done was to measure water uptake simply by adding hot water from a measuring cylinder to samples of known weight (10 or 20 g) until the material was judged to be fully reconstituted.

Composite foods

Many composite foods have a fluid as one of their components, e.g., rice pudding, beans or spaghetti in tomato sauce, meat and vegetable stew (with gravy), and the method described for soft fruit was the most suitable for measuring the rehydration of such items.

RECONSTITUTION IN THE EXPERIMENTAL KITCHEN

The products of the Factory were examined in the Experimental Kitchen with emphasis on practical application, i.e., the reconstitution and cooking properties combined using trial and error methods, samples being soaked in water (hot and cold) for varying periods, and then cooked for various lengths

of time. Sugar or salt were added before cooking or soaking, according to the commodity. The simplest method of soaking and cooking, consistent with the best ultimate quality, was accepted as the proper method of preparation, the quality of the finished product having been judged by the taste panels.

By such methods it was found that raw meat and fish were much superior when reconstituted with cold water, (hot water produced a tough fibrous texture and a 'twice-cooked' or processed flavour), and the material left soaking for the required period (which varied for different products between 30 seconds and 15 min) before heat was applied for cooking. The only difference between the addition of hot and cold water to vegetables was the time factor; starting with hot water decreased the reconstitution and cooking times, and in some cases eliminated the soaking time altogether. As vegetables were normally dehydrated uncooked they required cooking for periods of time depending on the nature of the fresh material.

Fruits were similar to vegetables. Dehydrated fruits could be compared with fresh fruit only as cooked, not as raw material since, for adequate reconstitution, they required a short period of cooking. The use of hot water, therefore, decreased the preparation time. With soft fruits, where no period of soaking was required, either hot or cold water could be used, followed by a few minutes' simmering.

Composite dishes generally contained both meat and vegetables. They were, therefore, added to cold water initially and then heated for the required length of time.

The following are general recommendations for reconstituting and cooking the main types of AFD foods, derived from experience in the Experimental Kitchen. (Rehydrated meat can be satisfactorily cooked in a pressure-cooker.)

Raw meats

(Raw beef steaks and cubes. Raw mutton steaks, chops, cubes. Raw pork steaks, chops, cubes).

Completely immerse the dehydrated meat in cold water and allow to stand till the lean tissue becomes soft and plump. Normally this will be from 5–15 min but due to the incidence of fat, gristle, etc., some pieces may require longer. The fat will not, of course, soften in cold water but will do so in cooking. The beef steaks may now be grilled or fried and the others braised, stewed or casseroled as with fresh meat. It is recommended that the gravy thickening be added towards the end of the cooking as the meat softens more readily in unthickened gravy.

Minced cooked meats (all types)

Cover the mince with cold water, season well, bring to boil and simmer gently for 5 min. The mince is now ready for serving.

If desired, minced cooked beef and mutton may be prepared as a stew in the following manner:

Cover the mince with cold water, allow to stand until soft (10–15 min approximately). Drain. Brown some onion in smoking fat, draw to side of pan, add reconstituted mince and fry lightly. Add thickening and cover with cold water. Season well. Simmer for 10–15 min.

Cooked meat slices

Completely immerse the cold meat slices in cold water. Allow to stand until the lean is quite soft. This will normally take from 5–15 min but owing

to the incidence of fat and gristle may take longer. Remove from water and drain.

The meat slices may now be used with salads, or as a sandwich filling.

Cooked chicken (white)

Completely immerse in cold water and allow to stand until quite soft (approximately 10–15 min).

Cod steaks

Completely immerse the dehydrated cod steaks in cold water and allow to stand till soft and plump. This will normally be in about 2–5 min but may take up to 15 min.

The fish may now be poached and served with a suitable sauce, or drained, coated with batter and fried.

Flaked cooked fish

Place the fish in a suitable container and break any clumped pieces. For every 2 oz of fish stir in ¼ pt. of cold water and mix thoroughly. The fish reconstitutes immediately and may be used in a variety of made-up dishes.

Prawns (scampi)

Immerse the prawns in cold water for 5 min and drain. They are now ready for serving as such or may be dipped in batter and fried in deep fat.

Vegetables

Most vegetables may be reconstituted and cooked according to the following general method. Place the vegetable in boiling salted water at the rate of 1 oz vegetable to ¾–1 pt. water and allow to stand without further heating for 3–5 min. Bring to boil and simmer gently for 3–10 min. Drain and serve.

Cabbage

Place the cabbage in boiling salted water at the rate of 1 oz cabbage to 1 pt. water, simmer for 5–10 min.

Potato chips

Place the chips in boiling water at the rate of 1 oz chips to ¾ pt. of water and allow to stand without further heating for 5–10 min (until they appear to cease taking up water). Drain off the water and dry thoroughly. Fry in deep fat till golden brown, preferably using a fat rather hotter than usual (a fat with a high smoking point is advised).

Beetroot

Place the beetroot in boiling salted water at the rate of 1 oz beetroot to ½ pt. water, and simmer for 2–5 min. Leave to cool, add vinegar to taste and serve.

Cold peas (precooked AFD peas)

Place the peas in salted *cold* water at rate of 1 oz peas to ½ pt. water and allow to stand for approximately 30 min. Drain and serve in normal way.

Fruit

The soft fruits or skinned fruits require very little or no cooking, the time being extended to 8–10 min with tough skinned fruits. In general the method is to place the fruit in boiling water at the rate of 2 oz fruit to ½ pt. of water,

stand 1–5 min, bring to boil and simmer gently for 1–10 min, sweeten to taste and use as a stewed fruit or as an ingredient for a sweet.

One exception is:

Fruit Salad
Cover with hot water and leave to cool. Serve.

Meat bars (including curried beef and rice, meat and vegetable block, veal and ham, and steak and kidney)
Crumble block into cold water, bring to boil and simmer for 5–10 min: the proportions of water used depend on the nature and fat content of the block, which should be appropriately labelled.

The beef-and-pork meat bar is designed to be eaten dry as a munch when conditions do not allow for reconstitution.

Meat and vegetable stew, spaghetti and pork in tomato sauce, beans in tomato sauce, chicken suprême
Crumble 8 oz into 1¼ pt. of cold water and allow to stand for 3–5 min, bring to boil and simmer gently for 3–5 min. More water may be added if necessary.

Meat paste (5 oz block)
Crumble into a container, ensuring that there are no lumps. Add ¼ pt. cold water and mix to a smooth paste. Stand 5–10 min. Use as sandwich filling. More water may be added if desired.

Sausage meat (10 oz pack)
Crumble into a container. Add ½ pt. cold water and stir well until all water is absorbed. More water may be added if necessary. Shape into cakes and fry.

Fish cake mix (compressed and uncompressed)
Crumble and mix in sufficient almost boiling water to form a stiff paste (approximately ¼ pt. water to 2 oz mix). Form into cakes, cover with egg and breadcrumbs and fry in shallow fat on both sides till golden brown.

Cheese omelette (8·5 oz)
Crumble the block into a container. Gradually add ¾ pt. cold water, stirring vigorously all the time. When no more lumps remain, cook in normal manner.

Ham omelette (8·5 oz)
Instructions are similar to those for the cheese omelette, except that 1¼ pt. of water are used; allow to stand for 5 min before cooking.

Cooked rice
Place rice in boiling salted water at rate of 1 oz rice to ½ pt. water, simmer for 3 min. Drain and serve.

Rice pudding, Sago pudding
Crumble 8 oz into 1¼ pt. of cold water and allow to stand for 5 min. Break down any lumps, then bring to boil and serve.

The above instructions for reconstituting and cooking AFD foods were devised mainly for expeditions, and under such conditions the minimum of handling and preparation was essential. The methods produced the simplest

of dishes with no frills or trimmings, but of course this would have been equally true of fresh material prepared under similar conditions.

Taste Panels

The eating quality of a product is the final criterion by which it is judged, and in the quality control department of a food factory or research organization this judgment is normally carried out by presenting the food, prepared by the simplest practical method, to a number of people to taste, and for them to assess its quality in terms of appearance, flavour and texture.

There are many ways of conducting such 'taste panels' and of choosing the members. For certain purposes a so-called 'analytical panel' is valuable and this is the type which was normally used at Aberdeen. A 'consumer panel', which should indicate the acceptability of a product to the public, obviously cannot be reliably conducted with only a small number of tasters; but an analysis of qualities can be undertaken by a few trained persons, who are able to assess small differences in colour, flavour and texture, resulting perhaps from differences in production techniques and storage conditions.

Choosing a panel

Because of the large range of foodstuffs investigated at Aberdeen a single panel for all commodities was out of the question. One person could not be expected to possess sensitivity to all the flavours under test. Instead, four panels were formed, each corresponding to one of the major divisions in food, i.e., Meat, Fish, Vegetables and Fruit. All members of staff available for tasting were tested to discover if they could detect small variations in flavour in all four sections. Material was specially prepared so that differences were judged to be detectable but not too obvious. (This could be verified by the results. If over 75 per cent were correct they were too obvious and if under $33\frac{1}{3}$ per cent were correct, the differences were not great enough for the purpose.) The samples were presented, under code, in triangular tests, i.e., two samples of one and one sample of the other. Panel members were asked to pick out the odd one and state their reason. Each test was duplicated and the results analysed by a statistician. Those people with no flavour sensitivity were discarded. Four provisional panels were now composed of those people whose palates were found to be most sensitive to small flavour changes in either meat, fish, vegetables or fruit. The people who gave correct results for more than one commodity were at this stage included in each panel. These provisional panels were further tested in their own section only, by triangular tests in which the flavour difference was barely detectable. The results were again statistically analysed and the 9–10 members giving the best results were nominated to that specific panel. A period of training now followed when the members were familiarized with the score sheets and introduced to the various known off-flavours which occurred in their particular commodities, and the terms used to describe them. By presenting this panel with identical groups of samples at intervals of time their reliability was further judged and the final selection of 7–8 members made. A panel of six was considered the smallest number of significance and the additional members were included to allow for absentees.

Score sheets: The commonly used difference-test and a numerical scoring method based on a graduation from 'very good' to 'very bad' did not give

sufficient information about the material under test. Therefore with the guidance of the statistician score sheets were devised appropriate to the commodity and the form of the experiment. (As the range of foods under investigation was large so also was the range of score sheets.) The general principle was one of devising a set of descriptions of the range of characteristics found, under the headings of 'flavour', 'texture', 'odour', etc., for each commodity, with sometimes the addition of a quantitative record of intensity or frequency of such qualities.

Table 16 is a specimen copy of the score sheet used for samples of carrot coming from the processing line, the numbers having no mathematical significance, but merely acting as codes for a verbal description. Table 17 is the score sheet for samples of carrot from storage tests, and indicates the forms of deterioration to be expected. Table 18 is a score sheet for dehydrated meat devised at the Low Temperature Research Station, Cambridge, and Table 15 one for fish. These latter two incorporate a quantitative numerical score. It was found very difficult to devise a suitable scoring system for the various fruits and some of the vegetables, and the crude system of scoring was used for these items.

4 — excellent
3 — good
2 — fair
1 — poor
0 — very poor

Presentation of Samples: The taste-panel room contained six individual and entirely separate cubicles entered from a common corridor. Each cubicle was furnished with a shelf, a cuspidor flushed by a hot water tap, a set of various coloured lights and a sliding panel enabling the kitchen staff to pass samples to the taster. The coloured lights were used extensively in order to eliminate any prejudice caused by the appearance of the food. Panels were held daily, at set times. All members were aware of the times at which they were liable for tasting but were reminded 15 min beforehand when they would actually be required. It was considered that the tasters gave more accurate results and were more consistent in the mornings although it was never statistically proved. On presenting themselves for a panel each member was allocated a cubicle, given a score sheet and marking sheet and all the samples under test at the one time. The sliding panel was then closed. The tasters now assessed the samples entirely free from surrounding influences. Some form of palate cleanser was always given. The three used in Aberdeen were—orange juice for fish and hot meat; hot tea when tasting cold meat minces, to ensure that all fat was removed from the palate; and soda water at vegetable and fruit panels. (As these commodities were sometimes treated with sulphite it was felt that the sulphite present in orange juice might be misleading.) On completion of the assessment of flavour and texture the coloured lights were changed to a mixture of incandescent white and fluorescent day-light for colour assessment. It was found that this mixed light gave a more natural appearance to the foodstuff than either on its own.

This type of taste panel was the one used most extensively at Aberdeen as being most suitable to the work there. However, the triangular test or the two-out-of-five test was occasionally used when a direct comparison of samples

was required. The disadvantage of such comparisons was that they only indicated the presence or absence of a difference but no more helpful information.

For some purposes informal tasting sessions were valuable. At these all members of the team concerned with the particular experiment, and also the kitchen staff, tasted and discussed the merits of the product round the table. If it was considered unsatisfactory further development by the experimental team was indicated; if promising, the sample then went forward for formal reconstitution tests and taste-panel assessment.

CHAPTER XIII

Nutritive value

Fresh Foods

The importance of a food in the diet depends not only on its content of the various nutrients but also on the quantity in which it is usually eaten.

The contributions made to the total nutrient content of the average diet by the fresh foods which have been used for accelerated freeze-drying are given in Table 19.

Accelerated Freeze-dried Foods

Very little is known about the nutritive value of accelerated freeze-dried foods. Some knowledge of the effect of the AFD process on the proteins and vitamins in various foods has been obtained by animal feeding tests, microbiological estimations and chemical analyses, but improvement of the techniques of the drying process, selection of the most suitable varieties for dehydration and determination of the storage life of the dehydrated products have taken precedence over detailed analyses of individual foods for all nutrients. The information which follows is therefore of a general nature only.

Meat and fish

The effect of dehydration on the nutritive value of meat and fish can be considered from two aspects:

Changes in the value of the protein

Changes in the content of vitamins of the B group.

The nutritive value of the protein is unaffected by the low temperature employed during drying.

Thiamine is heat labile and water soluble; up to 30 per cent of the initial value may be lost during processing (but it is also lost to about this extent in ordinary cooking, and thiamine, moreover, is widely distributed in the diet).

It has been found that while there is a loss of about 30 per cent of the riboflavin from mutton during processing, this loss does not occur when beef, pork or cod are dehydrated. The suggestion that this might have been due to faulty extraction is considered unlikely because the loss has been consistent in the samples tested. The retention of nicotinic acid, pantothenic acid and vitamin B_{12} in meat and fish varies between about 70 and 100 per cent.

It is to be expected from such evidence that when the reconstituted AFD products are cooked, the combined processing and cooking losses will be of the same order as for corresponding fresh foods. The nutritive value of foods which are cooked *before* dehydration and eaten directly after reconstitution is also likely to be of the same order.

Vegetables

Vegetables are valuable as a source of vitamin C, together providing over half the daily intake. Because of the large amounts eaten, potatoes alone provide about one-third of the daily total. Root vegetables are important as a source of vitamin A in the diet, and potatoes as a source of thiamine; vegetables also provide nicotinic acid and iron (see Table 19).

TABLE 19

Contribution to total nutrient content of diet[1] *(per cent)*

	Protein	Calcium	Iron	Vitamin A	Thiamine	Riboflavin	Nicotinic Acid	Vitamin C
Carcass meat	14	1	14	1	8	8	20	0
White fish, fresh and processed	2	—	1	0	—[2]	1	1	0
Potatoes	5	2	9	0	14	8	14	33
Green vegetables	2	2	3	4	4	2	1	14
Root and other vegetables	3	2	5	15	3	3	2	5
Fruit	1	2	4	6	4	2	3	36

[1] from National Food Survey, 1959.(46)
[2] less than 0·5 per cent.

Nutrients may be lost from vegetables in cleansing and preparation, during scalding and processing, and during cooking. These losses are caused both by oxidation and by leaching of the soluble nutrients from the tissues. As the method of pre-treatment for accelerated freeze-drying is in general the same as for conventional air-drying, the same principles may be applied.

The estimated losses of nutrients from vegetables during scalding as reported by Allen and Mapson(47) are given in Table 20.

TABLE 20

Nutrient	Percentage of fresh value lost during scalding	
	by leaching	by oxidation
Protein	7	0
Sugars	20	0
Starch	0	0
Ascorbic acid: (sulphite used in processing)	20	9
Thiamine (sulphite used in processing)	(a)	(a)
β-carotene	0	0
Iron	7	0
Calcium	5(b)	0

(a) When sulphite is present most of the thiamine is usually destroyed by fission of the molecule.

(b) With spinach 0

With the very low moisture contents obtained by freeze-drying, the need for sulphur dioxide for the retention of vitamin C and prevention of non-enzymic browning during storage was greatly diminished; and sulphite was therefore only used in reduced concentrations when processing cabbage and Brussels sprouts to intensify their green colour. When sulphite was added any thiamine present was destroyed, but in the absence of sulphite it is likely that the loss during processing would be low (as for unsulphited peas).

Chemical analyses have shown that the loss of total carotenoids from steam scalded carrot dice ($\frac{5}{16}$ in. × $\frac{5}{16}$ in. × $\frac{3}{8}$ in.) during dehydration was negligible and there was no loss during cooking. The loss of thiamine from steam-scalded peas during processing was small; the loss of ascorbic acid was found to vary between about 8 and 20 per cent (see Table 21). When cooked for eating the amounts of these two nutrients were only slightly less than in cooked fresh peas of the same varieties.

TABLE 21

Ascorbic acid content of fresh and dehydrated peas, 1959 season

Variety	Size Grade	Ascorbic acid content (mg/100 g dry wt.)	
		Fresh	Dehydrated
Kelvedon Wonder	Small	116	107
	Medium	71	75
	Large	50	45
Witham Wonder	Small	136	115
	Medium	116	90
	Large	84	77

Accelerated freeze-drying has been used for the preparation of potato chips and potato dice, but the vitamin C content of these products has not been determined. It has, however, been found that steam-scalded air-dried potato strips provide, when cooked, one-third of the ascorbic acid found in a similar portion of fresh mashed potato. In these experiments an average of 61–65 per cent of the initial ascorbic acid in the thin strip potatoes ($\frac{1}{8}$ in. × $\frac{5}{16}$ in.) was leached into the cooking liquor, compared with an average loss of 55 per cent in thick strips ($\frac{3}{16}$ in. × $\frac{5}{16}$ in.) processed in the same way. Thus it is to be expected that the loss of ascorbic acid from reconstituted AFD potato chips ($\frac{1}{2}$ in. × $\frac{1}{2}$ in.) by leaching during cooking will more nearly approach the loss from unprocessed potatoes during cooking (i.e. 12–19 per cent). It is possible that the stability of ascorbic acid in AFD potatoes may be affected by the fact that sulphite is not used during processing.

Fruits

The main nutritive importance of fruit in the diet is as a source of vitamin C and, as with vegetables, different pre-treatments before dehydration affect the vitamin C content of the product.

Steam scalding causes heavier losses of ascorbic acid than other pre-treatments, apparently because of the loss of juice which results. Black-currants retain 90–100 per cent of their initial ascorbic acid when blast frozen or sulphited, 70–100 per cent when raw dried and 60–80 per cent when steam scalded. The loss of ascorbic acid in raspberries is somewhat greater than in black currants.

Effect of Storage on Nutritive Value

Meat and Fish

Animal feeding tests on dehydrated cooked minced beef, raw minced beef and raw minced fish have shown that there is little or no loss of net protein utilization value (i.e., $\frac{\text{retained nitrogen}}{\text{food nitrogen}} \times 100$), even under the severe conditions of accelerated storage tests. Indeed, the value remains high[48] even beyond the stage of inedibility due to other deteriorative changes.

Cod, silverside, pork and mutton packed in nitrogen have been stored at −20°C, 18°C and 37°C, and estimations of the B vitamins made at varying intervals up to 2 years. Microbiological assays have shown that in cod there was a loss of about 7 per cent of the riboflavin and 30 per cent of the nicotinic acid after 18 months storage at the comparatively high temperature of 37°C, pantothenic acid and vitamin B_{12} being stable. All these vitamins were stable over a similar period of storage at 18°C.

Storage did not appear to have any great effect on the vitamins of the B complex in the limited number of samples of silverside and pork examined. The retention of riboflavin varied between about 50 per cent in pork and 70 per cent in silverside and mutton at both storage temperatures. In general, nicotinic acid, pantothenic acid and vitamin B_{12} were stable during storage, although there was some loss of nicotinic acid in silverside and mutton and of pantothenic acid in silverside. Samples of beef, pork and cod stored at the same temperatures and examined after a year showed that there was very little change in the thiamine content during storage. (Previously chemical estimations of thiamine in meat bars made of air-dried material had shown that thiamine was destroyed after 4–6 months storage at 37°C. This was not found to be the case with freeze-dried raw meat, possibly due to the lower temperatures employed during drying.) Pyridoxine also remained stable, only a very slight loss occurring in meat and no loss in cod after 12 months.

Vegetables

Storage tests on accelerated freeze-dried peas showed that the ascorbic acid content of peas packed in nitrogen declined gradually at a temperature of 18°–20°C; when packed in air in cans or pouches and stored at the same temperature the peas lost ascorbic acid rapidly. The storage life of AFD peas was very much longer at 37°C than that of air-dried peas of the same variety and size grade (Table 22), presumably because of the higher moisture content of the latter. At 18°C also, the air-dried material deteriorated rather more rapidly than the AFD product. It should be noted that the scalding time of the air-dried peas was 1½ min, compared with the 5 min cooking given to AFD peas before drying. It is conceivable therefore that enzymic reactions could have occurred in the air-dried peas.

Peas packed in air pouches consisting of 0·0015 in. rubber hydrochloride and 0·001 in. cellulose acetate with adhesive lamination and stored at 18°C and 60 per cent R.H. increased in moisture content and lost ascorbic acid more rapidly than similar material packed in nitrogen in cans (Table 23).

TABLE 22

Ascorbic acid content (mg/100 g) of air dried (AD) and accelerated freeze dried (AFD) peas held at 37°C and 18°C. (Variety Canners 99, 1957 season)

Moisture contents AD = 6·1 per cent; AFD = 2·7 per cent;
scalding times AD 1½ min; AFD 5 min

Storage temperature	Mode of drying	Period of storage (months)						
		0	2	4	6	8	10	12
37°C	AD	64	20	28	17	—	—	—
	AFD	64	56	50	60	58	58	—
18°C	AD	64	—	58	46	36	—	—
	AFD	70	—	50	47	49	—	31

TABLE 23

Storage behaviour of AFD peas at 18°C

	Mode of Packing	Period of storage (months)					
		0	1	2	3	6	12
Ascorbic acid (mg/100 g)	Can (N_2)	66	36	50	41	37	29
	Pouch (air)		57	46	29	19	17·9
Moisture (per cent)	Can (N_2)	2·29	2·06	2·81	1·93	1·55	2·39
	Pouch (air)		3·59	3·91	4·13	4·48	7·74

Fruit

The ascorbic acid content of dehydrated fruit decreased when stored in air. Fruit bars (apple, lemon and sugar) stored in air at 18° and 37°C lost ascorbic acid rather more rapidly than those stored in nitrogen; at 18°C the loss in 12 months varied between about 35 and 70 per cent for fruit bars stored in air (Table 24); there was a smaller percentage loss in the fruit and sugar mixture with added ascorbic acid, whether stored in air or nitrogen.

TABLE 24

Retention of ascorbic acid (expressed as mg/100 g) in fruit bars during storage in air and nitrogen

Materials 1. Apple, lemon and sugar: m/c 3·9 per cent, SO_2 78 p.p.m.
2. Apple, lemon and sugar: m/c 3·9 per cent, SO_2 80 p.p.m. with added ascorbic acid

Type of material	Stored in air or nitrogen	Storage temperature °C	Period of storage (months)			
			0	3	6	12
1	Air	−5	42	40	34	34
1	N_2	−5	42	40	30	34
2	Air	−5	400	310	320	292
2	N_2	−5	400	311	—	290
1	Air	18	42	38	23	11
1	N_2	18	42	36	27	18
2	Air	18	400	296	252	253
2	N_2	18	400	288	337	267
1	Air	37	42	7	5	—
1	N_2	37	42	14	9	—
2	Air	37	400	41	19	—
2	N_2	37	400	201	51	—

CONCLUSION

In conclusion it may be said that although detailed analyses are not yet available for accelerated freeze-dried foods, there is reason to believe that because drying at such low temperatures is advantageous for heat sensitive materials the combined loss of nutrients during processing, reconstitution and cooking are of the same order as for the corresponding fresh foods after cooking.

(When accelerated freeze-dried foods are reconstituted they usually absorb less water than was lost during dehydration, so when comparing nutritive values allowance has to be made for differences between the moisture contents of the cooked unprocessed and the cooked dehydrated foods, the latter sometimes being less.)

CHAPTER XIV

Quality control

THE object of a Quality Control Department is normally to ensure that the foodstuff leaving the factory is within the specifications which have been adopted for that product. In the case of accelerated freeze-drying, however, there have been no definite specifications, other than the legal limitations on the sulphur dioxide content of dehydrated foods. There is thus scope for manufacturers to decide for themselves to what standards these new products should conform, bearing in mind the use to which their products will be put, the shelf life required and the probable conditions of storage.

The laboratory is not, of course, solely confined to carrying out analyses of the finished product. If the laboratory is to function effectively as a quality control department it will have to carry a share, at any rate, of the responsibility for ensuring that the correct raw material is used, and that its quality is good. It will also be concerned with the conditions of processing in the factory, checking on such steps as scalding and sulphiting. Routine bacteriological control is also a vital part of the work of the quality control laboratory.

The minimum essential laboratory determinations differ with the products. In all cases moisture content must be ascertained. If vegetables or fruit have been sulphited it is necessary to check the final level of sulphur dioxide content. The adequacy of preparation of root vegetables must be checked by a routine blemish count; and in certain vegetables and meat products, where disintegration leads to the production of powdery material with poor texture, the proportion of fines should be determined. Bacterial counts should be routine for fish and meat products, and should be carried out at intervals on fruit and vegetable products to ensure that proper factory hygiene is being maintained. When the finished products are packed in nitrogen regular checks of the efficiency of gas packing, as indicated by the proportion of oxygen in the containers, are essential; and if vacuum packing is used care should be taken to see that the vacuum is, in fact, adequate, although the rough test of trying to pull the packaging material away from the contents is usually sufficient indication. A special case arises with certain meat products, where the proportion of fat must be known, e.g., in the meat bar, where 40 per cent fat is commonly specified. In such instances the fat content has to be determined in the laboratory.

If a check on the efficiency of the factory operation in terms of wastage is required it will be necessary for the moisture (or dry matter) content of the raw material to be determined as well as that of the product, and for wastage such as trimmings to be measured, so that a balance sheet indicating the fate of the dry matter entering the factory may be drawn up.

In the case of vegetables scalded in water regular determinations of the soluble solids content of the scald liquor are necessary, and of its sulphur dioxide content if sulphite is used. This does not arise when steam scalding

is employed; only occasional checks of the sulphite solution in the dosing tanks are required. Regular tests for peroxidase activity of the vegetable emerging from the scalder are essential; a positive reaction to this test may be the first indication of the development of a fault in the scalding system.

The various tests mentioned above ensure that the product has been prepared in such a way that it will have certain desired characteristics and that it will stand up to the appropriate periods of storage, but the most important practical test is, of course, that of culinary quality. This is again the responsibility of the quality control department, and in order to ensure comparable results it is necessary to state precise conditions of preparation and cooking, and to use standardized systems of evaluation. This whole matter of culinary tests is discussed in Chapter XII. Since the reconstitution ratio (a measure of the amount of water taken up when the foodstuff rehydrates) is so closely allied to culinary quality, this also is dealt with in the same chapter.

It may happen, especially in laboratories concerned with development as well as routine control, that more detailed examination of the foods, in their raw or in their dehydrated state, may be required. The number of possible analyses required is legion, and in the present Report only a few of those most likely to be required are described.

The quality and storage behaviour of fish and meat may be affected by the degree of unsaturation or of oxidation of the fats; these quantities are indicated by iodine values and peroxide values respectively. Browning may develop during dehydration or storage, and a method of expressing this quantitatively is often valuable. The sugar content of certain foodstuffs, notably potatoes, profoundly affects the liability of the dehydrated product to browning, and descriptions have therefore been given for methods of determining sugars in potatoes.

Assessment may be required of the effect of processing on the nutritive quality of foods. Accordingly methods are given for the determination of β-carotene (provitamin A), and ascorbic acid, for the determination of nitrogen (protein) and the biological value of protein.

Before describing the various analytical techniques, however, it is desirable to emphasize one aspect of good factory management. Procedures should be so ordered that it is possible to trace any particular sample of the finished product back through the process to the raw material from which it was manufactured. Only if this can be done will the analytical results have real value in pin-pointing faults in procedure. Since the accelerated freeze-drying process was carried out as a batch process in the Experimental Factory it was not difficult to arrange things in this way.

The methods described in the following pages are those which were employed at the Aberdeen Experimental Factory. In most cases they were not basically original but were derived from techniques which had been used for other purposes, modified to suit dehydrated foods. Most are well known and these are described without explanation of the reactions involved; in a few cases, however, the underlying principles are explained.

Sampling

Every factory chemist is aware of the problems of sampling from large quantities of product or of raw material, and though the extreme importance

of using a representative sample must be emphasized, it is impossible to lay down precise rules. Both the raw material and the product, even of a single consignment or production batch, are liable to wide variation; in the case of a consignment of potatoes in sacks, for example, a handful of potatoes from one sack just will not do; one potato from every fifth or tenth or twentieth sack would give a much more representative sample. One or two cabbages cannot represent a consignment; but if a sample of the edible portion is required, a few shreds taken from the shredding machine at intervals during the run would provide a much better indication of the consignment as a whole. One tin of dehydrated product will not necessarily be representative of its production batch; samples of the finished product are best taken from the trays, a small quantity from every second tray, for example. The best guide to sampling is a commonsense appreciation of the facts of biological variation.

Summary of applicable tests

ROUTINE EXAMINATION OF VEGETABLES AND FRUIT

Moisture content
Sulphur dioxide content
Peroxidase activity (for scalded materials)
Soluble solids in scald liquor
Sulphite in scald liquor
pH of scald liquor
Proportion of fines
Oxygen content of containers
Bacterial count
Reconstitution ratio
Culinary quality

ROUTINE EXAMINATION OF FISH AND MEAT PRODUCTS

Moisture content
Bacterial counts
Fat content
Proportion of fines
Oxygen content of containers
Reconstitution ratios
Culinary quality

ADDITIONAL ANALYSES WHICH MAY OCCASIONALLY BE REQUIRED

Factors affecting quality and storage behaviour:

Iodine value of fats
Peroxide value of fats
Browning
Sugar content of potato

Factors affecting nutritive value:

Ascorbic acid content
Vitamins of the B complex
β-carotene
Nitrogen (protein)
Biological value of protein

METHODS OF ANALYSIS

The following methods, used at the Experimental Factory, are offered in such detail as seems necessary for the guidance of future operators.

1. Moisture content
   - Dehydrated vegetables and fruit
     - (a) Vacuum-oven method
     - (b) Dean and Stark method
   - Raw vegetables and fruit
   - Dehydrated fish and meat
     - (a) Vacuum-oven method
     - (b) Dean and Stark method
   - Fresh fish and meat
2. Sulphur dioxide content of vegetables and fruit
   - (a) The Monier-Williams method
   - (b) Direct titration method for cabbage
3. Peroxidase activity
4. Sulphite in scald liquor
5. Soluble solids in scald liquor
6. pH of scald liquor
7. Blemish count in potato
8. Proportion of fines
9. Oxygen content of containers
10. Fat content of meat or fish products
    - (a) Rapid Soxhlet
    - (b) Determination using Gerber tubes
11. Iodine value of fats
12. Peroxide value of fats
13. Browning in dehydrated vegetables
    - (a) Browning in potato and green vegetables
    - (b) Browning in carrot
14. Sugar content of potato
    - (a) Hexose, sucrose and fructose
    - (b) Reducing sugars by the picric acid method
15. Ascorbic acid
    - (a) General method
    - (b) In products with reductones
    - (c) In strongly coloured materials
16. β-carotene
17. Nitrogen
18. Biological value of protein
19. Microbiological quality control

Moisture Content

Dehydrated vegetables and fruit

Vacuum oven method. The sample was rapidly ground in a hammer mill to such an extent that 95 per cent would pass a 40-mesh sieve, and approximately 3 g samples were accurately weighed into tared aluminium containers.

(The containers were approximately 3 in. diameter and ½ in. deep and were dried in an air oven for 1 hr and cooled in a desiccator before tare weighing.)

The container with the ground sample was placed, with its lid off, on the heating plate of a shelf-type vacuum oven thermostatically controlled at 70°C. The vacuum was *slowly* applied and maintained at not more than 1 mm Hg for 5 hr. At the end of this period the vacuum was released *slowly*, the lids placed on the containers, and these cooled in a desiccator and weighed. The loss in weight represented the moisture loss and the moisture content could then be directly calculated.

Dean and Stark method. For samples of relatively high moisture content the Dean and Stark distillation method, which had the advantage of being comparatively rapid, was employed. Cleanliness of the apparatus was vital, for if the condenser and the receiver were not absolutely clean droplets of water stuck to the sides and could not be measured. Chromic acid was the most effective cleaning agent. After cleaning, the apparatus was rinsed very thoroughly in tap water to remove all traces of acid. Tap water was better than distilled water which may have been stored in an aspirator bottle with a greased tap. Most of the water was drained off but the apparatus did not need to be dried.

The still wet apparatus was assembled and about 130 ml of toluene or n-heptane were put into the 250 ml. round-bottomed flask with some pieces of porous pot. If the apparatus was dry a few drops of water were added. The solvent was refluxed until the water level in the calibrated stem was constant. This was reading 'X'. Then the accurately weighed sample was put into the flask and refluxed until the water level was again constant. This was reading 'Y'.

The sample size was arranged to suit either the 2 ml or the 10 ml Dean and Stark receiver, and the moisture content was calculated from

$$\frac{(Y-X)}{\text{wt. of sample}} \times 100 = \text{per cent moisture}$$

Raw vegetables and fruit

There are several standard methods of determining the moisture content of fresh foodstuffs and these will not be detailed here.

Since hot-air drying facilities were available, however, a satisfactory method of estimating the moisture content of raw materials was to take a fairly large sample, as representative as possible of the consignment, shred or mince it (if this has not been done before sampling) and dry 5 to 10 lb on trays covered with butter muslin to avoid mechanical losses, by the usual hot-air drying techniques. Under these conditions there were no measurable losses of substances other than water and no breakdown of carbohydrates. After dehydration the foodstuff was accurately weighed, and the moisture content of sub-samples determined in the way described above; from which result the moisture content of the original could be calculated.

Dehydrated fish and meat

Vacuum oven method. The sample was ground rapidly by passing twice through a mincer using a plate with ¼ in. orifices. The resulting powder was placed in a dry jar, shaken to ensure random distribution of the different sizes of particle (which have been shown to contain different proportions of

moisture), and then dried *in vacuo* at 70°C for 5 hr in the manner described. (Some workers prefer to use a slow air-leak into the vacuum oven, operating at 2–3 bubbles per second.)

Fresh fish and meat. The sample was minced and mixed to ensure homogeneity. The minced sample was pounded thoroughly in a mortar to obtain as uniform a particle size as possible. Deep aluminium containers without lids were used for the determination. The containers, each containing some clean sand and a small glass stirring rod, were dried in the air oven at 100°C, cooled in a desiccator, and weighed to the nearest milligram. About 10 g of the fresh sample were put into each container and the exact weight determined.

The sample was now throughly mixed with the sand in order to break down lumps and to avoid caking during drying. The containers were then placed on a boiling water bath and the contents mixed at intervals for 1 hr. The mixing again prevented caking or the formation of a hard skin on top of the sample, which would have stopped the water escaping.

After this preliminary heating in the water bath the containers were placed in an air oven at 100°C for 3 hr, cooled in a desiccator and weighed. They were then put back in the oven for further periods of 1 hr until constant weight was reached. The percentage moisture content was then calculated.

Sulphur Dioxide Content of Vegetables and Fruit

The Monier-Williams method. An 18 in. Liebig condenser was connected at its lower end with a 3-necked 1-l. flask. The second neck of the flask carried an inlet tube for nitrogen passing almost to the bottom of the flask. The third (wide) neck, closed by a ground glass stopper, permitted rapid addition of the sample without disconnecting the apparatus. The top of the condenser connected with an absorption system consisting first of a 250 ml CO_2 flask and then a 10 ml Peligot tube.

The reagents used were DILUTE HYDROCHLORIC ACID: 66 ml of A.R. concentrated hydrochloric acid sp. gr. 1·18 were diluted to 1 l. with distilled water. This was prepared some time before use, because it was found that on mixing a mist formed above the solution which cleared only after prolonged standing. If the acid were used too soon, the mist would be swept over in the stream of gas and estimated as SO_2.

HYDROGEN PEROXIDE: To 100 ml of 100 volume H_2O_2 900 ml of distilled water were added and sufficient bromo-phenol blue indicator (approximately 6–8 ml). The peroxide was neutralized by titration with sodium hydroxide solution.

N/20 SODIUM HYDROXIDE SOLUTION: Standardize against a solution of dried A.R. potassium hydrogen phthalate.

The procedure was as follows:

Two hundred and fifty millilitres of dilute hydrochloric acid were added to the three-necked flask, 50 ml of hydrogen peroxide to the CO_2 flask, and 3–5 ml of hydrogen peroxide to the Peligot tube and the apparatus assembled. The sample (previously weighed and wrapped in filter paper) was added through the wide centre neck of the three-necked flask. A stream of nitrogen (2 or 3 bubbles per second) was started through the apparatus and the acid brought to the boil. Boiling continued for 60 min. After this period, the side

assembly was removed, the contents of the side tube poured into the CO_2 flask and the lead and side tube washed, adding the washings to the CO_2 flask. The sulphuric acid formed was titrated against N/20 sodium hydroxide.

For a 5 g sample, 1 ml N/20 NaOH was equivalent to 320 p.p.m. SO_2 in the sample.

Direct iodimetric titration for cabbage.[49] In each of two 600 ml beakers 8 g of ground dehydrated cabbage were suspended in 400 ml water, and 5·0 ml of 5 N sodium hydroxide added. The mixture was stirred gently, being careful not to beat air into the solution, and allowed to stand for 20 min. To one of the samples (A) 7·0 ml 5 N hydrochloric acid were added, stirring to avoid local concentration. Ten millilitres of the 1 per cent starch solution were now added, and 0·05 N iodine solution (diluted from a stronger stock in KI solution) run in at once, until there was a stable blue colour which was best seen in the supernatant liquor when the cabbage particles had settled to the bottom of the beaker. It was important that the acidified sample was titrated at once before the sulphite liberated by the acid had been able to recombine.

The above measured the total iodine-reducing power of the cabbage. To determine the reducing material other than sulphite, the second sample (B) was acidified in the same manner, and 2 ml of 3 per cent hydrogen peroxide immediately added, to oxidize the sulphite to sulphate. The sample was now titrated with iodine as before.

When a sample of 8 g was used, then:

(ml of iodine to titrate sample A)—(ml of iodine to titrate sample B) × normality of iodine × 4000 = sulphur dioxide content in parts per million.

Peroxidase activity

The reagent was made by mixing equal volumes of a 1 per cent solution of guaiacol in distilled water (which would keep indefinitely) and hydrogen peroxide (strength 5 vols.) (which would last for some weeks in a refrigerator). The mixture was not stable for more than 2 to 3 weeks and was discarded when it developed a slight brown colour. The activity of the mixture was checked daily by making tests on raw vegetables, when an intense chocolate-red colour would appear.

For the test on dehydrated vegetables a 10 oz wide mouthed bottle was about one-quarter filled with the product and about 20 ml of the reagent added. The bottle was closed and well shaken for a few seconds before tipping the contents out on to a white tile or porcelain dish for examination. Development of a red-brown colour indicated the presence of active peroxidase; if no red colour appeared within 1 min then no significant amounts of peroxidase were present.

Sulphite in Scald Liquor

Samples of liquor were passed through a muslin or stainless steel mesh strainer and cooled (this could conveniently be done by passing through a coil condenser with cold water circulating in the jacket). Twenty millilitres of the cooled liquid were titrated with N/32 iodine solution, using 1 per cent starch as indicator. (No addition of starch was necessary when potato was being processed.) One millilitre of N/32 iodine was equivalent to 50 p.p.m. SO_2 in the scald liquor.

Soluble Solids Content of Scald Liquor

Small quantities of acid-washed sand were put into evaporating dishes, dried in an air-oven at 101°C, cooled and weighed. Duplicate 10 ml portions of the scald liquor were placed in two of these dishes, evaporated to dryness on a water bath, and then dried in the air oven at 101°C for 1½ hr. The results were then expressed as percentage w/v of the scald liquor.

Sugars formed the greatest part of the solutes leached into the scald liquor, and the sucrose content of the liquor, as read by a refractometer, gave a satisfactorily close approximation to the actual content of total soluble solids. The relatively inexpensive Bellingham and Stanley 0–28 per cent Pocket Refractometer was found as suitable for this purpose as more elaborate and expensive instruments.

pH of Scald Liquor

Samples of the scald liquor were taken at suitable intervals and tested with pH papers, ranges 5·2–6·7 for potato and 6·8–8·3 for cabbage and carrot. The pH as indicated by test papers was quite sufficient for routine control purposes.

Blemish Count in Potato

Duplicate representative 50 g samples of the dehydrated product were reconstituted and the number of blemishes noted. By blemish was meant any conspicuous piece of skin, eye, worm-hole, diseased or discoloured area. (It may be noted that the maximum blemish count permitted in the current Ministry of Agriculture, Fisheries and Food specifications for air-dried dehydrated potatoes to be supplied to the Services was 8.)

Proportion of Fines

In the case of vegetables there was a Ministry of Agriculture, Fisheries and Food specification (for air-dried vegetables)[50] which read as follows:

the proportion of fines which passes a British Standard No. 8 sieve to be not more than:

	per cent
Dehydrated Potato Strip	1
Dehydrated Cabbage	15
Dehydrated Carrot	5
Dehydrated Parsnip	5

Similarly there was a specification of not more than 20 per cent fines fo air-dried minced cooked meats, estimated as percentage of the dry, fat-free meat passing a 16 mesh BSS sieve (i.e., with 16 meshes per linear inch and average aperture 1·37 mm).

The method of determining fines in meat was as follows:

Twenty-five grams of the minced meat were covered with 60°–80° petroleum ether in a beaker, and stirred gently with a glass rod for 1 to 2 min, taking care not to break the granules. The material was transferred, with the aid of petroleum ether from a wash bottle, to the standard sieve placed in a glass crystallizing dish. The sieve was transferred to another dish containing fresh solvent and agitated in the liquid until the meat was washed free from fat and no more material passed through the mesh.

The *granules* remaining on the sieve were dried at 80°–100°C and weighed.

The *solvent* was decanted from the two dishes (but not from the original beaker), the residue transferred to a filter paper, washed with petroleum ether, dried at 60–80°C, and weighed. The weight of fines was then expressed as a percentage of the total, fat-free solids.

Oxygen Content of Containers

Almost any of the standard gas analysis techniques could be used but the apparatus and method described below were relatively simple and are recommended for a number of estimations in rapid succession without cleaning or replenishment of reagent.

The apparatus operated on the constant volume principle, Fig. 14 showing the main glass parts, which in practice were mounted on a frame of laboratory scaffolding standing in a tray to retain spilt mercury in case of accident.

A connection *A* for the sampling line led through the two-way stopcock *B* to the measuring bulbs *C*. There were two bulbs in series, with capacities of about 5 ml and 20 ml so that the size of sample taken could be related to the size of can being examined, the larger sample being taken whenever possible. Index marks were etched on the connecting tube below each bulb. The measuring bulbs were surrounded by a water jacket fitted with hand stirrer and thermometer.

The measuring bulbs were connected by stout rubber tubing carrying a screw clip for fine adjustment of levels to the manometer tube *F*, which was backed by a mirror scale graduated in mm, and further through the stopcock *G* to the mercury reservoir *H*.

The other side of the stopcock *B* was connected to the absorption chamber *D*, which was packed with glass tubes of 4–5 mm bore, the lower end of each tube being bevelled at an angle of about 60°. There was an index mark above the absorption chamber. The absorption chamber communicated with a reagent reservoir *E*, which was connected to the mercury reservoir *K*. The lower end of the absorption chamber was closed by a rubber bung through which passed the glass tube *L*. This was connected by rubber tubing (closed by a screw clip) to the funnel *M*, which was suspended by a wire hook from a suitable point of the frame.

The mercury reservoir *H* was hung from one of three hooks made from brass wire, the lengths of which were such that when the reservoir was hung from the topmost one the mercury level at atmospheric pressure was at the bend above the measuring bulbs; when the reservoir was hung from the second or third hook the mercury level was approximately at the upper or lower index mark on the measuring bulbs. When these lengths had been established, it was only necessary to move all the hooks up or down together to allow for different quantities of mercury in the reservoir *H*; the relationship remained unaltered.

The mercury reservoir *K* was hung from a hook mounted on a threaded rod about 5 cm long. This gave all the fine adjustment necessary on this side once the level had been approximately adjusted on the frame.

The sampling line comprised a four-way metal connector with stopcock *N*, connected through a mercury trap to a vacuum pump, and on the other side by small bore rubber pressure tubing to a sampling tool. The connector also carried a small mercury manometer closed at the free end ('short form barometer'), suspended from it by a short length of pressure tubing. The

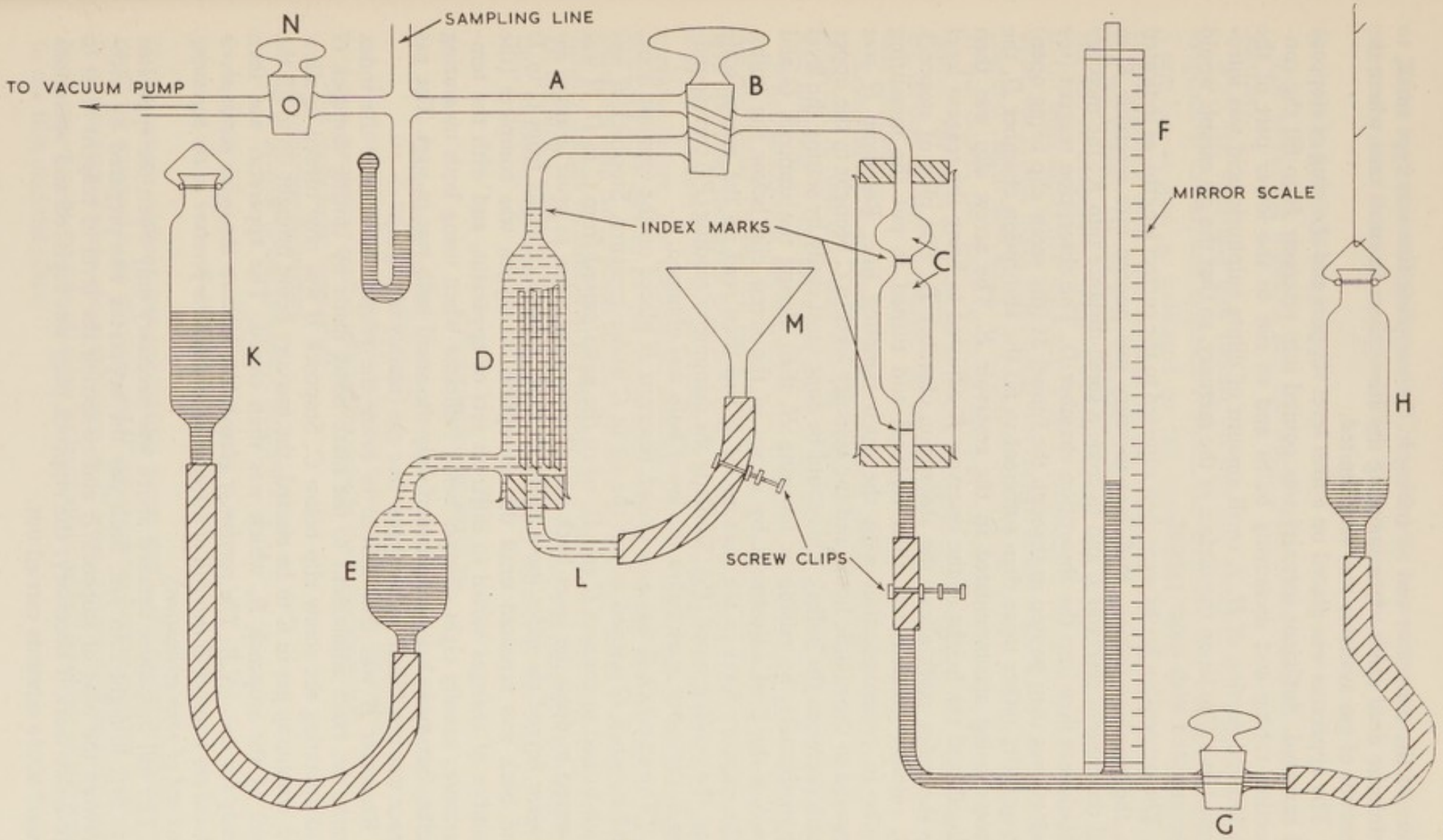


FIG. 14. *Diagrammatic sketch of gas analysis apparatus (not to scale)*

bore of the connector and all tubing in the sampling line was kept small, to minimize dead space when sampling the atmosphere in small cans where the amount of gas available was very limited.

The apparatus was placed on a firm level support and the plug of stopcock *B* removed. Sufficient mercury was poured into reservoir *H* to fill the connecting tubing and measuring bulbs and to rise to the lower part of the cylindrical portion of *H*. A small amount of dilute sulphuric acid was introduced into *C* to lie on the surface of the mercury, so that the gas sample would be saturated with water vapour.

The suspension hooks were then adjusted to the correct lengths, as indicated in the previous section. Sufficient mercury was poured into reservoir *K* to fill the reagent reservoir *E* and to come a small distance into *K*; the mercury should not flow into the absorption chamber *D*. The absorption reagent (see below) was then poured in through the funnel *M* (the screw clip being open) in quantity rather more than sufficient to fill the absorption chamber *D*, the excess being accommodated in the reservoir *E*. The screw clip was then closed, and the height of the reservoir *K* adjusted to bring the reagent level to the index mark above the absorption chamber *D*. The plug of stopcock *B*, suitably greased, was then replaced and turned to put the measuring bulbs in communication with the open sampling line. Reservoir *H* was lowered to the middle hook, and the mercury level in *C* brought to the upper mark (between the bulbs). This could be done most easily by setting the level approximately, by raising or lowering *H*, then closing the stopcock *G* and making the final adjustment by means of the screw clip below the bulbs. The mercury level in the manometer *F* was then read on the mirror scale and the reading noted (P_1). Similarly the manometer reading (P_2) corresponding to the index mark below the lower bulb was obtained.

*The stopcock *B* was now closed, reservoir *H* placed on the topmost hook, and stopcock *G* opened to put the air in *C* under pressure. Stopcock *B* was then turned to connect *C* and *D*, when the gases passed from *C* to *D*. *H* was lowered to draw the gases back into *C*, *care being taken to avoid drawing the reagent beyond the index mark above D*. This passage of gases from *C* to *D* and back was repeated until the absorption of oxygen was complete (the number of changes varied in different sets of apparatus, and with the temperature; usually eight changes being sufficient when using both measuring bulbs, but more being required if only the small bulb was in use). The gas space was now filled with oxygen-free gas (mainly nitrogen).

Reservoir *H* was now moved to bring the reagent level near the index mark, the exact adjustment to the mark being made by closing stopcock *G* and operating the screw clip below *C*. Stopcock *B* was now turned to allow the remaining gas in *C* to be ejected, the mercury being brought up to fill the bore of the stopcock *B*, which was then closed. The apparatus was then ready for use. (*N.B. The portion of glass tubing between the index mark above D and the stopcock B was full of nitrogen at atmospheric pressure at the beginning and end of each estimation*).

The full procedure detailed above was necessary only when the apparatus was first brought into use. Each day the apparatus was prepared by withdrawing the plug of stopcock *B* and re-setting the level of reagent in *D* to the index mark if necessary; the stopcock plug was replaced and operations from* above onwards carried out.

If the apparatus had been moved, the reagent level was rechecked in the same way, and in addition the readings P_1 and P_2 were checked.

Estimation Procedure

The measuring bulbs *C*, and the upper bore of stopcock *B*, were full of mercury.

With the sampling tool resting on but not piercing the can, and with stopcock *B* closed, and stopcock *N* open, the sampling line was evacuated to 5 mm Hg or less, as indicated on the manometer; stopcock *N* was then closed, and if there was no leak in the sampling line the manometer level did not change. (If the level changed, indicating a leak, this was rectified and the line re-evacuated.) With *N* closed, the can was pierced, when the mercury in the manometer flew back with a click. Stopcock *G* being open and reservoir *H* on the lowest hook, stopcock *B* was turned to allow gas from the sampling line and can to flow into *C* until one or both bulbs were full of gas. Stopcock *B* was closed, the water in the jacket was stirred and its temperature (T°C) noted. The level of mercury in *C* was brought to the appropriate index mark by closing *G* and operating the screw clip. The manometer reading (S_1) was then taken.

Reservoir *H* was hung on the topmost hook, stopcock *G* opened, and stopcock *B* turned to pass the gas into the absorption chamber. By raising and lowering *H*, the gas was passed to and fro the requisite number of times.

When absorption was complete the reagent level was brought to the mark, approximately by moving reservoir *H*, then exactly by closing stopcock *G* and operating the screw clip. Stopcock *B* was closed, stopcock *G* was opened, and the mercury level in the measuring bulb was adjusted to the appropriate mark as before. The manometer reading (S_2) was noted, and the thermometer read. (There had to be no temperature change during the estimation; any change would have caused serious errors if not allowed for in the calculation.) Finally the gas remaining in the measuring bulbs *C* was discharged by raising *H*, opening *G*, and turning *B* to allow the gas to flow out through the sampling line, following up with mercury until the upper bore of *B* was full.

Calculation of Results

P = manometer reading corresponding to index mark at atmospheric pressure (= P_1 or P_2 as appropriate)
S_1 = manometer reading with sample at index mark before absorption
S_2 = manometer reading with sample at index mark after absorption
T = water jacket temperature
B = barometric pressure
W = aqueous vapour pressure at temperature T°C

$$\text{Then \% oxygen} = \frac{S_1 - S_2}{B - W - P + S_1} \times 100$$

If the oxygen content was low (under 5 per cent), the barometric pressure could be assumed to be 76·0 cm and W to be 1·5 cm (corresponding to 18°C.) If in addition S_1 was about the same as P(+0·2 cm) the denominator could be assumed to be 75·0 in all cases without affecting the accuracy of the result to the first decimal place.

Then the expression became

$$\% \text{ oxygen} = \frac{(S_1 - S_2)}{75} \times 100 = \frac{4}{3}(S_1 - S_2)$$

which gave sufficient accuracy for factory control purposes.

The absorption reagent was alkaline pyrogallol or an alkaline solution of 1, 2, 4-triacetoxybenzene. The latter was found very satisfactory and was made up as follows: 10 g 1, 2, 4-triacetoxybenzene were dissolved by gentle warming in a closed flask with a solution of 13·5 g potassium hydroxide in 100 ml water.

Alkaline pyrogallol could be prepared by mixing equal volumes of solutions of 25 g pyrogallol in water to 100 ml and 100 g potassium hydroxide in water to 100 ml, and keeping in a well-stoppered bottle.

Fat Content of Meat or Fish Products

Determination with petroleum ether (Rapid Soxhlet)

This method could be used directly for dehydrated material but wet material had to be dried in sand first (see moisture determination in fresh meat) and transferred quantitatively to the Soxhlet thimble.

A 100 ml round bottomed flask (B.24 neck) was dried in an air-oven at 100°C, with a Soxhlet thimble lined with No. 1 filter paper.

The flask was weighed and then about 5 g of dry material was accurately weighed into the thimble. Forty millilitres of petroleum ether (40°–60°C) were poured into the flask and the thimble put into a plain Soxhlet extractor. After refluxing under a coil condenser for 4 hr (making sure that the solvent dripped back through the thimble), the extractor and thimble were removed and replaced by an extractor with siphon fitting. Most of the solvent was taken off, leaving a little in the flask so that the fat did not burn. The flask was put on a boiling water-bath and air blown into the neck *very gently* to remove traces of solvent. When there was no further odour of petroleum ether the flask was placed in an air oven at 100°C for about 15 min. At the end of this period it was removed from the oven, air blown gently over the fat and the flask cooled in a desiccator. After weighing, it was replaced in the oven and heated for a further 10 min, and the process repeated to constant weight.

N.B. If left in a desiccator overnight, sufficient oxygen could be taken up to affect the weight.

Determination using Gerber tubes (5 g cheese butyrometer tubes)

A quicker, but less accurate method, was as follows:

Approximately 10·5 ml of 55 per cent sulphuric acid were placed in 5 g cheese butyrometer tubes, to each of which samples of about 2·5 or 5 g (weighed to the nearest milligram) were transferred. This was best done by weighing the sample on a weighed polythene funnel in a stand and transferring through the funnel using a rounded spatula to push the meat or fish into the tube. Care was taken to clean the funnel thoroughly with a cloth before and after weighing each sample. The acid level in the tube was adjusted until it was within about $\frac{1}{2}$ in. from the neck constriction. This level varied slightly according to the tube and had to be determined by experience. Overfilling would cause the final fat level to be above the scale, and too low a level would take the fat off the scale at the other end. The tube was placed upright in a

water bath at 90°C without a stopper and left there for about 10 min. The contents of the tube were then mixed thoroughly by inserting a solid rubber bung and shaking. The tube was returned to the water bath and left there until digestion was complete (normally about ¾ hr but varying with the sample). The tube was removed from the bath and 1 ml of amyl alcohol added. The neck of the tube was carefully dried with filter paper, a cap inserted and after inversion the tube was centrifuged for 2 min at 1,500 r.p.m. After centrifuging, the tube was placed upside down in the water bath at about 70–80°C for about 10 min and then the percentage of fat read off the scale. Each division corresponded to 1 per cent fat. Since only 2·5 g of sample could be used in a '5 g' tube, the reading had to be multiplied by 2 to obtain the correct percentage.

Iodine value of fats (Wijs method)[51]

When an unsaturated fat or oil is treated with iodine, the iodine adds on to the molecule at the double bonds. The amount of iodine taken up in this way is thus an indication of the degree of unsaturation of the fats, and is expressed as the *iodine value*, which is defined as the weight of iodine absorbed by 100 parts by weight of the fat.

The Wijs solution was prepared by dissolving 8 g of iodine trichloride in about 200 ml of glacial acetic acid. This was mixed with 9 g iodine dissolved in about 500 ml of glacial acetic acid, the mixture diluted to 1 l. with glacial acetic acid, and the solution heated on a water-bath for 15 min to improve its keeping powers.

Some prior knowledge of the expected iodine value was required, and the weight of sample taken was roughly 20 g divided by the expected I.V.

The sample of fat (usually 0·2–0·3 g) was weighed into a glass stoppered flask of about 250 ml capacity, and dissolved in 10 ml of carbon tetrachloride. Twenty millilitres of iodine monochloride solution were pipetted in and the stopper, which had previously been moistened with 10 per cent potassium iodine solution, inserted. The mixture was allowed to stand in the dark for 30 min. A blank was carried out using the same reagents but omitting the fat. At the end of the 30 min 15 ml of 10 per cent potassium iodide solution and 100 ml of water were added and the mixture titrated with standard N/10 sodium thiosulphate solution.

$$\text{I.V.} = \frac{(\text{titre of sample} - \text{titre of blank}) \times 1{\cdot}269}{\text{wt. of fat}}$$

Peroxide Value of Fats

This was determined by a modification of Lea's iodometric method.[52]

A sample large enough to contain about 4 g of fat (approximately 10 g of dehydrated meat) was homogenized for about 2 min with 45 ml of chloroform, and the meat particles filtered off through a No. 1 paper with suction. It was important to have a large enough volume to pipette three 10 ml portions, and if some chloroform evaporated during filtration more was added. The fat solution was thoroughly mixed and 10 ml pipetted into a weighed porcelain dish and evaporated on a water-bath. The weight of the residual fat was determined.

Ten millilitres of glacial acetic acid were poured into a 150 ml wide-necked flask fitted with a two-hole bung carrying two short glass tubes. Carbon dioxide or nitrogen was bubbled into the flasks through one of the inlet tubes for about 5 min. Ten millilitres of the fat-chloroform solution were pipetted in and gassing continued for several minutes. Finally, on removing the gas delivery tube 1 ml of saturated potassium iodide solution was added and both inlet tubes stoppered. The flask was gently shaken and placed in the dark for 1 hr.

At the end of the specified time 30–50 ml of boiled-out (air-free) distilled water were added and the solution titrated with N/500 sodium thiosulphate solution using starch as indicator. A blank determination was carried out using 10 ml of chloroform in place of the fat-chloroform solution.

One millilitre of N/500 thiosulphate per gram of fat was equivalent to 1 millimole or 2 milliequivalents of peroxide, 16 mg of *active* oxygen or 32 mg of total peroxide oxygen per kilogram of fat.

$$\text{Thus:}\quad \frac{\text{ml of N/500 thiosulphate} \times 2}{\text{Wt. of fat in g}} = \text{m. equivalents of peroxide per kilogram of fat (i.e. peroxide value)}$$

Browning in Dehydrated Vegetables

In the course of storage at elevated temperatures, and occasionally during dehydration itself, melanoid pigments appeared in dehydrated foodstuffs as a result of non-enzymic browning, and some reasonably precise estimate of the extent of browning was required. It was not possible to measure the browning in absolute terms, but a quantitative comparison could be obtained between extracts of samples and a blank or between extracts of different samples. The usual basis of these comparisons was to determine the extinction value (Log I_0/I) where I_0 = intensity of incident light and I = intensity of transmitted light) of suitably prepared extracts as indicated by one or other of the usual spectrophotometers.

Little difficulty was found in preparing extracts from pale coloured or green vegetables or fruit, but where there was a high proportion of carotenoid pigments the extraction was more complex.

Browning in potato and green vegetables

The sample of dehydrated vegetable was prepared by grinding in a hammer mill to pass a 40-mesh sieve. In the case of potato 5 g samples were then extracted with 100 ml 60 per cent aqueous ethanol, left to stand overnight and filtered. The extinction value of the filtrate was read in a suitable photo-electric colorimeter (e.g., a Unicam SP 400), using wave length 400mμ and 66 per cent aqueous ethanol as a blank.

In the case of cabbage or other green vegetables 5 g samples were extracted with 100 ml 60 per cent aqueous ethanol, left overnight, and filtered. The filtrate was washed by shaking vigorously with an equal volume of benzene, centrifuged, and the benzene layer (containing chlorophyll) discarded. The process was repeated two or three times, until no more chlorophyll appeared in the benzene layer. The extinction value of the filtrate was then read as for potato.

Browning in dehydrated carrots[53]

The sample was ground in a hammer mill so that the carrot powder produced would pass through a 40-mesh sieve. The sample was then broken down to a convenient size by quartering.

0·5 g of this sample was weighed out, and placed in a centrifuge tube (1 in. × 6¼ in.), 10 ml of 60 per cent ethyl alcohol added, and the tube placed in a well-fitting rack which was clamped into a shaker in such a manner that the rack was inclined within the shaker at an angle of about 45 deg. to the horizontal. The tube was removed from the rack and shaken by hand to bring the carrot powder into suspension. The tube was then replaced in the rack and the shaker started immediately. The speed of shaking was such that none of the contents of the tube were lost by spilling, but enough to ensure thorough agitation and mixing. This shaking was continued for about 1 hr.

After shaking, the tube was centrifuged for 5 min at full speed (2,000 r.p.m.) and then allowed to stand for several hours or overnight.

The liquid was then sucked off with a teat pipette and placed in a 100 ml plastic-stoppered measuring cylinder. As much liquid as possible was removed from the centrifuge tube at this stage without removing any of the carrot powder in the process. Five millilitres of 60 per cent ethyl alcohol were then introduced into the centrifuge tube and again the tube was shaken by hand to bring the carrot powder into suspension. The tube was allowed to stand for about 1 hr when it was again centrifuged. This process of extraction with 5 ml portions of 60 per cent ethyl alcohol was carried out four times, and thus the final volume of alcoholic extract in the stoppered measured cylinder was a little less than 30 ml (a little being lost by evaporation and a little absorbed by the carrot powder).

This combined volume in the stoppered measuring cylinder was made up to 50 ml with 60 per cent ethyl alcohol. Ten millilitres of 60–80 petroleum ether were then added and the stopper replaced in the cylinder, which was shaken by hand a few times, the pressure being released occasionally by loosening the stopper. The cylinder was then clamped horizontally in the shaker with its long axis in the direction of the shaking movement. It was then shaken at about ⅓ speed for 15 min, i.e., at such a speed as to ensure thorough mixing of the contents.

The contents were then poured into a centrifuge tube and centrifuged for 5 min at full speed. The petroleum ether layer on the top was a bright clear yellow, and was removed with a teat pipette and discarded. The alcohol layer was a pale yellow-grey colour. Twenty-five millilitres of this were diluted to 50 ml with 60 per cent ethyl alcohol, and allowed to stand for a few hours or overnight. This diluted solution was stirred by allowing a stream of nitrogen to bubble through it.

The extinction value of the solution thus obtained was read on a suitable spectrophotometer at wavelength 450mμ, with 60 per cent ethyl alcohol as a blank.

Sugar Content of Potatoes

In the case of dehydrated potato, 5 g of the sample (ground to pass a 40-mesh sieve) were added to 100 ml of 60 per cent w/v ethyl alcohol refluxing in a 250 ml flask on a water-bath. The last traces of the powder were

washed in with a further 25 ml of alcohol. After an hour's boiling the flask was cooled and determinations carried out on aliquots of the filtered solution.

Raw potatoes were washed and samples taken by passing a cork borer through the centre of each tuber (the longitudinal axis) and retaining the cores, which were chopped up and mixed and 10 g lots weighed out for extraction. These were homogenized to a slurry with 40 ml 50 per cent ethyl alcohol, and transferred to a 250 ml volumetric flask, being washed in with 80 ml alcohol to make the total volume 120 ml. Two grams of calcium carbonate were added to adjust the acidity of the extract, which was then boiled in a steam bath for an hour, using a small funnel in the neck of the flask to condense vapour. It was then cooled and allowed to stand for several hours, preferably overnight. After making up to volume with neutral 95 per cent alcohol it was mixed thoroughly and allowed to settle. After filtering 200 ml were pipetted into a beaker and evaporated to 20–30 ml on a steam bath (*not* to dryness). This was transferred to a 100 ml volumetric flask and the beaker rinsed thoroughly, the washings being added to the contents of the flask.

Enough saturated neutral lead acetate solution (about 2 ml) was added to produce a flocculent precipitate, the flask was shaken and left standing for 15 min. Then the contents were made up to the mark with distilled water, mixed thoroughly, and filtered through a dry paper. Sufficient anhydrous sodium carbonate (or potassium oxalate) was added to precipitate all the lead and again it was filtered through a dry paper. (The filtrate being tested with a little sodium carbonate to test for complete removal of lead.) Twenty-five millilitres aliquots (equivalent to 2 g of original potato) were diluted to 100 ml for the determination.

An alternative technique, since drying facilities were available, was to dehydrate the raw potato (without scalding) and treat as above, which eliminated the tedious clearing of the extracts.

Estimation of sugars

After extraction, hexoses could be determined by the Nelson-Somogyi method,[54,55] and sucrose by the same method after hydrolysis. This was done by neutralizing a 25 ml aliquot to methyl red (pH 5·5) with 5 per cent acetic acid, adding 2 ml of invertase concentrate previously diluted 1 : 20, making up to 100 ml, and incubating for an hour at 37°C. Fructose was estimated by the resorcinol method[56] and the presence of glucose did not interfere with the reaction.

The picric acid method[57]

A less accurate method, but one which was useful for a quick check of the sugar content of a consignment, depended upon a colour reaction between reducing sugars and picric acid.

The consignment was sampled as adequately as possible, e.g., by taking one potato from every third bag. The potatoes were washed and quartered. One quarter was taken from each potato, all the quarters thoroughly mixed and then put through a mincer set to give a coarse cut. The resulting mince was mixed and sub-sampled by the quartering technique, the final sample being about 200 g. This material was then blended into a slurry in a homogenizer and filtered through muslin, the filtered juice being centrifuged to remove suspended starch, etc., and the supernatant diluted 1 : 4 for determination. If a homogenizer was not available juice could be extracted from the

minced potato by wringing out in muslin. If no centrifuge was available, filtering through fluted paper could be substituted, but it was important to keep the period of handling as short as possible.

One millilitre of the diluted juice was pipetted into a test tube graduated at 25 ml. Three millilitres of the picric acid solution (2 g/l) and 0·5 ml of 5 per cent NaOH were added. Five drops of 50 per cent acetone (prepared freshly each day by dilution with water) were added, and the tube promptly immersed in a boiling water bath. In 12 min the tube was removed, cooled and the contents diluted to 25 ml. After mixing the contents, they were compared with permanent standards in tubes of the same dimensions and the amount of sugar estimated by reference to Table 25.

TABLE 25

Determination of reducing sugars in raw potatoes, by comparison of colour produced with picric acid and the permanent standards

Total solids in the raw potato*	Reducing Sugars in Raw Potatoes, expressed as per cent of Dry Weight							
	Std. 1	Std. 2	Std. 3	Std. 4	Std. 5	Std. 6	Std. 7	Std. 8
per cent								
16	1·3	2·6	3·9	5·3	6·5	8·5	10·5	12·8
18	1·1	2·3	3·4	4·6	5·7	7·4	9·1	10·8
20	1·0	2·0	3·0	4·0	5·0	6·5	8·0	9·5
22	0·9	1·8	2·7	3·5	4·4	5·8	7·1	8·4
24	0·8	1·6	2·4	3·2	4·0	5·1	6·3	7·5
26	0·7	1·4	2·1	2·8	3·6	4·6	5·7	6·8

*If total solids were not determined in every case, the best approximation possible was used, taking into consideration the variety and the region in which the sample was produced.

The permanent standards were set up by mixing solutions of ferric chloride and cobalt chloride with hydrochloric acid according to the proportions in Table 26 below. The ferric chloride solution consisted of 200 g A.R. $FeCl_3$. 6 H_2O made up to 500 ml and filtered. The cobalt chloride solution was 150 g A.R. $CoCl_2$. H_2O made up to 500 ml and filtered. The hydrochloric acid was 5 ml of concentrated acid diluted to 50 ml with distilled water.

TABLE 26

Preparation of permanent standards

Standard Number	Ferric Chloride Solution	Cobalt Chloride Solution	Dilute HCl
	ml	*ml*	*ml*
1	11	2	8
2	11	4	8
3	22	9	8
4	27	15	8
5	22	22	8
6	16	30	8
7	14	40	8
8	12	50	8

One hundred millilitres volumetric flasks were used to make up the standards, and in each case the quantities shown were made up to the 100 ml mark with distilled water.

Ascorbic Acid (vitamin C)

General method

The determination of ascorbic acid with 2 : 6-dichlorophenolindophenol was accepted as a satisfactory method provided that the appropriate precautions were taken. The background is fully discussed in the literature.[58,59] For ordinary foodstuffs the simple titration gives accurate results but in certain caramelized and fermented products, and often in dehydrated foods, reducing agents of the nature of gluco-reductones are formed. These can react rapidly with the indophenol dye and lead to serious inaccuracies in the determination of ascorbic acid. Occasionally freshly dehydrated products contain such very small quantities of reductones, and the simple technique will suffice, but any material that has been in storage for more than a few weeks is liable to contain them.

It is not possible to remove either the reductones or the ascorbic acid from an extract by the simple addition of some reagent, and thus allow one or the other to be determined separately, but under certain conditions formaldehyde condenses more rapidly with ascorbic acid than with reductones. Thus titration of an extract without formaldehyde would measure the indophenol reducing power of the ascorbic acid plus reductones. Successive titrations of samples containing formaldehyde, spread over an appropriate period, would show first a rapidly decreasing reducing value while the ascorbic acid was being removed by condensation with formaldehyde, and then a slowly decreasing reducing value while the remaining reductones were being consumed. Under the conditions used for the determination the rate of disappearance of reductones is virtually linear and if a plot is made of reducing value (ml dye) against time, and extrapolated to cut the vertical axis (ml dye), the initial value of the reductones can be estimated and the initial ascorbic acid value then calculated.

The conditions under which the reaction of formaldehyde and reductones is linear and sufficiently slow for the present purpose, while the reaction of ascorbic acid is still comparatively rapid, are temperature approximately 20°C, pH 2·0 and formaldehyde concentration approximately 8 per cent.

A further complication is introduced by the presence of sulphite, since it reduces indophenol dye. It is possible, however, to remove sulphite, and this is done by bringing the extract to pH 0·6, adding formaldehyde, and allowing to stand for 8 min at 20°C. The lowering of the pH to 0·6 is necessary to prevent the condensation of ascorbic acid while the sulphite is reacting with the formaldehyde. This removal of sulphite is necessary only before the initial titration in which the total reducing value due to ascorbic acid and reductones is required. In subsequent titrations it will have gone.

Titration—usually of extract into dye—is commonly used for the actual determination, but some workers have difficulty in recognizing the end-point, especially if the extract is itself coloured. A photoelectric method of determination has proved highly satisfactory, almost any absorptiometer being satisfactory for the purpose.

Cabbage and peas are the two vegetables which contain abundant quantities of ascorbic acid and the procedures following are described for these vegetables. For other commodities (except strongly coloured fruit for which see the following section), which usually contain less ascorbic acid, larger quantities should be used.

Two grams of the finely ground dehydrated vegetable were weighed out and blended with 75 ml 5 per cent metaphosphoric acid in a homogenizer for 5 min at high speed. Longer blending was avoided as the heat generated could destroy some of the ascorbic acid. The extract so produced was made up to 100 ml with 5 per cent metaphosphoric acid, centrifuged for 10 min at 2,500 to 3,000 r.p.m. and filtered through a fluted filter paper.

If sulphite was present, 20 ml of the extract were pipetted into a 25 ml volumetric flask, 5 ml of acetone were added and the mixture thoroughly shaken. There was usually a very slight contraction in volume which was made up to 25 ml with water.

All operations were done as quickly as possible and if there were any delay the extract was placed in the dark, since riboflavin enters into a photochemical reaction with ascorbic acid.

The dye had to be standardized by accurately measuring 0·1 ml by means of a micro-pipette into a 10 ml pointed centrifuge tube. A micro-burette (5 ml) was filled with standard ascorbic acid solution (20 mg to 100 ml with 5 per cent HPO_3, then diluted 1 : 5) and this solution run rapidly into the centrifuge tube until the red colour of the dye was just discharged: this was the end-point. The whole titration had to be completed within 20–30 seconds from the moment of the first addition of the acid.

Then the extract from the vegetable was titrated from the micro-burette into 0·1 ml of the dye, and for the best results, 0·1 ml of dye required 1 to 2 ml extract; if the extract were weaker than this, less dye was used; if it were stronger it was diluted with 5 per cent HPO_3 solution.

As an alternative, a spectrophotometer was sometimes used, reading extinction coefficients of dye plus varying quantities of extract at the wavelength of maximum light absorption (approximately 510mμ). These were compared with the results of known additions of ascorbic acid.

Ascorbic acid in products with reductones

Since most products with which we were concerned did contain reductones, the technique at Aberdeen is given below in detail. Exact quantities of the buffer and other solutions used to establish the correct pH conditions are stated, but occasional checks should be carried out.

From the extract prepared as above, two 25 ml aliquots of the extract were pipetted into 50 ml conical flasks and marked A and B.

To A was added 0·48 ml 50 per cent w/v H_2SO_4 followed by 2·83 ml 40 per cent formaldehyde solution and the time noted. (This brought the pH to 0·6 and formaldehyde concentration to 4 per cent, while the total volume was increased to 28·31 ml.) Eight minutes was allowed before proceeding further.

To B was added 2·73 ml 30 per cent w/v sodium citrate solution followed by 6·93 ml formaldehyde and the time noted. (The pH was now 2·0 and the formaldehyde concentration 8 per cent, with the total volume increased to 34·66 ml.)

After 8 min, the reducing power of solution A; and after 30, 60 and 90 min that of solution B were measured thus:

To a colorimeter tube containing 7·5 ml buffer (pH 3·5)+0·53 ml 30 per cent sodium citrate+2·48 ml water, 3 ml dye were added, followed by 1·5 ml extract A. After shaking, the extinction value was read in the colorimeter after 30 seconds (a). The dye remaining was then decolorized by the addition of 1 drop of concentrated ascorbic acid solution and the blank reading (b) taken. By reading off the calibration curve, (a-b) was converted to ml of indophenol dye (c). The amount of indophenol reduced was (3-c), which was equivalent to the sum of ascorbic acid and reductones.

To another colorimeter tube containing 7·5 ml buffer+0·09 ml sodium citrate solution+2·91 ml water was added 3 ml dye followed by 1·5 ml extract B. (Again the pH should now be approximately 3·5.) The extinction value was read after 30 seconds, and the blank after decolorizing; and the difference was converted from the graph to ml of indophenol dye, as above. From this the amount of indophenol reduced could be calculated.

These readings of solution B were taken at 30, 60 and 90 min after the addition of formaldehyde and the results graphed. Extrapolation to 0 min gave a value equivalent to the reductones present, and subtracting them from the result of A gave the true ascorbic acid.

Ascorbic acid in strongly coloured materials

In the case of highly coloured fruit the intensity of pigment in the extract made any method of determination based on colour changes impracticable, but by the use of electrodes and a suitable circuit the end-point of the reaction between indophenol dye and ascorbic acid could be detected because of the ability of the indophenol dye to discharge the polarization at the electrodes.

There was no special sampling technique associated with this method, and the usual efforts to ensure random sampling were, of course, essential, but with fruit it was desirable to keep handling to a minimum, the ascorbic acid content being liable to fall off rapidly with bruising. Similarly the ascorbic acid content fell quickly during storage and if the ascorbic acid content of the fresh fruit were required, the determination had to be carried out with the minimum of delay. When storage was essential, cool storage was an advantage, but if the delay was necessarily prolonged beyond a few hours the ascorbic acid loss could be minimized by freezing the sample and holding deeply frozen—the colder the better. It should be noted that while tissues were held frozen there was some evaporation of ice and consequently a reduction in the moisture content of the material which had to be taken into account when calculations were made.

The ascorbic acid content of fruit varied very considerably,[60] and in the case of fruit with medium or low ascorbic acid content (less than 100 mg ascorbic acid per 100 g fresh weight), 50 to 100 g of the fruit was homogenized with 5 per cent metaphosphoric acid for a period sufficiently long to allow disintegration of the tissue but not long enough to allow the temperature of the homogenate to rise (usually about 1 min); the lower the temperature of the homogenate the better.

If the fruit was high in ascorbic acid (over 100 mg per 100 g) a more suitable echnique was to homogenize 100 g with 20 ml 20 per cent metaphosphoric acid, then weigh out 30 g of this homogenate and redisperse in 5 per cent

metaphosphoric acid. In each case the final volume of the homogenate was made up to 500 ml and filtered through B.P. fine-quality surgical gauze.

If frozen material was used it was not allowed to thaw until it was covered with the extracting medium.

Short lengths of 24 gauge platinum wire, cut by a piece of glass, were arc-welded to a suitable length of copper wire. Immediately after welding the region of the weld and the copper was covered with shellac to prevent any possibility of atmospheric corrosion.

The sealing of platinum wire into Pyrex or borosilicate glass was difficult and could only be done really effectively by the use of lead glass and a graded seal. The alternative to this difficult and time-consuming method was to draw out one end of a suitable length of glass tubing so that the platinum wire would just pass through the drawn-out section. The surplus glass was broken off, then collapsed on to the platinum, and the seal made liquid-proof by running wax down the inside of the electrode. The area of glass/platinum contact was kept as small as possible since liquid could seep into this area and set up electrode reactions which would interfere with the titration. Corrosion of the wire of the circuit or the electrode could also set up interference.

The amount of platinum required to protrude beyond the glass was less than a millimetre, only a pin-head. Any surplus was cut off by the glass cutting technique or ground down with 'frosted' glass. The electrodes were cleaned with concentrated nitric acid and then kept in metaphosphoric acid and dye, further cleaning with strong acid only being required very occasionally. Routine cleaning was done by wiping with filter paper.

The method used was a modification of that of Curtis.[61] If a very small current was passed across two platinum pin-head electrodes in a solution of ascorbic acid in metaphosphoric acid, they polarized and current ceased to flow. The potential across the two electrodes could be measured on an electronic millivoltmeter. If indophenol dye was added this removed the ascorbic acid and when in very slight excess discharged the polarization causing the current to flow. At this point the potential across the two electrodes changed and this change could be recorded on a potentiometer which could in turn be coupled to an automatic titrator. A 1·5 volt dry battery was used in series with a high resistance and the electrodes. Since the resistance of both solutions and electrodes varied considerably it was necessary to select the resistance according to the working conditions. It had to be of a magnitude which gave a potential drop across the electrodes when polarized of 800 to 1000 mV. A resistance box with steps of 1, 2, 3, 5, 10, 20, 40, 50 megohms was suitable and gave a wide range of resistances from which to choose.

The electrodes were placed one centimeter apart in a glass cell of approximately 30 ml capacity, e.g., a small weighing bottle. Stirring was by constant speed magnetic stirrer. The filtered dye solution was placed in a burette which could be part of an automatic titrator assembly.

Ten millilitres of the diluted ascorbic acid solution (which contained 0·1 mg per ml) were pipetted into the cell and titrated with dye (400 mgm 2 : 6 dichlorophenol indophenol/litre). The titration was carried out by adding increments of dye every 30 seconds and reading the EMF 20 seconds after the addition of dye. Near the end-point the increments of dye could be reduced. The end-point coincided with a permanent fall in the potential

across the electrodes from 800–1000 mV to 200–400 mV, which indicated the discharging of the polarization of the electrodes with the first slight excess of dye. This slight excess of dye also gave the solution a faint permanent pink colour. By plotting the EMF against the volume of dye added a typical titration curve was obtained. If tangents were drawn to the horizontal and vertical portions of the curve, then the vertical dropped from the point of intersection to the base line indicated the end-point.

Once this technique had been mastered the same effect could be obtained by setting the cut-outs on the automatic titrator. Careful adjustment then gave the same result as the manual method.

Stirring could be done by nitrogen bubbler using absolutely oxygen-free nitrogen. This had the advantage of giving an inert atmosphere above the surface of the solution. The same effect could be achieved with magnetic stirring by projecting a stream of nitrogen across the surface of the liquid. If nitrogen bubbling was employed then the rate of bubbling had to be kept constant by means of a needle valve and flowmeter and not changed after standardization of the dye and setting of the titrator.

Once good duplicate runs had been obtained with the dye and standard ascorbic acid, determinations were carried out on the fruit extracts. A 5 or 10 ml sample of the filtered metaphosphoric acid extract of fruit was placed in the titration cell and titrated with the indophenol dye to the same end-point.

β-Carotene (Provitamin A) in Carrots

Since approximately 90 per cent of the carotenoid pigments in carrot is β-carotene, a reasonable approximation to the β-carotene content may be obtained by measuring the total carotenoid content.

Samples of 2 g dehydrated product were convenient. The material was covered with cold water and allowed to stand until reconstituted. Surplus water was drained off and the sample covered with acetone and stood in the dark for approximately 1 hr, with occasional shaking. Subsequent operations were carried out in very subdued light.

The sample was transferred to a Buchner funnel with a bed of pulped filter paper. After the acetone had been drawn through the suction was disconnected and more acetone added to the sample in the funnel, together with about 20 ml petroleum ether (60°–80°). The sample was allowed to soak for a few minutes, with occasional stirring, and the liquid was then drawn through and the procedure repeated five times.

The acetone-petroleum ether solution was then transferred to a large separating funnel, a few ml of petroleum ether (which had been used for rinsing out the Buchner funnel) added, and the funnel shaken until the liquids formed a single phase. Distilled water was then added until the petroleum ether separated from the acetone-water phase. The water layer was run off and the washing process repeated six times.

The petroleum-ether solution, which contained the carotene, was transferred to a 250 ml conical flask containing sodium sulphate and swirled until it became clear (dried by the sodium sulphate); it was then transferred to a volumetric flask of suitable size and made up to the mark with petroleum ether which had been used to wash the remaining colour out of the sodium sulphate. The estimation of carotene was carried out by measuring the extinction value of this extract in 1 cm silica cells in a suitable spectrophotometer

at a wavelength of 450mμ. The basis of the subsequent calculation was that for β-carotene E 1 per cent 10 cm is 2590 at 450mμ and there is a linear relationship between the extinction coefficient and the carotene content.

Nitrogen (Kjeldahl)

Because of the heterogeneity of most foodstuffs, the material had to be minced (or ground) and mixed before sampling. The weight of sample taken depended on the nitrogen content but did not exceed 1 g dry weight. When the nitrogen content was very low, as in some plant products, it was advisable to dilute the digest to a smaller volume than 500 ml.

A suitable weight of sample was accurately weighed in a small glass tube or in an ashless filter paper and transferred to a 150 ml Kjeldahl flask. 10·7 g of catalyst (130 g K_2SO_4 + 4 g HgO) were added, followed by 25 ml conc. H_2SO_4. The contents of the flask were heated, gently at first, then more strongly until clear and then for a further 2 hr. The flask was cooled, the contents transferred to a 500 ml standard flask and made up to 500 ml with distilled water.

An aliquot, of up to 5 ml, was transferred to a Markham distillation apparatus[62] with 10 ml of sodium hydroxide/thiosulphate solution (500 g NaOH + 50 g thio to 1 l.).

The distillate was collected in 10 ml of boric acid solution and the ammonia titrated with standard H_2SO_4. (The boric acid solution was made by dissolving 5 g boric acid in 200 ml alcohol and 700 ml water, adding 10 ml indicator solution, and making up to 1 litre; the indicator solution by dissolving 0·033 g Bromocresol Green and 0·066 Methyl Red in 100 ml alcohol.)

With 0·00714 N sulphuric acid, 1 ml was equivalent to 0·1 mg nitrogen (after the usual blank had been subtracted).

Biological Value of Protein

Animal experiments are expensive and time-consuming, but chemical hydrolysis has not always proved satisfactory for determining the nutritive value of protein, especially in foodstuffs that have been heated during processing. The Minstry of Agriculture, Fisheries and Food Research Establishment tried out a microbiological technique, recently developed[63] at the National Institute for Research in Dairying, which seemed very promising.

The test organism was *Streptococcus zymogenes* (National Coll. Dairy Organisms 592), which is itself vigorously proteolytic; but pre-treatment of the test samples with papain improved the assay. It speeded growth, and improved the linearity and reproducibility of the dose-response curve.

Microbiological Quality Control

In the accelerated freeze-drying process no multiplication of bacteria or formation of toxins occurred during the drying stage itself; as was demonstrated on many occasions in practice. On the other hand the drying process was in no way a sterilizing process. Microbiological control, therefore, was concerned with the state of the material entering the drying cabinets and with avoidance of contamination at the packaging stage. In general, therefore, the conditions necessary for maintenance of satisfactory standards were those applicable to all dehydration processes, and these have been described for

vegetables,[64,65] though little has been published about fish or meat, presumably because neither of these commodities have ever been important products of the hot-air drying industry.

The microbiological control of the process involved the use of various sampling and bacteriological techniques, appropriately modified for the particular purpose; it involved plant hygiene and the development and application of measures which would not only reduce microbial contamination of the foodstuff to a minimum, but which would also be practicable in a production factory; it involved the setting of standards for the finished product, and some consideration of what happened when it was reconstituted and prepared for eating.

Sampling and bacteriological technique

For a complete appreciation of the microbiology of the process, samples were taken at all stages of the process, from the raw material right through to the packed product. The information so obtained was invaluable in showing where plant hygiene could be improved. Such extensive sampling would normally be unnecessary in commercial operation, and only random sampling of the dried material should be required. It would be ideal to sample each production batch but if this were impossible sampling should be carried out as frequently as was practicable and at least twice per day. Sampling should be by the usual aseptic techniques direct into sterile containers.

The systems used for the microbiological examination of dehydrated foods were based on the work of Rishbeth[64] on vegetables, modified to suit the circumstances, e.g.,

> Serial dilutions using ¼ strength Ringer's solution were used for the MacConkey broth dilutions when examining for coliform organisms.
> Thermophilic counts were obtained using dextrose-tryptone agar. Vegetables may be contaminated with thermophilic organisms from soil but none have been isolated from AFD meat.
> Malt (or wort) agar was included when examining vegetables to determine the load of yeasts and moulds.
> The 22°C count on dehydrated fish was examined after 5 days incubation and the 37° count after 3 days. This compared with the 3 and 2 days incubation periods for meat and vegetables, and was due to the somewhat slower rate of growth of the fish microbial population. Aged sea-water (stored for at least 1 month) was used in preparing salt-water agar for the examination.
> Tetrathionate broth and Selenite F were used in parallel when examining for the presence of Salmonella, the enrichment media being subcultured at 24 hr and at 48 hr on to MacConkey agar, Hynes desoxycholate citrate agar and Wilson and Blair agar.
> The range of diagnostic media for the classification of bacteria was contracted or extended as circumstances dictated.

A scheme for the examination of dehydrated foods is given in Table 27.

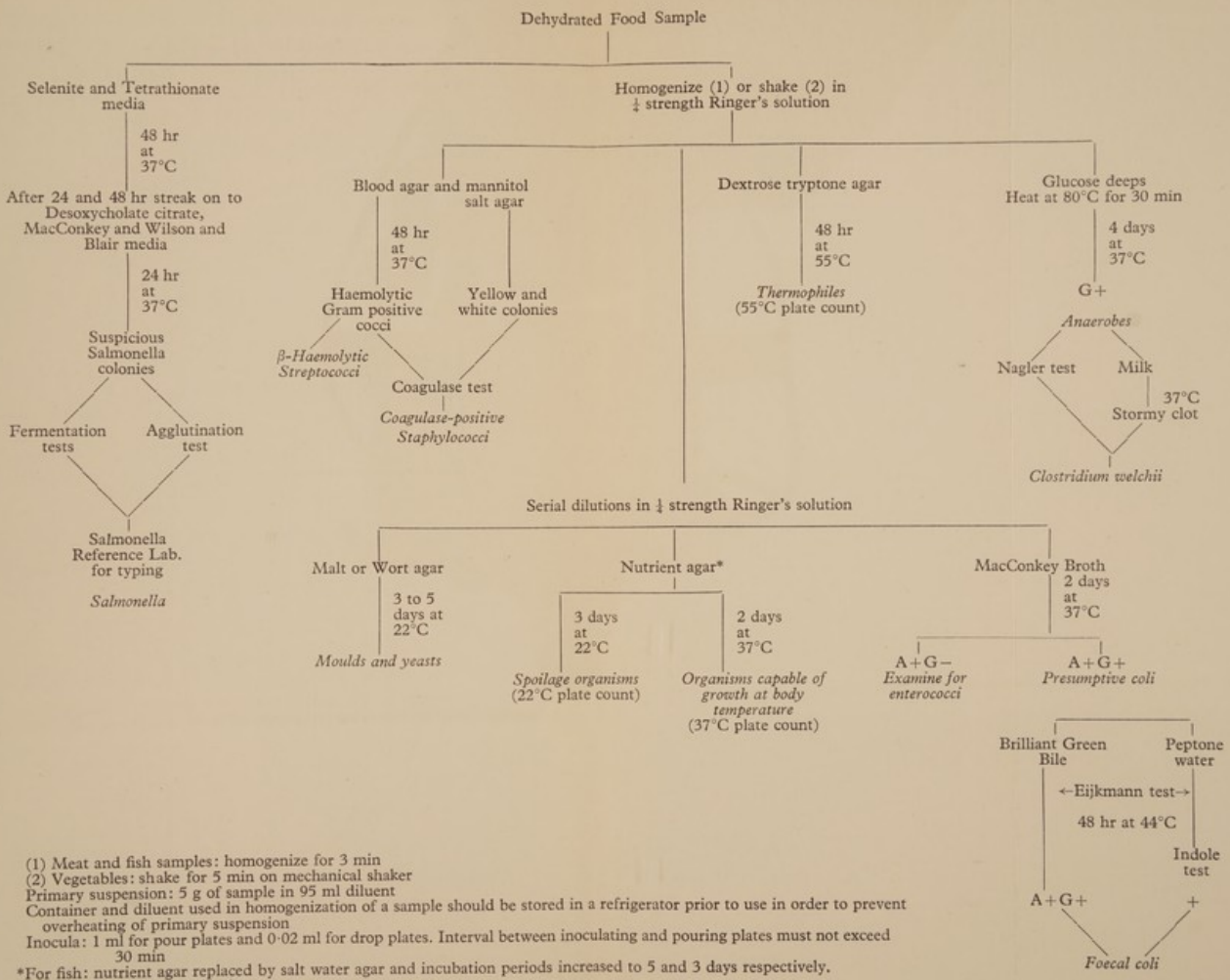
Plant Hygiene

It is, of course, axiomatic to say that all food equipment must be kept clean and that routine bacteriological examination is essential if high standards are to be maintained. Similarly high standards of cleanliness and protective clothing (covering of hair, use of overalls and of gloves wherever possible) are required from the operatives. One or two points, however, seemed, from experience at Aberdeen, to be of particular importance.

In the preparation of meat, the butchers' knives could spread contamination. The raw meat carried a substantial bacterial population, and the build-up on

TABLE 27

Outline of General Plan for Microbiological Examination of Dehydrated Foods



(1) Meat and fish samples: homogenize for 3 min
(2) Vegetables: shake for 5 min on mechanical shaker
Primary suspension: 5 g of sample in 95 ml diluent
Container and diluent used in homogenization of a sample should be stored in a refrigerator prior to use in order to prevent overheating of primary suspension
Inocula: 1 ml for pour plates and 0·02 ml for drop plates. Interval between inoculating and pouring plates must not exceed 30 min

*For fish: nutrient agar replaced by salt water agar and incubation periods increased to 5 and 3 days respectively.

A+: Acid production
G+: Gas production

knives was considerable. They were, therefore, changed frequently and scalded prior to re-use. The tables on which the meat was cut were thoroughly scrubbed with detergent and scalded after use. Drying trays were scalded and stored in dust-free cupboards. They were then swabbed with 70 per cent alcohol before use. Meat should not have been pre-cooked well in advance of the drying cycle, but if for any reason this occurred then apart from being cooled quickly it was held in cold storage.

TABLE 28

Proposed microbial levels for dehydrated foods

Product	Organisms per g of food at					
	22°C		37°C		55°C	
	Should not exceed	Never greater than	Should not exceed	Never greater than	Should not exceed	Never greater than
	A. MEAT					
AFD Raw Beef Steak	750,000	2,500,000	250,000	1,000,000		
AFD Raw Mutton	750,000	2,500,000	250,000	1,000,000		
AFD Raw Pork	75,000	250,000	50,000	75,000		
AFD Pre-cooked Meats	1,000	1,000	100	100		
	B. FISH					
AFD Raw Cod	500,000	1,000,000	75,000	250,000		
AFD Pre-cooked Fish	10,000	10,000	1,000	1,000		
	C. VEGETABLES					
AFD Vegetables (blanched	5,000	500,000	2,500	100,000	100	1,000
AFD Pre-cooked Vegetables	1,000	1,000	100	100	—	100

Raw vegetables after trimming usually had a count of about 10^4 to 10^5 bacteria per gram. After scalding they were sterile or almost so, but if water scalding was used, followed by the usual cooling conveyor, contamination was likely to occur during cooling, even when hot-water jets were arranged to keep the conveyor as free from build-up as possible. With steam scalding, where the vegetable was spread before scalding on trays which were subsequently placed in a rack-car for cooling, this problem was avoided, and even after exposure to the air of the factory for periods of ½ to 1 hr the vegetables remained virtually sterile.

No build-up of bacteria occurred in the strip-washer or water-scalder provided that they were kept scrupulously clean, the former cleaned by water jets, the latter with boiling detergent solution.

Hand spreading of vegetables on the drying trays involved risk of contamination, but spreading with metal rakes was slow and did not give the uniformity of bed which could be attained by use of the fingers. Good personal hygiene was very essential here, and equally essential at the packing stage.

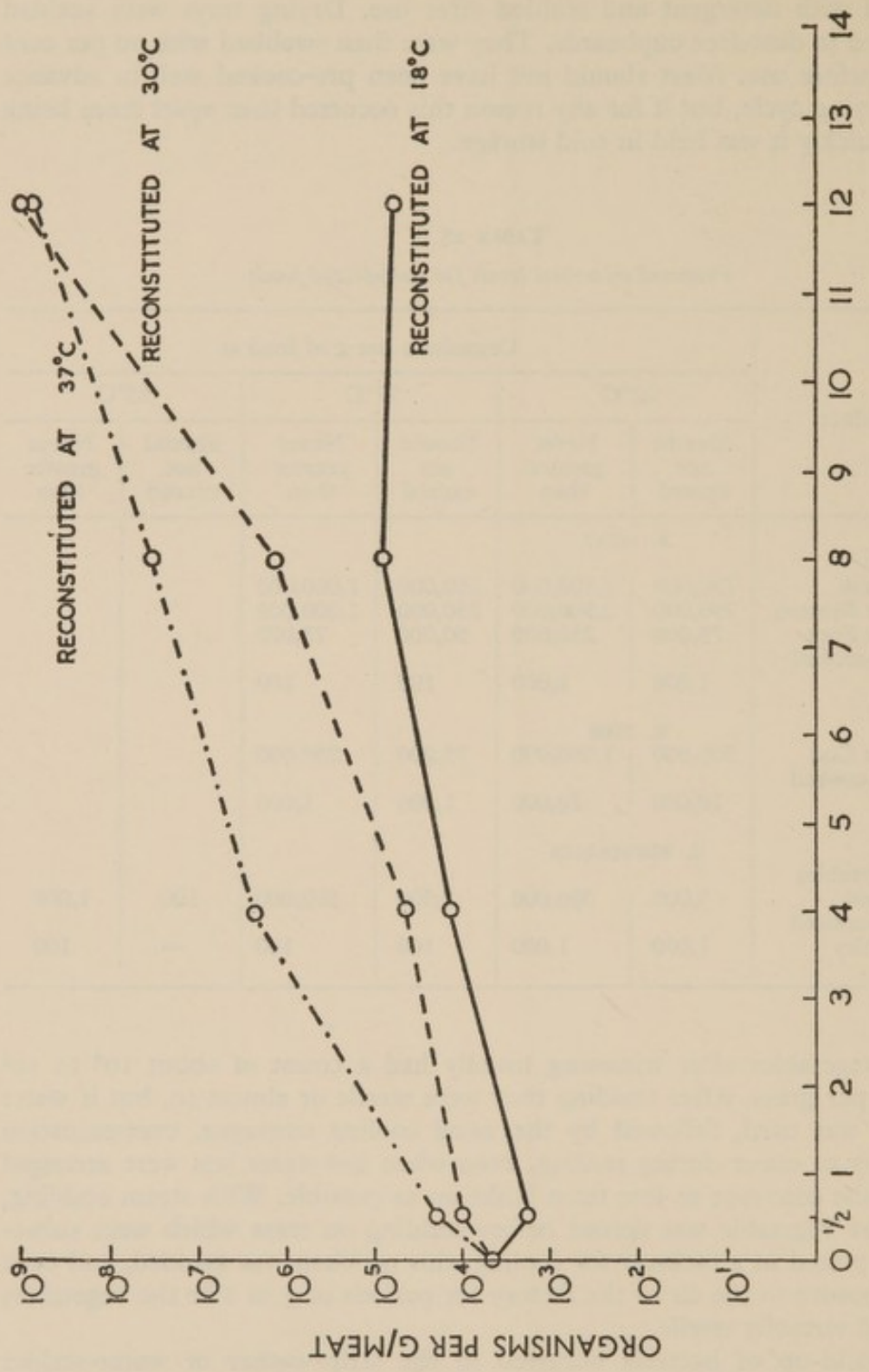


FIG. 15. *Microbiological evaluation of reconstitution: Beef*

The blast-freezer, which was used for freezing many commodities immediately before drying, could also cause serious bacteriological contamination if the interior were not kept scrupulously clean.

Proposed microbiological tolerances for dehydrated foods

The work of Rishbeth and Haines[64,65] laid the foundations on which standards were based for dehydrated vegetables supplied to the armed services. There were no legally enforceable standards for dehydrated foods but it was shown that in commercial practice the limits indicated for vegetables need not be exceeded, and it was felt that such limits could be accepted by manufacturers as a 'code of practice'. Experience of AFD meats produced at the Experimental Factory under conditions closely approaching commercial practice suggested that the figures proposed for meat and fish also could reasonably be attained. The levels are given in Table 28.

With the majority of vegetables the lower microbial level could be easily reached. The upper level was intended to cover leaf vegetables, e.g., the Brassica family, and beetroot.

Coliform organisms and coagulase-positive staphylococci could be absent both in 0·05 g of either raw dehydrated meat or fish, and salmonella absent in 10 g. With vegetables, coliforms could be absent in 0·1 g.

Reconstitution

Microbial growth was not possible during storage of a dehydrated food (provided it was packed in such a manner that it was not able to absorb moisture from the atmosphere to enable such growth to commence), and indeed some diminution of the bacterial count was found as storage proceeded.[64] Even so, the food was in many cases not sterile and there was, therefore, a possibility of bacterial growth once the food had been immersed in water for reconstitution. AFD foods required only short periods of soaking and were therefore particularly safe in this respect, but there is always the possibility of abuse. Fig. 15 shows the effect of soaking a sample of AFD beef in water at temperatures of 18°C, 30°C and 37°C for periods ranging from 30 min to 12 hr. At 18°C soaking for up to 8 hr brought about relatively little increase in bacterial population but at the higher temperature there was a considerable and rapid rise.

References

1. Forrest, J. C. (July, 1959). Large scale freeze-drying equipment for foodstuffs. *Brit. Chem. Eng.*, 1–5.
2. Hanson, S. W. F. (1959). Accelerated freeze-drying of food. *Food*, **28**, 245–8.
3. Rolfe, E. J. (1956). An improved method for dehydrating meat. *Food*, **25**, 199.
4. Harper, J. C. and Tappel, A. L. (1957). Freeze-drying of food products. *Advances in Food Research*, **7**, 171–234. New York Academic Press Inc.
5. Salwin, H. (1959). Defining minimum moisture contents for dehydrated foods. *Food Tech., Champaign*, **13**, 594–5.
6. Forrest, J. C. (Sept., 1960). The accelerated freeze-drying process. Residential course on 'Recent Advances in Food Science', *Royal College of Science and Technology, Glasgow* (in the Press).
7. Forrest, J. C. (March, 1961). Evacuating equipment for the accelerated freeze drying of foodstuffs. Symposium on user experience with large scale evacuating equipment, held by the Institute of Mechanical Engineers (in the Press).
8. Record, B. R. and Taylor, R. (1958). Freeze-drying equipment. *Biochem. J.*, **68**, 420–430.
9. American Society for Testing Materials. Standards on Plastics, Designation E96–53T. 1018.
10. Taylor, A., Karel, M. and Proctor, B. E. (June, 1960). *Mod. Packag.*, **33**, 131.
11. American Society for Testing Materials. Standards on Plastics, Designation D1434–58, 460.
12. Ward, K. (1961). Accelerated freeze drying II—Fundamental design problems. *Food Manuf.* **2**, 60.
13. Lederman, A. N. (Sept., 1960). Proper selection of freeze drying equipment. Research and Development Associates Military–Industry Conference on 'Freeze Drying and Other Dehydration Methods of Food Preservation', Q.M.F.C.I., Chicago, U.S.A.
14. Tucker, W. H. and Sherwood, T. K. (1948). Vacuum dehydration using liquid absorbents. *Industr. Engng. Chem.* (*Industr.*) **40**, 832.
15. Copson, D. A. and Decareau, R. V. (1957). Microwave energy in freeze drying procedures. *Food Res.* **22**, 402.
16. Smithies, W. R. and Blakeley, T. S. (1959). Design of freeze drying equipment for the dehydration of foodstuffs, *Food Tech., Champaign*, **13**, 610.
17. Brown, H. D., Miller, Mary K., Alban, K., Short, R., Schulkers, Ruth and Murnave, Cecelia (1944). Carotene, flavour, colour and

refractive indices of carrots grown at different fertility levels. *Proc. Amer. Soc. hort. Sci.*, **44**, 463.

18. Refrigeration Research Foundation (1956). Information Bulletin No. 56–1, 5.
19. GOODING, E. G. B., ROBERTSON, JEAN, TAPSFIELD, D. and WALMSLEY, RUTH (1959). *Min. of Ag., Fish and Food.* Unpublished work.
20. HARPER, R. H. and ZSCHEILE, F. P. (1945). Carotenoid content of carrot varieties and strains. *Food Res.*, **10**, 84.
21. BURTON, W. G. (1948). *The Potato*, (London: Chapman and Hall, Ltd), 223.
22. MULLER-THURGAU (1882). Quoted in Winton, A. L. (1935). The Structure and Composition of Foods. **2**, 1610. New York. (John Wyllie and Sons).
23. GOODING, E. G. B. and DUCKWORTH, R. B. (1956). The effect of post-harvest storage conditions of raw potatoes on the storage life (at tropical temperatures) of their dehydrated products. *J. Sci. Food Agric.*, **7**, 6.
24. JACOBS, MORRIS B. (1958). The Chemical Analysis of Foods and Food Products. Third Edition (New Jersey). D. van Nostrand Co. Inc. 393 and 843.
25. TAPPEL, A. L. (1956). Freeze-dried meat II—The mechanism of oxidative deterioration of freeze-dried beef. *Food Res.*, **21**, 195.
26. PENNY, I. Unpublished work.
27. Annual Report of the Hormel Institute, 1958–59, University of Minnesota, U.S.A.
28. KROENER, W. and WAGNER, H. (1942). Substances in potato responsible for odour and taste. *Naturwissenschaften*, **30**, 586.
29. TOMKINS, R. G., MAPSON, L. W., ALLEN, R. J. L., WAGER, H. G. and PARKER, J. (1944). The storage of dried vegetables, *J. Soc. chem. Ind., Lond.*, **63**, 225.
30. URI, N. (1956). Metal ion catalysis and polarity of environment in the aerobic oxidation of unsaturated fatty acids. *Nature*, **177**, 1177.
31. SHARP, J. G. (1953). Dehydrated meat. *D.S.I.R. Special Report No.* 57, 101 London, H.M. Stationary Office.
32. ROSS, A. F. (1948). Deterioration of processed potatoes. *Advances in Food Research*, **1**, 257.
33. JONES, N. R. (1959). Browning reactions and the loss of free amino acid and sugar from lyophilised muscle extractives of fresh and chill stored codling (*Gadus callarias*): *Food Res.*, **24**, 704.
34. HODGE, J. E. (1953). *Agric. and Food Chem.*, **1**, 928.
35. ANET, E. F. L. G. and REYNOLDS, T. M. (1957). Chemistry of non-enzymic browning I—Reaction between amino acids, organic acids and sugars in freeze-dried apricots and peaches. *Australian J. Chem.*, **10**, 182.
36. ANET, E. F. L. G. and REYNOLDS, T. M. (1957). Some crystalline amino acids and deoxysugar amino acids. *Australian J. Chem.*, **10**, 193.
37. ANET, E. F. L. G. and REYNOLDS, T. M. C.S.I.R.O. Australia Division of Food Preservation and Transport. Annual Report for year ending 30 June, 1959, 4.

38. REGIER, L. W. and TAPPEL, A. L. (1956). Freeze dried meat III. Non-oxidative deterioration of freeze-dried beef, *Food Res.*, **21**, 630.

39. GOODING, E. G. B., DUCKWORTH, R. B. and HARRIES, J. M. (1956). The effect of post harvest storage conditions of raw potatoes on the storage life (at tropical temperatures) of their dehydrated products. *J. Sci. Food Agric.*, **7**, 444.

40. GOODING, E. G. B. and TUCKER, C. G. (1958). A comparison of the suitability of clamp-stored and barn stored potatoes for processing. *J. Sci. Food Agric.*, **9**, 448.

41. GOODING, E. G. B. and TUCKER, C. G. (1955). Dehydration of carrot. *Food Manuf.*, **30**, 447.

42. WAGER, H. G. (1955). The browning reaction in dehydrated carrot and potato: Its initiation and the separation and partial characterization of an intermediate from dehydrated carrot. *J. Sci. Food Agric.*, **6**, 57.

43. LEGAULT, R. R., HENDLE, C. E., TALBURT, W. F. and RASMUSSEN, L. B. (1949). Sulphite disappearance in dehydrated vegetables during storage, *Industr. Engng. Chem.* (*Industr.*), **41**, 1447.

44. HEARNE, J. F. and TAPSFIELD, D. (1956). Some effects of reducing during storage the water content of dehydrated strip potatoes; *J. Sci. Food Agric.*, **7**, 210.

45. SHEWAN, J. M., MACKINTOSH, RUTH G., TUCKER, C. G. and EHRENBERG, A. S. C. (1953). The development of a numerical scoring system for the sensory assessment of the spoilage of wet white fish stored in ice. *J. Sci. Food Agric.*, **4**, 283.

46. Ministry of Agriculture, Fisheries and Food: National Food Survey Committee (1961) Domestic Food Consumption and Expenditure, 1959. London, H.M. Stationery Office.

47. ALLEN, R. J. L. and MAPSON, L. W. Unpublished work.

48. MILLER, D. S. and ROLFE, E. J. Unpublished work.

49. POTTER, E. F. (1954). *Food Tech., Champaign*, **8**, 269.

50. Vegetable Dehydration Practice, 1958. London, Ministry of Agriculture, Fisheries and Food.

51. NICHOLLS, J. R. Aids to Analysis of Food and Drugs, 1942, 6th Edition, 323 (London: Balliere, Tindall and Cox).

52. LEA, C. H. D.S.I.R. (1938). Food Investigation Special Report No. 46, 106. London, H.M. Stationery Office.

53. WAGER, H. G. (1953). Unpublished work.

54. NELSON, N. (1944). *J. biol. Chem.*, **153**, 375.

55. SOMOGYI, N. (1952). *J. biol. Chem.*, **195**, 19.

56. ROE, J. H., EPSTEIN, J. H. and GOLDSTEIN, N. P. (1949). *J. biol. Chem.*, **178**, 839.

57. ROSS, A. F., HILBORN, M. T. and JENNESS, L. C. (1945). *Food Pack.* **26**, 10, 38.

58. MAPSON, L. W. (1943). *J. Soc. chem. Ind., Lond.*, **62**, 223.

59. HARRIS, L. J. and MAPSON, L. W. (1947). *Brit. J. Nutrition*, **1**, 1.
60. JOYCE, A. E. (1959). *Scientific Horticulture*, **14**, 116.
61. CURTIS, R. C. (1958). *Analyst*, **83**, 7.
62. MARKHAM, R. (1942). *Biochem. J.*, **36**, 790.
63. FORD, J. E. (1960). Unpublished work.
64. RISHBETH, R. (1947). The bacteriology of dehydrated vegetables. *J. Hyg. Camb.*, **45**, 33.
65. HAINES, R. B. Unpublished work.

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