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Dehydration of food products

A review

S. D. HOLDSWORTH

Introduction

This review of dehydration of food products is concerned with the physical aspects of drying and their significance in the preservation of the organoleptic qualities of food. The literature on dehydration is extensive and it has been necessary to limit the scope of this article to topics which have not been dealt with in previous reviews. The more recent information has been included together with an extensive bibliography directing the reader's attention to food products. It is hoped that this review will be useful in giving guidance to those who have to search the literature, although no attempt has been made to cover the subject exhaustively. The earlier references on dehydration are given in Baker & Foskett (1958) covering the period from 1936–56 and this is supplemented by the three-volume bibliography of Bickle (1951) covering all aspects of industrial drying. A bibliographical guide to dehydration and dehydrated products has recently been published (Holdsworth, 1971). The major textbooks on dehydration of food products are by Von Loesecke (1943, 1955), Morris (1947) and Van Arsdel & Copley (1964). Other texts and monographs on specific topics are included in the text.

One of the more important themes of this review is the usefulness of the basic study of the transport processes—heat transfer, mass transfer (moisture diffusion) and momentum transfer (fluid flow)—in relationship to the design, development and operation of processing equipment. Examples are given showing that, by studying the basic mechanisms, it has been possible to make modifications to existing techniques or development of new techniques as well as to scale up pilot plant. This is, of course, associated most closely with the chemical and organoleptic aspects, although the emphasis here will be on the former, whereas the latter have been widely discussed in other reviews.

Drying techniques other than freeze drying

Factors affecting the rate of drying

The development of a satisfactory drying process depends upon the biochemical and biophysical properties of the particular foodstuff. It is necessary to establish the

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drying rate under various conditions prior to designing the plant in order to obtain reliable correlations in relation to scale up. It is also possible by studying the heat and mass transfer characteristics to establish the mechanism of the drying process. This type of information is very important since it will be possible, by studying various theoretical models, to establish the rate-determining stage and consequently to improve the drying time by suitable modifications. Several examples will be given throughout this review to substantiate this concept—such as the role of inert gases in freeze drying and explosion puffing as an intermediate process.

The most important requirement in industry is to have a relatively rapid process commensurate with high quality. It is, of course, important not only to study those factors which lead to a reduction in drying time but also to consider the rate of reconstitution of various food products, since rapid or 'instant' reconstitution is demanded today.

The main factors which affect the rate of drying and the time of the drying cycle are: (a) physical properties of the product; (b) geometrical arrangement of product in relation to heat transfer surface or medium; (c) physical properties of drying environment, and (d) characteristics of the drying equipment.

Physical properties of the product

The main aspects to be considered here are particle size and geometry. Any theoretical formula for drying time must incorporate a term expressing the fact that the thicker the product the longer the time it will take for moisture to be removed. The migration of moisture from inside a particle to the surroundings is controlled either by internal migration of moisture to the surface or diffusion of moisture from the surface to the atmosphere. In general the chief factor controlling the rate of mass transfer is the migration of the water within the sample and through the residual dried mass. The diffusion away from the surface is usually rapid and may be accelerated by either applying vacuum—i.e. increasing the water vapour pressure differential—or by blowing dry air over the surface. It is, however, restricted by excessive loading on the drying trays, when the diffusion and movement of water vapour between particles becomes restricted. With solid food products the size and shape are predetermined by the product requirements and it is often not possible to reduce the drying time—for example of whole carrots. However with liquid products rapid rates of drying may be obtained by using sprays of fine droplets as in spray drying. The effect of temperature and heat transfer within the products must also be taken into account.

Typical drying curves showing the effect of the size of potato strips on the residual moisture content have been obtained by Ede & Hales (1948). These show that the drying time is considerably affected by particle size and shape.

It is generally observed with many products that initial rate of drying is constant and then the rate decreases, sometimes at two different rates (see Fig. 1). The drying

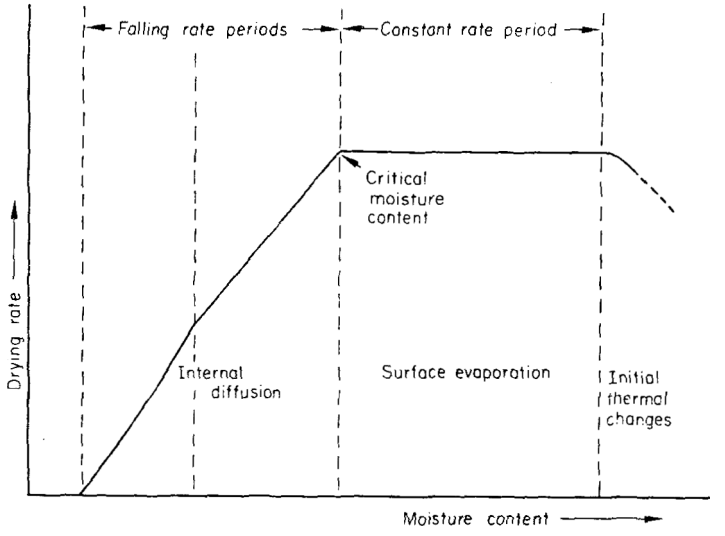

FIG. 1. Idealized curve for drying rate.

curve is divided into the constant rate period and falling rate period areas as these represent different mechanisms of drying. The constant rate period indicates that excess surface moisture is being removed and the falling rate periods indicate the progressive receding of the interface between dry material and wet material. Several internal mechanisms of moisture transfer have been suggested (Treybal, 1968; Perry, 1950), for example:

(a) laminar viscous flow of water vapour (Poiseuille flow) would be expected to cause the permeability to vary inversely with temperature;
(b) capillary flow processes in which surface tension, density and viscosity are important;
(c) thermal diffusion which requires large temperature for gradients to be effective;
(d) vapour diffusion would be expected to cause the diffusion coefficient to vary with square root of the temperature cubed;
(e) surface diffusion; and
(f) molecular diffusion (Knudsen flow) would be expected to cause the diffusion coefficient to vary with square root of the temperature.

However, most results are interpreted either in terms of a capillary flow process or, alternatively, vapour diffusion process. The former is obviously applicable to the drying of particles and granules and the latter most appropriate to the drying of foodstuffs. Jason (1958) has examined the diffusion model to predict the transition between the constant rate and falling rate phases during the drying of cod muscle slabs. The main conclusions from this extensive study are that the duration of the constant rate period is related to the rate of evaporation per unit area, effective diffusion constant, sample size and free water concentration. Two phases were also observed for the falling

rate period, one in which the diffusion coefficient was constant and the other with a considerably reduced diffusion coefficient.

Haque & Eubank (1964) have studied the unsteady state diffusion of moisture during drying of the spherical shell of copra and have shown that the appropriate equations for a diffusive mechanism hold. The value of the diffusion coefficient $2{\cdot}9 \times 10^{-5}$ cm^2/sec compared well with the self diffusion of water, namely, $2{\cdot}5 \pm 0{\cdot}3 \times 10^{-5}$ cm^2/sec.

Chirife (1971) has examined the through circulation drying of tapioca root slices and has concluded that water migrates by a process of liquid diffusion on the basis of the fact that the diffusivity varies with temperature in accordance with an Arrhenius type relationship and also the exponential nature of the residual moisture-time relationship. Saravacos & Charm (1962) studied the mechanism of fruit and vegetable dehydration and found a constant drying period present with most of the products examined. From the study of potato dehydration it was concluded that a diffusion type mechanism operated. This is contrary to the conclusions of Gorling (1958) who considered that a combination of capillary forces and vapour diffusion transported water to the surface of potato samples. It is interesting to note that the role of osmotic forces has not been fully explored and few workers have discussed their importance. The need to take into account the changes of internal structure due to shrinkage has been discussed by Crank (1958) and Gorling (1958).

Mathematical representation

For the falling rate period, when liquid diffusion is controlling, equation (1) applies to a slab of thickness $2L$ with diffusion from the opposite forces.

$$\frac{W - W_e}{W_o - W_e} = \frac{8}{\pi^2}\left[e^{-b} + \frac{e^{-9b}}{9} + \frac{e^{-25b}}{25} + \ldots\ldots\right] \qquad (1)$$

where W is the average moisture content (dry basis) at any time θ, W_o is the average moisture content at the start of the diffusional period $\theta = 0$, W_e is the average moisture content when the sample is in equilibrium with the surroundings, $b = D\theta\pi^2/4L^2$ where D is the liquid diffusivity, and L the half thickness of the sample. The equation also applies to an initially uniform moisture distribution. If θ is large then $W - W_e/W_o - W_e = 8/\pi^2\ e^{-D\theta\pi^2/4L^2}$ and the drying rate $dW/d\theta = -\pi^2 D(W_e - W)/4L^2$. Thus the drying rate for materials requiring large drying times is proportional to the square of the thickness of sample when diffusion controls.

The falling rate equation for coarse or granular materials in which migration of water between particles is occurring has been shown (Perry, 1950) to give a linear relationship between rate of drying and thickness of product. Thus a relatively easy test can be

applied to determine the broad differences in mechanism. Equations for other geometries are given by Newman (1931), and by Crank (1956) for several geometries, variable diffusion coefficients and evaporation at the surface.

Geometrical arrangement of product

The important factor here is to determine the effect of tray loading —i.e. of the depth of particles in a layer—on the time for drying. The lower the tray loading and the greater distances between the particles the shorter the drying time and the more uniform the product. However to achieve reasonable throughputs in practice it is necessary to load the trays on the conveyor to a reasonable depth. In practice it is usual to attempt to have the same weight of material on each tray and to have a uniform thickness of particles. The nature of the product also has to be considered, since there is always a possibility of damage or adhesion with softer products. With granular materials which are themselves porous it is necessary to consider the restriction to flow of water from the particles by the bulk packing.

The physical properties of the drying environment

The temperature, humidity and velocity of the air being used for drying have a predominant effect on the rate of drying and on the economics of the process. The combined effect of humidity and temperature of air is determined by the psychrometric relationship and is obtained by measuring the wet bulb temperature (adiabatic saturation temperature for water). The wet bulb temperature is a unique function of temperature and humidity. The drying rate has been shown to be proportional to the wet bulb depression for a number of vegetables (Ede & Hales, 1948), although Perry (1944) has shown that when the relative humidity of the air is less than about 40% the rate of drying is independent of wet bulb depression.

The dry bulb temperature of the air has considerable effect on the rate of drying at low moistures but not at high, i.e. below 0·1 moisture content measured as pounds of water per pound of dry product. At these low moisture levels the cooling effect of evaporation is very small and consequently the heat is utilized in the internal redistribution of moisture. The limiting temperatures are determined by the biochemical changes which will cause the development of off-flavours and discoloration.

The effect of air velocity is complex. For individual particles the rate of initial drying period is proportional to $v^{0\cdot7} - v^{0\cdot8}$, where v is the velocity. This corresponds to evaporation from a free water surface but as the drying proceeds the effect is reduced. This assumes that radiation and conduction are absent, since these processes reduce the effect of varying the air velocity. The rate of drying for air flow (a) parallel to the surface of the product is proportional to $G^{0\cdot8}$ and (b) perpendicular to the surface is proportional to $G^{0\cdot27}$ and the constants of proportionality 0·01 and 0·37 respectively,

where G is the mass velocity of the gas in lb total gas/hr/ft^2. These are applicable for the range of G = 500–6000 lb/hr/ft^2 and H = 800–4000 lb/hr/ft^2 respectively.

There are, of course, limitations to the gas velocity which can conveniently be used. The rate of heat transfer is controlled by the thickness of the laminar boundary layer. This decreases with increasing velocity until a point is reached when increasing the velocity further produces no significant increase in heat transfer. Consequently there is a limitation on the velocity of the air used for drying. A second limitation is an economic one, since the power requirements for blowers are proportional to the cube of the velocity. Thus there is little incentive to use velocities which are higher than necessary.

By increasing the velocity however the average humidity in the drying area is substantially reduced and consequently there is a higher moisture gradient between the product and the drying atmosphere which accelerates the drying operation.

Characteristics of drying equipment

In the previous sections it has been assumed that the heat transfer was entirely by forced convection. However there are other mechanisms for heat transfer—i.e. conduction and radiation—which contribute significantly in other types of drying equipment. The overall heat transfer coefficients H for some other types of equipment are given in Table 1.

TABLE 1. Overall heat transfer coefficients for various types of drying equipment (Williams-Gardner, 1971)

Type of Drier	H	
Vacuum shelf	1	Btu/hr ft^2°F
Agitated tray	5–60	,,
Rotary vacuum	5–50	,,
Indirect rotary	2–10	,,
Jacketed trough	2–15	,,
Drum	200–300	,,

Radiation drying using infra-red radiation has been extensively discussed by Ginzburg (1969). With this technique high thermal gradients can be established and rapid uniform drying. It is necessary to have a detailed knowledge of the optical properties of raw materials in order to select the most appropriate wavelength. Radiation has, of course, been used extensively with freeze drying systems where greater thermal efficiency can be achieved.

In conclusion, it is necessary to consider each type of equipment in order to assess its suitability for a given drying operation. In particular a knowledge of the types of

heat transfer occurring will assist in producing a material of uniform moisture content.

In the following sections only a few types of drier are discussed; detailed information on the design, operation and economics are given by Perry (1950), Treybal (1968), Foust *et al.* (1960), Brennan *et al.* (1969), Webb (1964), Slade (1967) and Hall (1971).

Properties of food products

The physico-chemical properties of food products will be dealt with briefly in this section as they are particularly important in determining the overall organoleptic properties. The most important aspects are (i) structure and composition of raw materials, (ii) shrinkage during drying, (iii) loss of volatile components, (iv) browning reactions, and (v) moisture absorption and rehydration.

(i) *Structure and composition.* It is not possible to discuss this very complex subject in any great detail. The most important topics are the nature of bound water in foodstuffs and the type of structure through which the moisture migrates towards the drying surface. Kuprianoff (1958) has discussed the nature and binding of water in foodstuffs and the techniques for determining the 'free' and 'bound' moisture. The drying curve (Fig. 1) shows to some extent the state of the moisture since the constant rate drying period is attributed to free moisture and the falling rate to bound moisture. Where the latter is broken then there is a transition from one type of binding to another. The low moisture region can also be studied by the moisture isotherm techniques (see later). The most important information on the structure of raw materials in relation to dehydration has been given by Gane & Wager (1958) on plant produce and also by Brooks (1958); other information is contained in specialized textbooks as well as food science books.

(ii) *Shrinkage.* It is well known that except in freeze drying there are considerable structural changes which accompany dehydration (Gorling, 1958; Duckworth, 1962; Duckworth & Smith, 1963). The main mechanism operating is the diffusion of solutes which tend to be deposited in the outer layer of the product. Structural damage also occurs due to the stresses developed during shrinkage. The result of this action is to retard and limit the complete rehydration of dried products.

(iii) *Loss of volatile components.* The study of loss of volatiles during drying has been extremely limited. The relative volatility of the components governs the extent of volatile losses. The most important work on this subject has been presented by Menting, Hoogstad & Thijssen (1970), and Rulkens & Thijssen (1969).

(iv) *Browning reactions.* The chemistry of these reactions is quite complex and several different phenomena, including enzymic and non-enzymic reactions, are responsible for this type of behaviour. The browning can occur during dehydration due to excessive heating, or alternatively during storage due to oxidation and enzyme reactions. The extensive literature has been reviewed and summarized by numerous workers, Hodge (1953), Lea (1958), Ellis (1959) and Reynolds (1963, 1965, 1970).

A more extensive account is given in the section on freeze drying. Sulphiting is commonly used to prevent browning reactions (Van Arsdel, 1963).

(v) *Moisture absorption and rehydration.* The study of moisture absorption in dried products is very important for determining storage behaviour and shelf life. It is necessary to obtain information on the critical moisture level required to promote microbiological growth (Christian, 1963) or enzyme reactions (Acker, 1963 a, b) leading to deterioration. The literature on this topic is very extensive, although the interpretation of the data is often difficult—cf. Bate-Smith, Lea & Sharp (1943), Makower & Dehority (1943), Gane (1950), Gorling (1958), Stitt (1958), Salwin (1959), Taylor (1961), Charie, East & Vanderveen (1965), Labuza (1968) and Caurie (1970, 1971). The sorption isotherms are generally sigmoid and show a marked hysteresis.

Tunnel dehydration

It is only possible here to deal with some of the types of equipment which have been used for hot air dehydration, since each product in the particular shape and size specified requires its own individual conditions for drying. This means, as far as the manufacturer is concerned, that special care is required in the initial specification if the desired output and quality are to be achieved. The main reason for this is that the drying characteristics of foods vary enormously (Tucker, 1968) as well as the initial moisture contents.

It has already been mentioned that size, shape and structure affect the rate of drying of a product under the specified conditions. However, it is equally important that the moisture be removed from the product in the most suitable way to impart an acceptable quality as well as achieving this in the most economic way. Thus, in the design of air drying systems particular attention must be paid to the method of heat transfer and also the removal of moisture.

The tunnel dehydrator is by far one of the most flexible systems which is in commercial use. In its simplest form it consists of a rectangular tunnel which will accommodate trucks containing the trays on which the product to be dried is uniformly spread. The dimensions of these systems vary greatly, depending on the capacity of the plant, but a typical plant for dehydrating carrots at a rate of 75 tons per day would consist of four single-stage tunnels about 54 ft in length, $6\frac{1}{4}$ ft wide and 7 ft high and this would contain 14 trucks. The air would be directed in a counterflow direction and would reach a temperature of 160°F (approx.) 90°F wet bulb at the unloading end. The air velocities vary in practice from 600 to 1200 linear ft/min.

The tunnel system is essentially a continuous one in which the trucks gradually enter one by one and are similarly removed at a constant rate when equilibrium conditions are established.

Tunnel driers are usually classified by the direction in which the air traverses the product and several arrangements will now be mentioned.

Single stage

(i) *Pcrallel flow.* The 'wet' material encounters initially the driest and warmest air and leaves the dryer at the coolest end. This means that high rates of evaporation are achieved initially and there is little danger of overheating the product, since the surface temperature of the food is below the dry bulb temperature. This results from the fact that during the process of moisture loss heat is removed by the evaporation process. The product is also protected in the latter stages of drying since it comes into contact with progressively cooler air and this is accompanied by a decrease in the drying rate. However this has the ultimate disadvantage that very low moisture contents cannot be achieved irrespective of length of tunnel. Commercial plants based on single stage parallel flow are operated mainly for drying grapes; however, they are usually used in combination with counterflow principles.

(ii) *Countercurrent flow.* The alternative method of circulating the air is to direct it in the opposite direction to the movement of the product so that the dry product leaving the drier encounters the hot and dry air entering the system. Thus the 'wet' product is initially exposed to very slow drying conditions. However, provided precautions are taken not to overheat the product, it has ultimately a lower moisture content. Countercurrent processes are generally recognized to be more economical to operate and they are widely used for commercial production, especially with dried vegetables. A particularly important problem involves the shrinkage of the product using countercurrent rather than parallel flow. The result is that the former gives a material of relatively high density and slow reconstitution properties whereas the latter gives a product with much less shrinkage and lower density.

A typical root vegetable dehydration plant operating a countercurrent drying tunnel would have a capacity of about 0·5 ton/hr, involve 12 trucks, air temperatures 160°F w.b., 90°F d.b., and air flow 600–700 lin. ft/min. The total drying time required would be of the order of 8 hr.

Double stage

Several methods of operating double stage plants are known and, in particular, the two stages may be arranged as separate tunnels or, alternatively, together in the same tunnel.

(i) *Countercurrent/parallel system.* In this system the product first encounters air flowing in a countercurrent direction and then in a parallel direction, the air being fed in at a central point.

(ii) *Parallel/counter current flow system.* The bulk of two stage plants in commercial operation are of this type so that advantage may be taken initially of the high rate of evaporation available in parallel flow. The advantages compared with single stage units are more uniform drying, increased output from starter drying time and good overall quality.

It is often found in practice that the first stage is shorter in length than the second to compensate for low drying rate in the latter. However, equal length tunnels are widely used and it is possible by placing them side by side to arrange for loading and unloading to be carried out at the same end. In many systems the air from the secondary tunnel is used to supply the primary parallel flow system.

This system enables the temperature of the air in either system to be adjusted to desired values; it is usual to have the air of higher temperature in the parallel flow system and air of a lower temperature in the countercurrent. It is also important that maximum recirculation be carried out to economize on the heating load.

Multistage driers

The chief advantages of three stage driers are that they are more flexible and can achieve under nearly optimum conditions the drying of a wide range of products. However relatively few are in operation and those which are operate all three with countercurrent flow of air.

Three and five stage driers are also known to be in use with interstage reheating, the direction of flow being referred to as either cross or transverse flow (Kilpatrick, Lowe & Van Arsdel, 1955). Gooding & Tucker (1958) discuss the design and operation of a three stage crossflow plant with reheating at various stages.

A four stage system (involving cross flow) is also described by Gooding & Tucker (1958) in which the first two stages are short in length, the third longer and the fourth stage as long as the first three together. The first three are interconnected in the cross flow direction with interstage reheating already mentioned and the final stage may be of the same type or alternatively a through flow type (i.e. flow through the bed of particles). The first stage in this system can operate at temperatures as high as 230°F.

Kilpatrick *et al.* (1955) describe some other systems which have been tried, together with details of some commercial plants being used for fruits and vegetables. Of these only one is of some importance for the future and that is the total recirculation system which has been proposed as a solution to the odour problem with onion and garlic dehydrators. This involves interstage heat transfer with dehumidification.

Bin-type finishing driers

Bin-type finishing driers (Anon., 1944) are widely used for the equilibration and storage prior to packaging of dried products and also for obtaining final moisture levels. Basically they are cylindrical containers through which warm air is blown at a velocity not greater than 100 $ft^3/min/ft^2$. These bins provide a low cost method of removing moisture from particular products during the slowest stages of drying. Both static and portable units are available. The drying time required to reduce the moisture content of vegetables from 10% to 5% is 24–36 hr. For onion dehydration, for which a

final moisture content not in excess of 4% is required, an air temperature of 120°F, absolute humidity above 0·003 lb/lb dry air and an air velocity between 80 and 100 $ft^3/min/ft^2$ of bin cross section were required (Van Arsdel & Copley, 1964).

Spaugh (1948) carried out a series of experiments on dehydrated vegetables to determine the pressure drop when air was forced through these packed beds. The range of air velocities used was between 35 and 170 ft/min (ft^3/min per sq ft of bin cross sectional area). Since the data did not fall on a straight line, the results were correlated using a power law relationship (equation (2)).

$$\frac{\Delta P}{L} = C\rho U^n \tag{2}$$

where $\Delta P/L$ is the pressure drop per unit length,

C is a constant,

ρ is the density of air,

U is the velocity of air,

n is an exponent.

This equation was also modified to take into account the dimensions of the bin, i.e. the 'wall effect'. See equation (2). For large bins greater than 4 ft square-sided the ratio L/A (feet of perimeter per square foot of bin cross-section) is 1 or less, then equation (3) applies.

$$\frac{\Delta P}{L} = \rho \left(\frac{C_1 U}{C_2 + L/A}\right)^n \tag{3}$$

The values of n and C from equation (3) for several vegetables are given by Spaugh (1948).

For flow through packed beds a large amount of data have been tabulated (Carman, 1956; Holdsworth, 1963). It is generally recognized that for Reynolds numbers below about five the flow regime is laminar, whereas above Re = 200 it is fully turbulent. Between these values the flow is transitional and intermediate equations would apply. See equation (4).

$$\frac{\Delta P}{L} = au + b\rho u^2 \tag{4}$$

where au is the resistance due to laminar flow,

and $b\rho u^2$ is the resistance due to turbulent flow.

Thus it would appear that the values of n obtained by Spaugh (1948) indicate that the flow is transitional in most cases. However the values of the coefficients seem to call

for some further comment, since those for particles of identical shape differ by a factor of two. The pressure drop depends upon the size, shape and method of packing particles and consequently it can only be assumed that the method of packing may have been significantly different. Alternatively the results may represent the degree of accuracy that can be associated with this equation. There would appear to be no theoretical reason to include the constants in the power term in equation (3).

Conveyor type driers

This type of drying system is very similar to the tunnel system except that the material is conveyed through the hot air system on a continuous moving belt. The system has the advantage that the high cost of handling products both before and after drying using trays is substantially reduced. The wet material is loaded uniformly (6–8 in. deep) onto the belt (either a woven metal mesh or interlocking perforated plates) and dried initially by air blowing through the bed and finally by air passing down through the bed. The latter prevents material being lost from the system due to changes in density during drying. The belts vary in length from 30 to 60 ft and in width from 6 to 100 ft. The process conditions are primarily controlled by sectionalizing the system, thus it is possible to control independently flow rates, humidities and temperatures, and thereby give optimum output and quality. Air recirculation is also important in the initial stages. This type of drier is essentially for high capacity water removal and it is necessary to remove the material at a moisture level of about 15% to avoid the use of excessively long belts and waste of evaporative capacity.

A typical drying operation would involve 35 tons/hr raw material infeed onto a 75 ft long 8 ft wide conveyor belt loaded at a rate of 10 lb/hr and allowing 2½ hr for travel through the system.

Agitated and fluidized bed drying

The distinction between agitated bed and fluidized bed driers is a narrow one, since in each case an upward velocity of air sufficient to support the food particles is applied. In the case of the former the bed is less vigorously stirred than in the latter. It is necessary with driers of this type to decrease the air velocity as the particle dries in order to prevent the particles being lost from the system. The final drying is accomplished using bins.

The conditions for fluidizing particles of large size are outlined elsewhere (Holdsworth, 1970). Using the Froude number (V^2/dg) as a characterizing parameter the conditions for stable fluidization of food particles (size 3–20 mm) correspond to a range of Reynolds numbers ($Vd\rho/\mu$) from 500 to 10,000. Velocities of air vary from 3 m/sec to 6 m/sec. The data quoted here are for fluidized freezing of food particles but

can be used if consideration is given to the change in density of the product and air. Unfortunately very little information is available on heat transfer coefficients and also mass transfer coefficients. Scott, Tape & Aref (1967) have described a general purpose pilot plant for fluidized treatment of food products including drying, dehydrofreezing and freezing operations. The minimum velocity to produce fluidization was found to be 375 ft/min (19 m/sec) for $\frac{3}{8}$ in. diced apple and potato. The hot air temperature sequences were 30 min at 212°, 194° and 176°F (three separate experimental trials) followed by one hour at 158°F and finally 3–3$\frac{1}{2}$ hr at 140°F for apple. However the initial temperature had a negligible effect on the drying rate or overall length of time for drying. The drying curves for the two products were entirely different; the apple dried rapidly to 10% moisture in 1$\frac{1}{2}$ hr followed by very slow drying (i.e. a very sharp critical point), while the potato showed a continuous smooth drying curve, the 10% moisture point not being reached for over 4 hr. A further extension of the fluidized bed technique has been described by Farkas, Lazar & Butterworth (1969) and Lazar & Farkas (1971). A pilot was designed which employed a centrifugal force to allow the velocity of fluidization, and hence the heat transfer, to be improved. Using $\frac{3}{8}$ in. diced carrot it was found that the pressure drop increased linearly with increasing centrifugal force. Heat transfer coefficients and rates of drying have not yet been reported for food products.

The design of fluid bed drying systems has been dealt with by Vaněček, Markvart & Drbohlav (1966) and their book gives the most recent account of the subject. Developments in the use of fluidized beds for drying foodstuffs have been given in general terms by Tape (1970), Scott *et al.* (1967) and Anon. (1970b).

An interesting point with fluidized bed processing is the variation in the residence time of the food particles. Kelly (1965) and de Groot (1967) have studied this by using a known number of dyed peas and shown that a typical sigmoid curve is obtained indicating a wide spread of residence times. In this respect the fluidized bed technique differs from all other types since the particles are mobile.

An extension of the principle of the agitated bed is incorporated in the belt trough drier (Lowe *et al.*, 1955) in which air is passed through a moving bed of particles. This drier consists of an endless wire mesh belt arranged in such a way as to form an inclined trough such that the product travels in a spiral path. Partial fluidization is produced by an upward blast of air usually of the order of 400 ft/min and up to 300°F. Experimental work on diced carrot ($\frac{1}{4}$in. × $\frac{1}{4}$in. × $\frac{1}{8}$in.) indicated that about 20 min was required to remove half the initial water.

The fluidized bed principle represents an important advance and can be used for a wide range of particulate materials. Apart from the commercial drying of peas, beans and diced vegetables it is also used for drying potato granules (Neil *et al.*, 1954; Griffiths *et al.*, 1955), onion flakes (Van Gelder, 1962) and fruit juices (Mink & Nack, 1966; Dryden, Mink & Nack, 1966).

Explosion puffing

The demand for better quality and faster reconstituted dehydrated particles has led to the development of explosion puffing. This process was developed by the U.S. Department of Agriculture at the Eastern Utilization Research and Development Division near Philadelphia and it is now recognized as one of the most significant developments in dehydration technology. The products have many of the desirable attributes of freeze dried products but can be processed at a significantly reduced cost which is comparable with the cost of conventionally air dried samples. The well-known methods of hot air drying are slow, the time for drying being governed by the size of particle being dried. The products likewise require considerable times to rehydrate, for example $\frac{3}{8}$ in. cubes of vegetable require up to 1 hr in boiling water to reconstitute adequately. Cording *et al.* (1963) quotes 20–25 min for potatoes, 35–45 min for carrots and 45–55 min for beets. Explosion puffing, however, reduces the time for rehydration to a few minutes as well as for reconstitution of the products.

Process details

In explosion puffing, partially dehydrated pieces from a preliminary stage drying are heated in a closed rotating cylindrical container known as a 'gun' until the internal pressure has reached a predetermined value. When this point has been reached the gun is discharged instantly to atmospheric pressure (Cording & Eskew, 1962; Eskew, Cording & Sullivan, 1963). During this process a certain amount of water is vaporized but, more important, the explosive or flashing conditions cause a highly porous network of capillaries to be developed within the particles. This porosity enables the final dehydration to be achieved much more rapidly (approximately twice) than would have been the case with conventionally dried products. It also bestows on the product the ability to reconstitute extremely rapidly.

The gun described above was externally heated but it was subsequently redesigned (Heiland & Eskew, 1965). The heating cycle in the modified process was shortened by introducing superheated steam 500°F at 55 psig into the gun after the charge of partially dehydrated particles had been prewarmed by tumbling in the preheated (gas burners) gun. The latter procedure prevents steam condensation and also ensures that the particles are exposed to heat on all surfaces rather than the less efficient and more random contact with the heated walls alone. The modified gun was constructed of thin wall stainless steel being 10 in. in diameter and 30 in. long. The interior of the gun was furnished with integral fins to increase the area for heat transfer. A particularly important feature in the design is the rapid release mechanism for the lid together with a low inertia arrester for smooth absorption of lid impact. The gun is supported in a horizontal position by a swivel pivot and a mechanism for tilting into either filling or discharging position is incorporated. The capacity of the machine is quoted as being

nine 20 lb charges per hour of $\frac{3}{8}$ in. vegetable dice containing 22% moisture which is approximately $1\frac{1}{2}$ ton/day of finished product.

(i) *Carrots.* Cording *et al.* (1963) and Eskew *et al.* (1965) describe the complete process for producing dehydrated carrots. The preliminary drying was carried out using a continuous belt hot air drier which gave a product with a moisture content of 30–42% using air at 200°F (dry bulb temperature) and a velocity of 200 ft/min.

The explosive puffing operation was carried out next under optimum conditions, which in the case of $\frac{3}{8}$ in. carrot dice were 35% moisture and with 35 psig pressure developed. The final moisture content of the product was about 30% moisture. If the pressure is too high then low moisture products tend to be scorched and high moisture content particles disintegrate. On the other hand if the pressure is too low then the pieces fail to puff. The pressure–moisture relationship is given in detail in the paper by Cording *et al.* (1963).

The final dehydration stage was then carried out using a single stage, continuous belt hot air drier (150°F and 200 ft/min) in which the moisture content was reduced from 30 to 4%. From this pilot plant work it was considered that commercial bin drying would not be necessary.

The work was extended to cover the drying of explosive puff particles of different moisture contents 45·4%, 41·4% and 37·8%, from which it was found that the lowest moisture content particles required the least time for the final drying cycle, i.e. 110–120 min. The corresponding drying rates (at a moisture content of 0·4 lb water/lb material) were 3·8, 4·5 and 6·0 lb water/ft² drying area/hr compared with 3·4 lb/ft²/hr for non-puffed carrot dice. Due to an alteration in mechanism the slope of the drying rate curve changes at moisture content 0·12 lb/lb. At the higher moisture level the controlling mechanism is the diffusion through the pores whereas at the lower level the removal of moisture from the tissue controls the rate. Hence the role of porosity in determining the rate of dehydration decreases as the moisture content is reduced.

Comparative results on reconstitution of 'instantized' and conventional carrot dice $\frac{3}{8}$ in. × $\frac{1}{2}$ in. × $\frac{1}{4}$ in. in boiling water were 7 min and 30 min respectively for a coefficient of rehydration of 60%. (Eisenhardt *et al.*, 1962).

The product was recommended to be nitrogen packed at a moisture content not in excess of 4% and that it should contain 500–1000 ppm sulphur dioxide. No data on the storage properties were given in the original paper.

Further work on vegetable dehydration (Heiland & Eskew, 1965; Eskew & Gelber, 1964), using superheated steam as described, has been reported.

Dellamonica & McDowell (1965) have studied the β-carotene content of dehydrated carrots and found that there is little difference in the total content of the puff dried product compared with conventional and freeze dried products. However after cooking the puff dried sample was superior to the air dried sample but not as good as the freeze dried sample in relation to total β-carotene as well as the trans-isomer, provitamin A.

Sinnamon, Eskew & Cording (1965) have studied the manufacture of high density explosion puffed carrot dice using compression techniques. This was necessary to attempt to overcome the problem of the bulk volume of explosion puffed material which is greater than the conventional air dried product. The density of $\frac{3}{8}$ in. carrot dice explosion puffed is 21 lb/ft^3 whereas the corresponding density for conventionally dried material is 26 lb/ft^3. The technique used was to compress the product after explosion puffing by passing the dice through the rolls of an unheated double drum drier with the roll clearances preset at prescribed values. The experimental work showed that compression of the product resulted in a 25% increase in the drying time for the final stage but had little or no effect on the excellent redehydration rate of the product.

Turkot *et al.* (1965) have presented detailed costs of the total operation.

(ii) *Beets.* The procedure for beet dice $\frac{3}{8}$ in. (Cording *et al.*, 1963; Eskew *et al.*, 1965; Sullivan *et al.*, 1965) was similar to that for carrots, and the optimum conditions for explosion puffing were 45% moisture in the particles and 45 psi pressure development.

(iii) *Potatoes.* Cording, Sullivan & Eskew (1964) and Eisenhardt *et al.* (1962) have examined the problems associated with both white and sweet potatoes for the production of a rapidly reconstituted dehydrated product. Pre-treatment is very important for potato products and acceptable products were obtained by pre-cooking high solids potatoes for 15 min at 160°F and subsequently cooking in water at 50°F for 15 min. Optimum puffing conditions were found to lie between 24 and 53% moisture in the pre-dried material. Above 53% total disintegration occurred and below 24% a scorched product with poor reconstitution properties was obtained. The pressure required in the explosion puffing varied from 30 to 60 psi depending on moisture. The position with regard to optimum conditions is not as clearly defined as with carrots. Further work has been carried out by Cording *et al.* (1964), Sullivan, Cording & Eskew (1963) and Sullivan & Eskew (1964).

Turkot *et al.* (1967) have examined the economics of the complete process located as an adjunct to a factory already involved in making potato products.

(iv) *Celery.* Wilson (1965) and Sullivan & Cording (1969) have described the conditions for producing a rapidly reconstituting puffed celery which can be used in soup mixes. The initial air drying stage was continued for 3 hr (175°F dry bulb 110°F wet bulb) until the moisture content was 35–40%. The product from this stage requires a period of equilibration at 38°F in plastic bags for 24 hr prior to explosive puffing using superheated steam. The final drying process was accomplished in $2\frac{3}{4}$ hr using hot air 130°F dry bulb and 95–100°F wet bulb at 200 ft/min air velocity.

(v) *Fruits.* Explosive puffing has also been successfully applied to fruits, in particular apples (Eisenhardt, Eskew & Cording, 1964; Eisenhardt *et al.*, 1968) and blueberries (Eisenhardt *et al.* 1964, 1967). The process for apples involves preparation of the slices, sulphiting, initial drying to 20–30% moisture ($3\frac{1}{2}$ hr with air 300 ft/min 180°F dry bulb and 100°F wet bulb), equilibration at 38°F for 24 hr, explosive puffing to 25 psi, 33 rpm rotation (i.e. 40% of critical centrifuging speed), and final drying from

20% to 2% moisture using air 350 ft/min 150°F dry bulb and 100°F wet bulb temperature for 5 hr. The time taken is less than half that required for the unpuffed product.

The processing of blueberries is somewhat easier because of the small amount of preparation required. After washing and size grading the berries are air dried to a moisture level of 22% using air 300 ft/min at a temperature of 200°F dry bulb and 100°F wet bulb. Explosion puffing was found to be satisfactory for a range of moisture contents 19–30% using a pressure of 20 psi and the final drying carried out to 6% moisture level.

Both products could be rapidly reconstituted. With apples the porosity characteristics were determined and comparative figures, taken from Eisenhardt *et al.* (1968), are shown in Table 2.

TABLE 2

Property	Freeze dried	Explosion puffed	Conventional air dried
Surface connected pore volume (% total volume)	88·2	61·5	10·7
Rehydration ratio 6 min simmer (g Wet/g Dry)	5·61	4·44	3·23

From these figures it can be seen that the high value for the surface connected pores is directly related to the ease with which those products are reconstituted compared with the low value for the conventional air-dried product.

Consumer preference tests have been reported for blueberries processed by a variety of techniques including explosive puffing (Isidro *et al.*, 1968).

Apple slices produced by explosive puffing tend to be hygroscopic and lose their moisture under humid conditions. Strolle *et al.* (1970) have recommended that the sucrose content be increased from the normal 20–30% to 80% prior to initial drying by soaking in water for 3·6 hr to leach out the monosaccharide content, and then soaked in a 20–24% sucrose solution for several hours until the desired level of sucrose is imbibed. The treatment is based on the fact that sucrose has the highest softening point of the carbohydrates and consequently materials with a high level of sucrose will lose crispness more slowly than those with lower levels of sucrose or other carbohydrates when exposed to moisture. It was found that the treated samples retained their crispness to a 6·6% moisture level whereas for untreated samples the level of moisture was 4·3%. Wisenhardt *et al.* (1969) have found that dehydrated apple sauce stored under nitrogen for 9 months at 73°F and 6 months at 100°F was still acceptable.

Foam drying processes

The dehydration of liquid food products has progressed rapidly during the last two decades with the development of foam drying processes.

Vacuum puff drying

The development of processes for drying liquids under vacuum came from the observations made on the freeze drying of orange juice concentrate. Schroeder & Cotton (1948) and Schwarz (1948) found that the rate of drying of that product in the liquid state was double that in the frozen state (freeze drying) and the dried product had a highly porous structure which exhibited good rehydration properties. This was due to the fact that under vacuum conditions the liquid tends to foam and produce a film structure which dries to give a highly porous solid. The drying characteristics of a two-phase system air+water vapour/liquid differ appreciably from those of a single phase liquid. The result of foaming is, of course, to impart a more rigid structure to the product and to increase the surface area for moisture diffusion. With regard to heat transfer, however, such a system is initially less efficient since foams have an inherent low thermal conductivity. This is subsequently offset by the fact that the latter stages are mass transfer controlled. However, when all aspects are considered, foam drying is a relatively rapid process which does not require high temperatures. (Talburt, 1961; Schroeder & Schwarz, 1949; Strashun & Talburt, 1953; Hertzendorf & Moshy, 1970). With regard to product quality the foamed structure is particularly useful because it confers the property of rapid reconstitution. However, like all food products with high surface areas, these products present a storage problem in relation to moisture uptake and oxidation. Aroma retention also presents a problem, and add-back techniques are required with some processes, e.g. orange oil on sorbitol is added to the dry powder (Strashun & Talburt, 1958). Corn syrups were also found to be important in preventing undue hygroscopicity of the product. Batch drying of fruit juices can be achieved using a foaming technique incorporating a conventional vacuum tray drier with a vacuum system, e.g. three stage steam ejector capable of producing a pressure below 10 mm Hg in 3–4 min. The concentrate is fed onto the trays in a 1/16 in. thick film equivalent to $\frac{1}{2}$ lb/ft^2 loading and this increases under vacuum to a thickness of 1–2 in. The initial temperature of the trays is between 200–275°F depending on the product and this is reduced progressively to prevent deterioration of quality. Drying chamber pressures which are found to be most suitable are in the range of 1–4 mm Hg and it is essential to cool the product below 90°F prior to breaking the vacuum. The productivity of the plant was found to increase with increasing tray loading. Products from this type of operation have a moisture content of about 3% which can be reduced to suitable levels for storage (i.e. $\frac{1}{2}$–1%) using in-packet desiccation.

The process used in the drying of tomato paste involved stabilization with egg al-

bumen and the production by whipping of a foam of density 0·4 g/ml. This was then spread on the drying belt of teflon coated fibreglass in a layer $\frac{1}{8}$ in. thick and dried with air at a temperature of 160°F initially and then 120°F for the final stage. The product obtained after 65 min of drying had a moisture content of 2·5% and a density of 0·2 g/ml. The latter was increased to 0·6 g/ml by hydraulic compression at 1000 psi. Other products, such as milk, orange juice and coffee required the use of fatty acid monoglycerides and mixtures of mono and diglycerides and fatty acid esters of sucrose. Morgan *et al.* (1961b) discussed two modifications to the original process; these were:

(a) The 'Crater' process in which a layer of foam about $\frac{1}{8}$ in. thick was deposited on aluminium trays containing $\frac{1}{8}$ in. diameter holes (Mortan *et al.*, 1961b; Ginnette, Graham & Morgan, 1961). A blast of air was then directed through the holes in such a way that craters were produced in the foam sheet. By this means a larger foam surface area was exposed to the drying air stream and consequently the limitation of restricted mass transfer (Komanowsky, Sinnamon & Aceto, 1964) was improved. The process also makes more effective use of the heated air and permits increased tray loading. Low moisture contents were obtained, i.e. 2–4% using concurrent blasts of air at 200°F on the wettest material and the final drying accomplished with countercurrent low velocity air, 2 ft/sec at a temperature of 130°F and relative humidity 5%. The principle has been put into commercial practice using a plant with 200 lb/hr production capacity and 700 lb/hr evaporation capacity (Sjorgren, 1962; Rockwell *et al.*, 1962; Eolkin, 1962; Lawler, 1962;

(b) The 'Spaghetti' process in which strips of foam were extruded onto the plate thereby increasing the surface area for mass transfer; the highest throughput was obtained when threads of the thinnest diameter were dried at the highest possible temperature. A milk concentrate (40–46% solids) was foamed to a density of 0·5 g/ml, and extruded followed by drying with air 112°–232°F, velocities of 240–710 ft/min and humidities 1–24%. Komanowsky *et al.* (1964) studied the mechanism of the drying by following the change in moisture content with time and found that there was no constant rate drying period but only a uniform falling rate period indicating that diffusion was the main controlling factor. This was further confirmed by studying the effect of thickness of foam on the drying rate, where it was observed that the ratio of the effective diffusivity to the square of the thickness of the foam correlated well with specific moisture content.

With either process the product has a very low density and it is necessary to increase the density by passing the powder through double roll compaction units with heated rolls. This process not only facilitates handling but also removes very fine bubbles which tend to be incorporated in the reconstituted liquid. (Wagner, Bissett & Berry, 1964; Graham, Ginnette & Morgan, 1963; Graham, *et al.*, 1965; Berry *et al.*, 1967). With citrus products Berry, Bissett & Wagner (1965) and Berry *et al.* (1968) found that it was convenient to store these under vacuum for 3 days and then to admit carbon dioxide afterwards. Except for grape and apple juice all the other liquids foamed satisfactorily

and, in order to facilitate foaming these, it was necessary to add sucrose. Notter, Brekke & Taylor (1959) also studied other compounds, including sugars, cellulose and pectins as additives but the most effective additive was found to be sucrose.

The vacuum puff process has also been developed as a continuous drying operation. The type of equipment used for this consists essentially of a belt up to 60 ft long driven by drum rolls which can be heated or cooled, contained in a vacuum shell.

Foam mat drying

This process is a development of the former but, instead of employing a vacuum to foam the material, it is initially foamed by suitable agents and then subjected to drying under atmospheric pressure. The equipment required for this is similar to the continuous band drier described previously, without the vacuum facilities.

The success of the process is dependent upon producing foam of suitable structure. The most desirable characteristics are that it should consist of a large number of small uniform stable bubbles and should retain its structure during drying. In order to facilitate stability it is often necessary to use a film forming additive, such as solubilized soy protein, glycerol monostearate, propylene glycol monostearate or sucrose palmitate. The density of foams varies from 0·1 g/ml to about 0·8 g/ml with a preference for the more dense. Pseudoplastic and compressible behaviour are observed in varying degrees with foams and this affects their flow properties, however it is possible to handle them with ease either under shear or pressure conditions. The mechanism of the drying process has been studied by Morgan *et al.* (1959, 1961b) who considered that the drying rate was governed by heat transfer to the foam in the early stages coupled with a rate determining mass transfer operation. It was also shown in connection with the rate of evaporation that this was only 30% of that expected from a free water surface. Although the initial work was based on tomato paste, a range of products were examined including milk products, coffee, pineapple, lemonade, grape and orange juice, apricot puree and a prune product containing egg albumen.

The success of foam mat drying depends upon the production of a stable, uniform foam. Although some foods—e.g. egg white, beef extract concentrate and whole milk concentrate—foam easily and do not require additives, the bulk of food products require some form of stabilizing agent (Hart *et al.*, 1963; Morgan *et al.*, 1961b; Sjorgren, 1962; Bates, 1964; La Belle, 1966; Gunther, 1964a, 1964b).

Microflake ᵀ dehydration

This technique involves the drying of a continuous sheet of foam 20 ml thick on a continuous stainless steel belt (Anon., 1964). The latter is heated from below by steam and above by a high velocity air stream and drying times are reported to be about one tenth of the standard processes. The temperature required is of the order of 170°F and

care has to be taken to produce a very uniform film since the rate of dehydration is proportional to the square of the thickness. The rate controlling factor is the internal diffusion of moisture. Osborne *et al.* (1966) describe a commercial system using a 4 ft wide stainless steel belt 200 ft long which moved at the rate of 180 ft/min. Heat and mass transfer process operating in this type of system have been discussed by Moshy (1967). It was found that doubling the air velocity from 1000 ft/min to 2000 ft/min increased the average drying rate by 75%. This causes a decrease in the thickness of the laminar sub-layer of the boundary layer and the mass transfer of water vapour from the surface of the film increases with resultant decrease in drying time. Heat is transferred by conduction from the belt to the product and the rate controlling factor is the transfer of heat through the layer especially when the moisture content is low.

A commercial 3,000,000 lb a year drying unit using Microflake ^{T} process has been operated in Florida (Anon., 1968). The drying chamber is composed of ten sections each 12 ft long and this is followed by a 15 ft section for cooling the product. The temperature did not exceed 170°F and a drying time of 1 min produced a powder of 1·5% moisture content.

Foam spray drying

This is an extension of spray drying and involves the use of gases dissolved under pressure prior to spraying (Hanrahan & Webb, 1961). The main advantage is that the density of the product is reduced by half and, whereas spray dried particles are hollow spheres surrounded by thick walls of dried material, the foam process produces particles having many internal spaces and relatively thin walls. The pressures which have been used for this process are considerable—e.g. 4025 psig carbon dioxide for tea and coffee and 2000 psig air for acid-whey.

Storage stability of products

Foam dried products, because of their porous structure, present problems in relation to storage stability. Oxygen and moisture produce undesirable reactions which promote deterioration and off-flavour. The products are also very hygroscopic and require in-packet desiccation, (Morgan, Graham & Ginnette, 1962; Talburt, Hendel & Legault, 1954). Silica gel, and alumina are very suitable; calcined lime, on the other hand, packs down and has a high expansion when moisture is absorbed. Mylne & Seamens (1954) have studied the storage properties of orange juice powder and found that it was still acceptable after six months at 100°F and one year at 70°F. The dry powder was mixed with sorbitol stabilized oil and sulphite under vacuum.

Spray drying

This technique, involving the fine dispersion of droplets in a hot gas, has been widely used for liquid food products and has received much experimental study. The process

has considerable advantages over batch and roller drying methods since the high surface area obtained by a fine dispersion of droplets facilitates low drying times when coupled with high heat transfer rates. The temperature of the droplets remains below the wet bulb temperature of the drying gas until almost all the water has been removed, due to the high evaporation rate, and consequently high temperatures can be utilized. The particle shape produced is unique, being spherical but hollow, the solids being concentrated in the wall.

The design of spray driers has advanced rapidly during the last twenty years, the main aim having been to achieve high overall thermal efficiencies with high throughput. This has involved careful consideration of the dynamics of atomization together with methods of bringing the hot gas and atomized particle into contact. The essential stages of any spray drier are (a) an atomizing system, (b) a hot gas producing unit, (c) a chamber to contact the sprayed particles with hot gas, and (d) a recovery system. Seltzer & Settlemeyer (1949) classify spray driers as follows, according to the nature of the air flow:

1. Horizontal co-current driers;
2. Vertical co-current driers (i) upward and downward, and
   (ii) simple and complex;
3. Vertical countercurrent driers.

The importance of the correct atomizing system can be seen when the rate-controlling stages are considered—namely, heat transfer to the droplets, mass transfer by vapour diffusion from the material surface to the surrounding gas and diffusion of residual moisture through the dried layer of product. The main problems with the spraying operation are that the distribution of particle size is uneven, interaction and coalescence of droplets occurs and gas dynamics are complex.

However many designs of atomizer nozzle have been developed (Dombrowski & Munday, 1968) to attempt to overcome these problems. The first type consists of those in which pressure is used to develop a droplet system and these include fan spray, impact, swire and divergent pintle nozzles. The second class of atomizers involve the use of centrifugal energy and include spinning discs of various geometries (operating speeds between 100–600 ft/sec). The third class utilize gaseous energy with twin fluid or blast atomizers using fans, rotary blowers and reciprocating compressors. It is generally recognized that the method of forming droplets depends upon the production of a very thick sheet of liquid which then breaks down into droplets, the size, shape and nature of which depend on the surface tension and viscosity of the liquid product. Thus the primary action with pressure nozzles is the production of a conical sheet which subsequently disrupts to give a fine spray of droplets. Basically this follows from the fact that a column or sheet of liquid becomes unstable when its length is greater than its circumference; a uniform thread will in fact produce uniform size droplets. However, when most atomization processes are considered, the threads of liquid tend to be uneven with resulting lack of uniformity in droplets. Dombrowski & Munday

(1968) have studied the processes of disruption of sheets of liquid using a high speed cine-camera technique but very little information appears to be available on the behaviour of liquid food products; however it is well known that droplet formation is resisted by highly viscous and non-Newtonian food products. Once the droplet is formed it falls at a relatively high velocity until it reaches the terminal velocity which remains constant for the remainder of the journey. The amount of drying which the particle undergoes depends upon the distance of fall and rate of heat transfer. Thus there are two stages in the evaporation—during the retardation and during the constant fall period. An important aspect prior to the final stage of surface hardening is the loss of volatiles which impart flavour to food products; this is governed by the relative volatilities of the components.

Amongst the products which are spray dried, milk, eggs, beverages and tomato paste are commercially the most important. Clarke (1965) has discussed a number of aspects of the spray drying of liquid foods. Peri (1966) has studied the survival of micro-organisms at different levels of relative humidity, together with the mechanisms of heat and mass transfer.

With the expansion of the tomato products industry a demand arose for the production of tomato powder and spray drying was found to be commercially acceptable. The feasibility of this process was demonstrated on an experimental plant by Lazar *et al.* (1956). The main types of plant used for this are:

(i) *High temperature process.* This uses rotating vaned disc atomizers and an average drying chamber temperature of between 170°F and 190°F (Masters, 1970). The design of the drier is particularly important and special precautions have to be taken in relation to the flow characteristics of tomato paste and the hygroscopic nature of the product. The air entering the chamber is at a temperature of between 280 and 300°F and is preheated by passage through a jacket round the drying chamber. This also assists in the cooling of the lower conical wall section of the chamber. The conical section is designed so that the product is deposited on the wall up to 1 in. in thickness before it breaks away; this ensures sufficient residence time for the completion of the drying. The product in the form of brittle nodules (3–3·5% moisture) is recovered at the base of the drier and any powder entrained in the air stream removed by a cyclone. The powder is packed in an inert atmosphere and requires an air and moisture-proof container for storage. In-packet desiccation is also used to prevent the highly hydroscopic powder lumping. A French plant situated in Portugal uses a 26-ft diameter chamber which is 62 ft high. The drying temperature is 108°F although a special air disperser is used which utilizes air at 260°F. (Anon., 1970a).

(ii) *Low temperature process.* An alternative approach to the above process has been to use air at ambient low temperature which has been thoroughly dried by passage through silica gel. (Goben, 1967; Anderson, 1969.) This involves the use of extremely high towers and produces a dry tomato powder of 11% moisture—which requires bin

drying to 3% moisture. The Danish process (Goben, 1967) 'Tower/Dri' is slightly different in as much as warm air is used which together with a high tower reduces the moisture content to between 3 and 3·5%.

For fruit juice dehydration the previously described processes are also used. Urbanek (1966) has described a high temperature process for the drying of citrus juices. The drying chambers are 42 ft in height and the air is held at a constant temperature at 195°F. The powder is recovered by a series of cyclones. An important feature of the process is the incorporation of gum acacia to reduce the hygroscopicity of the products. The mode of action of spray drying aids—which include sucrose, corn syrup solids lactose, gums, starch, carboxy methyl cellulose salts and glyceryl monosterate—is not fully understood.

Drum drying

One of the more important techniques for drying liquid food products is the drum drier. The high rate of heat transfer is obtained by direct contact with the hot surface and the equipment may be used either under atmospheric or vacuum conditions. The success of drum drying depends on the application of a uniform film of maximum thickness. This is controlled by the temperature of the surface of the drum and the speed of rotation. The thickness tends to increase with increasing temperature and higher temperatures permit an increase in drum speed and hence capacity. Published data on heat transfer coefficients are very limited, but figures of the order of 200–220 Btu/hr ft^2 °F are considered reasonable. The heat transfer coefficient varies around the circumference and heat transfer tends to be uneven. The nature of the surface also affects the rate of drying. The final moisture contents are often not as low as required and a final drying operation involving a fluidized bed is required.

Drum drying is extensively used for the manufacture of 'instant' potato products, potato flakes, and also for all liquid food products including infant foods, fruit products, eggs, milk and beverages.

Freeze drying

In this method of removal of water the product is frozen and the temperature maintained below the triple point of the constituent aqueous solutions so that the water vapour can be sublimed from the frozen solution. There is therefore a direct transfer from solid to vapour without the ice melting and passing through the liquid phase. The process is carried out under vacuum to provide a high vapour diffusion potential and is accelerated by supplying heat in some convenient form, either radiant, conductive or from microwaves. It is generally considered that as a means of dehydration it produces a dried product of the highest quality and therefore is potentially an extremely attractive method. As a developing technique it provides a good example of the

author's initial contention that successful commercial exploitation of a competitive technique results from a study of the fundamental processes involved.

The literature on heat and mass transfer in freeze drying systems is extensive and the details will be dealt with later, but it clearly shows how the developments both in process design and improvement in quality have resulted from these basic studies. Unlike other techniques of dehydration the literature of freeze drying is very considerable and several important reviews and books are available by Flosdorf (1949), Harper & Tappel (1957), Hanson (1961), Cotson & Smith (1963), Burke & Decareau (1964), Guygo, Z.I., Zhuravskya N.K. & Kaukhcheshvili Z.I. (1966), Neumann (1955), Noyes (1968) and several proceedings of symposia, Smith (1963), Fisher (1962) and Rey (1960, 1964a, 1966).

Pretreatment and freezing of foods prior to freeze drying

Each product requires an extensive pretreatment process prior to freeze drying—blanching to inactive the enzymes—and final quality not only depends upon the preliminary specification but also on the correct pre-treatment (Rolfe, 1964; Smithies, 1962, 1966).

Considerable attention has been directed towards the effect of freezing rate on the reconstitution of freeze dried products since it is well known that the porous nature of the product is controlled by this factor (Luyet, 1962; Rey & Bastien, 1962; Kuprianoff, 1962). The basic process of vapour removal is governed by the size, shape and tortuosity of the pores and in general the faster the freezing rate the smaller the voids and the slower freeze drying rate (Karel, 1963; Lusk, Karel & Goldblith, 1965; Carl & Stephenson, 1965; Haugh *et al.*, 1968). King (1968) showed that this was the case with turkey meat by using mercury porosimetry (see Holdsworth, 1963) and surface area measurements. In some cases, e.g. coffee, rapid freezing leads to a loss of the desired colour, and consequently slow freezing or two step freezing is required (Elerath & Pitchon, 1968). The problem is complex since there is a greater retention of flavour volatiles as the drying time is reduced (Picallo, 1969). With rapid freezing there is less time for diffusion and concentration of the components and consequently the distribution of components is not as widespread as with slow freezing. Two further aspects should also be considered and these are the incomplete freezing of all the water and recrystallization during freeze drying. The latter is important with regard to drying of fruit juices where it is often observed that it is difficult to prevent the solid from melting. (Quast & Karel, 1968; Rey, 1964b). Heating to the recrystallization temperature and then recooling has been suggested as a useful method of improving the freeze drying operation.

The whole subject of prefreezing rate would appear to be a useful area for more detailed investigation, both with regard to freezing by vacuum control and also using a refrigerant cycle.

Physical mechanisms

The major aspects of the mechanism of freeze drying are (a) the removal of vapour from the subliming ice front within the material, (b) the removal of vapour from between the food particles, (c) the supply of heat to the food particles, and (d) the supply of heat to the ice within the food particles (Kramers, 1958). Of these the removal of the vapour from the dried particle and supply of heat to the ice within the particle to accelerate the sublimation are the most important. Recent reviews on the subject have been presented by King (1970), Meffert (1968), Goldsworth (1968), Cluistra & Meijer (1968a), Spiess, Wolf & Hederer (1970), Roissart & Laederich (1966), Cornaz (1966), Bouldoires & Bricout (1969) and Dyer & Sunderland (1967, 1968a, b). The main object of the study of transport phenomena in drying operations is to be able to predict from the physical properties and geometrical organization of particles the length of time required to dry a product to a given moisture level. This will consequently be related to the overall economics of the process. Although it is seldom possible to achieve total design with this type of approach a knowledge of the main mechanisms operating and resisting the progress of the process leads to modifications based on sound scientific data rather than '*ad hoc*' investigation. The reduction of drying rates is most important in freeze drying because of the long time cycles involved.

With regard to vapour transport the sharpness of the frozen front has been subject to much speculation and it is generally thought that at some stage the front is diffuse rather than sharp. This means that a proportion of water molecules remain on the surface of the pores after the ice has retreated and these are removed during secondary drying (Meffert, 1967; Brajnikov *et al.*, 1969; Luikov & Vasiliev, 1969). The sharp transition model is, however, considered to be reasonable for mathematical treatment (Bralsford, 1967; Beke, 1969; King, 1970).

It has become increasingly realized that the porosity and the permeability are important parameters in any model used for predicting freeze drying behaviour. The porous properties of freeze dried products have been summarized by Holdsworth (1968). Since that time there has been further interest and Gunn, Clark & King (1969) have examined the implications of permeability on the transport properties of freeze dried turkey meat. Spiess *et al.* (1969) have studied the effect of internal structure using eight structural models and have presented data for freeze dried egg white and potato starch solution. Berlin, Kliman & Pallansch (1966) and Blond-Coste & Medas (1969) have presented data for surface areas and densities of freeze dried foods with regard to the role of dry layer permeability. Quast & Karel (1968) have examined various permeability models and correlated the rates of freeze drying with these mathematical formulations. More recently Stuart & Closset (1971) have carried out experiments to determine the effect of pore size. The use of distribution of pore size has been stressed (Holdsworth, 1963) as a better method of characterizing pore properties and correlating dehydration rates.

In order to study the effect of the variables it is necessary to examine the simultaneous transfer of heat and mass. The method adopted by King (1970) was to consider a model with a heat source at temperature T_1, a partial dried solid surface temperature T_2, an ice front temperature T_3, partial pressure P_1, and a condenser for moisture with an equilibrium partial pressure P_2. If h_1 and h_2 are the heat transfer coefficients external to and internally within the material, and K_1 and K_2 are the corresponding mass transfer coefficients, then the basic flux equations are given by equations (5) and (6).

$$Q_{heat} = H(T_1 - T_2) \quad \text{where } \frac{1}{H} = \frac{1}{h_1} + \frac{1}{h_2} \tag{5}$$

and

$$Q_{moisture} = K(P_1 - P_2) \quad \text{where } \frac{1}{K} = \frac{1}{K_1} + \frac{1}{K_2} \tag{6}$$

The flux is also related to the internal heat transfer process by equation (7)

$$Q_{heat} = h_2(T_3 - T_2) \tag{7}$$

and the latent heat (L) equation $Q_{heat} = LQ_{moisture}$ (8)

thus

$$h_2(T_3 - T_2) = L\left(\frac{1}{1/K_1 + 1/K_2}\right)(P_1 - P_2) \tag{9}$$

Equation (9) gives the inter-relationship between the physical properties and the operating variables. The overall rate of the freeze drying process depends upon the magnitude of the driving forces which should be as high as possible. Thus it is necessary to examine equation (9) in order to see which factors maximize the driving forces. For a high moisture mass the transfer rate h_2 should be large compared with K_1 and K_2 and for a high heat transfer rate h_2 should be small compared with K_1 and K_2. There are, however, limitations to raising T_1, and consequently T_3, and these are either thermal damage or melting. If h_2 is low compared with either K_1 and K_2 then the former will occur and the process will be heat transfer controlled. The main endeavour will therefore be to increase h_2. The thermal conductivity of the freeze dried products is influenced by the surrounding gas and consequently gases such as hydrogen and helium have a beneficial effect on freeze drying (Harper, 1962; Harper & El Sahrigi,

1964; King, Lamb & Sandall, 1968; Triebes & King, 1966; Dyer & Sunderland, 1969; Sole & Spiess, 1969; Kan, 1966b). The thermal conductivity increases with the pressure over a range of 50–1000 mm Hg. (Kessler, 1962; Meffert, 1968). Mass transfer control on the other hand will occur when the melting limit is reached, thus an increase in total pressure decreases K_1 and tends to increase h_2. At some particular pressure a transition may take place from heat transfer to mass transfer control. Mass transfer control also tends to predominate when particles of frozen liquid foods are being dried on heated plates by conduction, since h_2 is large and the temperature of the sublimation form is very close to that of surface temperature.

Equipment and processes

The conventional freeze drying unit consists of a vacuum chamber into which trays of the material to be dried can be placed, and a method of supplying heat to the material so that the sublimation process can be accelerated. The usual method is to arrange the trays on or between the heated plates which are either electrically heated or internally heated with steam, pressurized hot water or oil (Ginzburg, 1969; Ginzburg & Lyakhovitsky, 1969; Zamzow & Marshall, 1952). The vacuum is produced either with a mechanical pump, suitable steam ejectors, or refrigerated condensers (Rowe 1963, 1964). Various arrangements of the last are used depending on the method of defrosting. Forrest (1962) gives a good account of the equipment available during the period 1950–1960.

Although the majority of freeze drying plants are batch in nature several continuous systems have been designed and some are operative. (Dalgleish, 1963; Hackenberg, 1966; Rockwell *et al.*, 1965; Rockwell, Kaufman & Lowe, 1967; Pfluger, Ewald & Elerath, 1968; Fuentevilla, 1966; Eilenberg, 1970.) The main problems involve the development of suitable vapour locks for introducing the food into and removing food from the drying chamber, and obtaining uniform distribution of particles of the foodstuff.

With regard to methods of accelerating vapour removal, liquid and solid desiccants have been proposed and used. With liquid desiccants these would continuously remove moisture from the vapour and require low temperatures for absorption. This is usually operated by allowing a thin film of refrigerant to fall down the condenser surface, or alternatively to spray the cold liquid into the absorption chamber. Solid desiccants, such as molecular sieves, have also been investigated by Rowe (1963) and more recently by Saravacos (1967).

A considerable amount of work has been devoted to improving heat transfer during freeze drying. In the early techniques heat was supplied by conduction from plates by clamping the food between them. This, however, restricted the vapour flow and also provided uneven contact. In order to overcome this, expanded metal inserts were used between the plates and the metal. This process referred to as AFD (Accelerated Freeze

Drying) was fully described by Hanson (1961). A method of accelerated heat transfer into meat steaks was developed by Smithies & Blakley (1959) and Cole & Smithies (1960) and this involved the use of spikes on the trays (Mink & Sachsel, 1962). A further development in the design of trays, aimed at improving heat transfer, has been the use of longitudinally ribbed trays. This method affords a way in which the surface area may be considerably increased for both conduction and radiation. The trays are made of aluminium which has a relatively high thermal conductivity and the design is such that the trays have a high mechanical strength which makes them suitable for mechanical handling (Abelow & Flosdorf, 1957; Hackenberg, 1966; Oetjen, 1966).

A technique which has evolved directly from the basic study of heat and mass transfer phenomena is that of using cyclic pressure variation. It was briefly mentioned earlier that the requirements for maximum thermal conductivity of the porous dried layer and also for moisture diffusion were not the same. Thus it was considered that it might be advantageous to vary the pressure such that one cycle favoured heat transfer whilst the other favoured vapour diffusion. Several workers have investigated this process and indicated that some reduction in overall process time may be achieved (Mellor, 1961, 1966, 1967; Mellor & Munns, 1968; de Buhr, 1966; Kan, 1966a). This improvement is probably associated with other factors, such as inert gases used.

Several workers have investigated the use of much higher pressures, such as atmospheric, for freeze drying (Meryman 1959; Lewin & Mateles, 1962; Woodward, 1964; Sinnamon, Komanowsky & Heiland, 1968). The only requirement necessary to achieve this in practice is to maintain the partial pressure of water vapour in the chamber at the equilibrium value of that in the frozen food and this can be done by the presence of another gas (Dalton's Law). Recirculation of the gases to remove water vapour is the method usually adopted. The only drawback is the length of time required for the process, namely 30–40 hr to dry 8 mm thick discs of carrot.

Fluidized beds have also been used for small particles of frozen liquid food products, however only shallow beds could be used because of the pressure drop causing melting at the bottom. The beds did not expand uniformly because of the expansion of gases during movement upwards towards the vacuum area (Mink & Nack 1966; Mink & Sachsel, 1968; Dryden *et al.*, 1966; Sachsel & Mink, 1967; Malecki *et al.* 1970). Azeotropic processes have also been used to remove the water from frozen products. The main problem is the removal of the residual traces of the azeotrope forming liquid—e.g. ethyl acetate or alcohol used to extract the water (Wistreich & Blake, 1962; Bohrer, 1970).

One of the more important developments in the equipment field has been the application of microwave heating to freeze drying. In theory this method of heating overcomes the normal barriers to heat transfer by supplying heat not at the porous dried surface but in fact at the point where it is most needed within the frozen mass. This is because microwave energy is utilized in products of high dielectric loss (Copson, 1962). Although water has a high dielectric loss factor the factor for ice is very low and

this restricts the usefulness of the process. However, it has been shown (Hoover, Markantonatos & Parker, 1966, 1967) that the drying times using microwave heating were similar despite differences in the size of the specimens being dried. It has been suggested that microwave energy may best be utilized in the removal of final moisture from almost dry products (Decareau 1962, 1963; Leatherman & Stutz, 1962; Gall & Plante, 1962; Anon., 1963; Horejsi, 1963; Meryman, 1964). The main problems to be overcome are non-uniform heating, impedance matching, and ionization causing glow discharge. The economics of the process have been studied by Hammond (1967) in relation to radiant heating. Longmore (1971) has given an account of the recent position on equipment.

Quality characteristics and deterioration during storage

The high quality associated with freeze dried foods is attributed to several factors, namely the mild conditions to which the product is subjected, the failure to shrink associated with concentration and diffusion of soluble components, and the retention of aroma.

With regard to the last point Thijssen & Rulkens (1969) have recently carried out considerable work on model systems. These workers, as well as Menting (1969), have shown that diffusion is the main mechanism for the retention of flavour volatiles. The binary diffusion coefficients for organic compounds (aroma models) in sugar-water systems decrease very rapidly with increasing sugar content. The diffusion of acetone and water in coffee-water solutions was also studied and it was found that the acetone diffusion coefficient was much lower than the corresponding water figure. This was taken as evidence for the selective retention of aroma compounds when water is removed by drying. From this work it was concluded that the degree of aroma retention should increase with increasing drying rate, (Speiss, 1964), with increasing thickness of eutectic solid and with a decrease in water concentration. Other work has been reported by Flink & Karel (1970a, 1970b), Lerici & Pallotta (1970), Kallistratos & Sensbusch (1964), Saravacos & Moyer (1968) and Saravacos (1968).

With regard to shrinkage of products during drying, it has been shown (Kluge & Heiss, 1967) that the amount of shrinkage is proportional to the amount of unfrozen water in the frozen product. It is this latter which determines the degree of collapse and consequent shinkage of the product. Thus, provided the samples are adequately frozen, little change in dimension would be expected with freeze drying. The condition most likely to cause shrinkage would be if the temperature of the sample increased sufficiently for melting to take place (King *et al.*, 1968; Beke, 1969).

The amount of uptake of water by the dried product is important in determining the quality of the product (Smithies, 1962) and, if there has been any significant biochemical change, e.g. denaturation of protein and surface hardening, then rehydration will be poor. Several detailed studies have been reported for pork (Beke, 1969), for

turkey meat (King *et al.*, 1969), for shrimps (Goldblith, 1964) and for haddock fillets (Karel, 1963). The more general aspects of reconstitution and cooking of individual food products have been described by Brown (1963) and Hanson (1961).

An interesting study on the histological changes which take place during freeze drying revealed that whereas total collapse of the cell structure was common to all air dried vegetable tissue, the freeze dried products showed no cell collapse but some wall rupture.

The present state of knowledge of the physico-chemical basis for changes in texture may be briefly summarized as follows. In the case of fruits and vegetables it appears that increased crystallinity of cellulose is responsible for changes in texture which occur during drying. The increase in crystallinity is considered to be due to increase in hydrogen and this occurs not only during dehydration but also during blanching. It is thought that this may also be the basis for varietal differences (Shimazu & Sterling, 1961; Sterling & Shimazu, 1961; Speiss, 1964.) With animal and fish tissue the problems of texture deterioration have yet to be completely solved. Goldblith & Karel (1966) consider that the actomyosin complex is responsible for changes in water binding and of associated texture deterioration. The changes may occur by one or more of the following mechanisms (i) cross linking of undenatured protein resulting in aggregation, (ii) denaturation or partial denaturation of the proteins followed by aggregation, and (iii) interaction of proteins or denatured proteins with lipids or carbohydrates. Several workers (Wismer-Pedersen, 1965; Hamm & Deatherage, 1960; Penny, Voyle & Lawrie, 1963, 1964; Suden, Pearson & Dugan, 1964) have studied the use of additives such as EDTA, and pH adjustment and have concluded that improvement in texture can be achieved by treatment prior to dehydration and not at the time of rehydration, although Sosebee, May & Pwers (1964) showed that two proteolytic enzymes, papain and rhozyme P-11, were effective in reducing the cross linking during rehydration. With regard to fish, it has been shown that there is a loss of solubility of fish proteins during frozen storage due to the enzyme (esterase) activity releasing free fatty acid from the phospho-lipid component. The free fatty acid then reacts with the protein. Lipid oxidation also plays a part in the storage deterioration of dehydrated products (Lea, 1958; Toyomizu, Matsumura & Tomiyasu, 1963; Miller & May, 1965). The main reaction is one in which linoleic acid is the primary product, and Karel (1963) found that the reaction was a function of surface area of the dried product. This and other oxidative reactions has resulted in the breaking of the vacuum after freeze drying with inert gases, and high oxygen impermeable packaging materials. Oxygen scavengers such as glucose oxidase and hydrogen/palladium catalysts have been used but these increase the expense of packaging (Goldblith, Karel & Lusk, 1964; Bengtsson & Bengtsson, 1968). It has also been shown that the moisture level has a marked effect on the kinetics of the deteriorative reactions inasmuch as high moisture contents tend to retard lipid oxidation (Martinez & Labuza, 1968; Maloney *et al.*, 1966; Labuza, Maloney & Karel, 1966).

An important deteriorative reaction which has been widely studied is non-enzymic browning due to the Maillard reaction, which produces insoluble brown polymeric compounds by the interaction of the carbonyl group of reducing sugars and the amino group of proteins and amino acids. Kluge & Heiss (1967) have studied in detail the reaction mechanism and kinetics of the browning reaction. The general conclusion was that the reaction was highly temperature sensitive and low temperatures were favourable for freeze drying.

Fruit juices are particularly susceptible to browning reactions and the work of Karel (1960, 1963) and Karel & Nickerson (1964) has shown that even at moisture contents as low as 0·8% browning occurred with orange juice powder. The rate of deterioration of colour was correlated with loss of vitamin C by oxidation. It is also known that oxygen is not necessary to promote this reaction since storage under high vacuum conditions also produces deterioration (Krebes & Behun, 1963). The identification of deterioration products has never proved easy, but Huang & Draudt (1964) isolated what appeared to be fructose-aspartic acid, fructose-asparagine and an ammonia/D-glucose complex from freeze dried peaches.

With regard to enzyme activity in freeze dried foods it is well known that unless a blanching process is carried out or inhibitor is present prior to freeze drying, the enzyme system survives the process and becomes active in causing deterioration when the product is rehydrated. The level of moisture required has been the subject of much investigation and has involved adsorption studies of water vapour on the dry product. The water penetration capacity is much greater than would be suggested from nitrogen adsorption isotherms and this is attributed to diffusion of the water through the cell wall into blind pores. It appears that for each product a moisture level has to be defined for which stability is achieved. Figures in the region of 2% are quoted although higher levels can be tolerated by some products (Acker, 1963a, b; King, 1968; Labuza, 1968; Salwin, 1962, 1963, 1959; Saravacos & Stinchfield, 1965; Simatos, 1964; Simatos & Blond-Coste, 1967; Salwin & Slawson, 1959; Brockmann, 1966).

In concluding this section on deterioration it may be stated that much more biochemical work is required on the study of deterioration reactions, in particular the role of changes during freezing, oxidation of the non-lipid components, the role of free radicals (Munday, Edwards & Kerkut, 1962; Munday & O'Grady, 1965; Malinovski & Kafalieva, 1964) and also the microscopic and morphological structure of freeze dried products (Chauffard, 1969). For general accounts of deteriorative reactions see Speiss (1966), Goldblith & Karel (1966), Pazola (1964), Gorling (1962), Cluistra & Meijer (1968b), King (1970), Burke & Decareau (1964), Hollingsworth (1963) and Goldblith (1969).

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Measurement of oxygen penetration into meat using an oxygen micro-electrode

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Summary

An oxygen microelectrode was applied to the measurement of the penetration of oxygen into lean meat exposed to the air. The variation of oxygen penetration with time, temperature and muscle type was investigated. Results show that penetration increases fairly linearly with time, until heavy surface contamination occurs. Fairly large variations were observed between different muscles. The results were compared with related colour and muscle-respiration data.

Introduction

The penetration of oxygen into lean meat, exposed to the air (either directly or inside a plastic film package), is governed by the rates of oxygen diffusion into the meat and oxygen consumption by the meat. The colour of meat depends on the degree of oxidation of the purple pigment myoglobin, bright red oxymyoglobin being formed near the surface where the oxygen tension is high, whereas slow formation of the brown pigment, metmyoglobin occurs where the oxygen tension is low. The resulting appearance of meat thus depends on the depth of penetration of oxygen. An attractive visual appearance of pre-packaged meat has assumed particular importance with the increasing preponderance of fresh meat sales by supermarkets.

Hitherto, oxygen penetration into meat has had to be estimated either from colour measurements (Brooks, 1929) or from oxygen consumption data (Brooks, 1936; Atkinson, Follett & Ratcliff 1969). The development of the oxygen electrode (Cater, Silver & Wilson, 1959; Cater & Silver, 1961, 1966) has enabled direct measurements to be made of the oxygen tension in living tissue. It was therefore of interest to investigate the applicability of this technique to the study of oxygen penetration into meat.

Measurements were made of the penetration of oxygen into different muscles of beef, pork and lamb and its variation during storage at 0°, 10° and 20°C.

Materials

Experimental samples of meat consisted normally of intact excised muscles, usually

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from freshly slaughtered animals, but sometimes 24 hr after death. In the case of large muscles, the longer axis was shortened to approximately 14 cm.

Methods

The oxygen electrode

The technique of making and using oxygen electrodes followed closely the methods developed by Cater *et al.* (1959) and Cater & Silver (1961, 1966). The oxygen electrode used was the rhodium plated needle type, made from a stainless steel surgical needle (Fig. 1 (a)). The tip of the surgical needle was electropolished to a diameter of about 20 μm, this giving adequate resolution of measurement yet being sufficiently robust. The electrode was then insulated with several coats of epoxy resin except at the extreme tip. This was done by dipping the electrode into suitably-diluted epoxy resin, withdrawing slowly and partially curing in an oven at 200°C for 1–2 min. The electrode was then lowered by means of a micro-manipulator, until the tip just broke the surface of

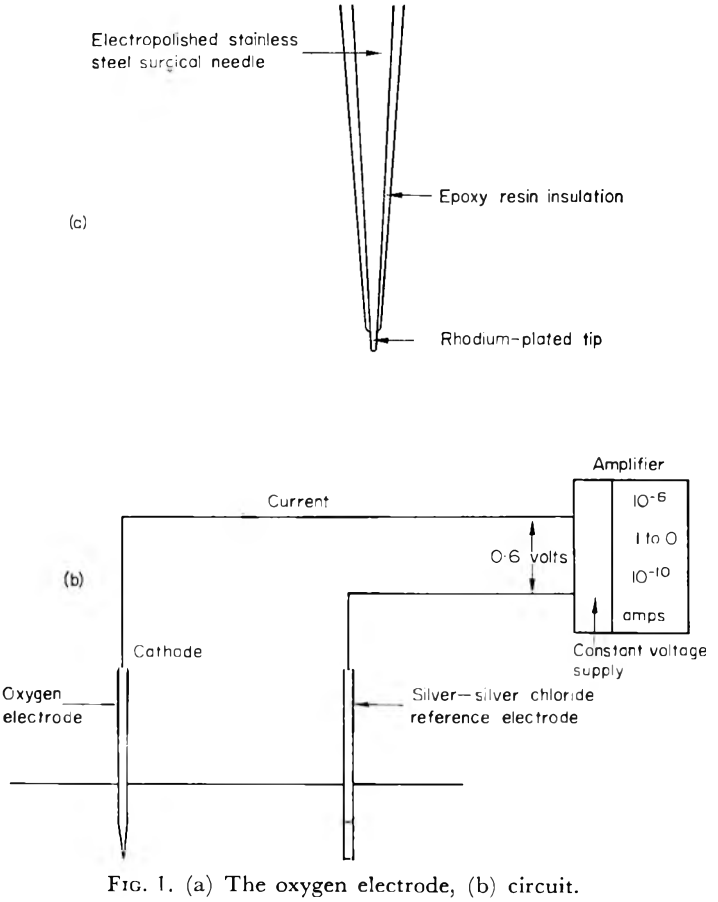

FIG. 1. (a) The oxygen electrode, (b) circuit.

some epoxy resin solvent. The insulation was then cured for 10 min at 200°C and the procedure repeated several times, the insulation being finally cured until golden-yellow in colour. Finally the tip of the electrode was plated with nickel and then rhodium.

A negative potential of 0·6 volts was applied to the oxygen electrode relative to a silver–silver chloride reference electrode in the medium, and the electrode current produced was measured using an electrometer amplifier incorporating a built-in electrode voltage supply (Fig. 1(b)). After less than 10 min, the electrode current reached a steady value, this current being proportional to the concentration of dissolved oxygen around the electrode tip. Electrodes were calibrated in physiological saline equilibrated with air (21% oxygen), oxygen-free nitrogen, and standard gas mixtures of 2·0%, 7·8% and 15·3% oxygen. A typical calibration is shown in Fig. 2, oxygen tension (Po_2) being expressed both as percentage oxygen, and as mm Hg for a dry atmospheric pressure of 760 mm Hg.

When the electrode current was measured at different applied potentials, the 'plateau region' was found to be between about 0·4 and 0·8 volts, the current changing relatively gradually between these voltages. Therefore a potential of 0·6 volts was always applied to the electrodes. Experiments were performed to verify whether the changes in the chloride concentration of the extracellular fluid in meat due to muscle fibre breakdown, would affect the electrode calibration by changing the potential of the silver–silver chloride reference electrode. An oxygen electrode was calibrated in physiological saline, which was connected by a salt–agar bridge to some meat, into which was

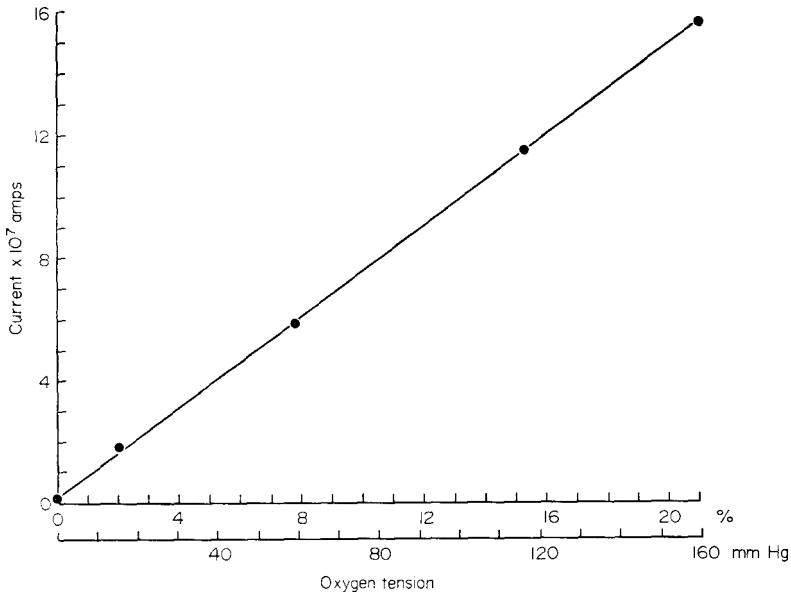

FIG. 2. Typical electrode calibration.

inserted a silver–silver chloride reference electrode. There was no change in calibration during 15 days of observation at + 1°C. The calibration was the same as that obtained with the reference electrode in the calibration saline or with the reference electrode in saline connected to the calibration saline by a salt-agar bridge.

On calibrating the air-saturated saline at different temperatures between 0° and 20°C and standardizing each calibration to a dry atmospheric pressure of 760 mm Hg, it was found that the electrode current increased by, on average, 2% per °C rise in temperature. When oxygen electrodes were used in meat, their calibrations were found to decrease gradually due to 'poisoning' of the rhodium-plated tips. It was found that the decrease in calibration was generally about 2% for every 8 hr of oxygen measurements in meat. The electrode calibration was checked at the beginning and end of each experiment and after every 40-hr period of measurement. A linear decrease was assumed between successive calibrations. Electrodes were restored to their original condition by washing in saline for several hours and then replating a fresh surface.

Oxygen penetration measurements

The oxygen electrode was inserted into muscle, using a micro-manipulator, and then withdrawn slightly in order to release the compression and thereby remove the depression in the muscle surface. The depth of insertion was determined by measuring with a cathetometer the distance between a fine reference mark on the electrode, at a known distance from the tip, and the top of a known length of hypodermic tube resting on the muscle surface, Fig. 3. Depth measurements were estimated to be accurate to within 0·05 mm.

Readings were taken at different points over the surface and at depths near the edge of the penetration profile, i.e. 0 to 1% Po_2 (0 to 7·6 mm Hg). The variation of oxygen

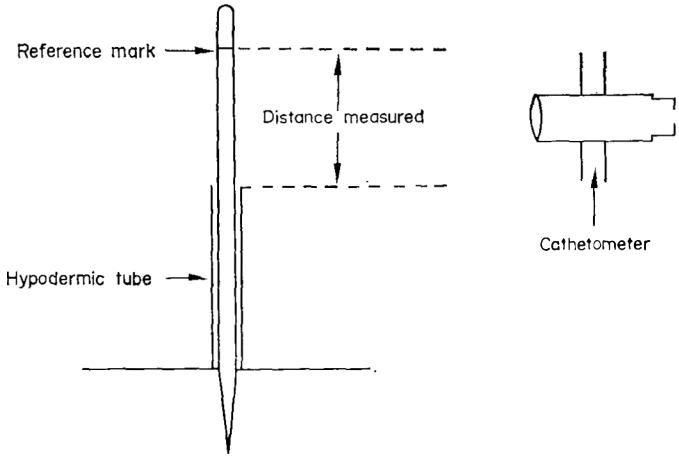

FIG. 3. Measurement of depth of insertion.

penetration during storage was measured for different samples of various muscles. Each muscle sample was stored in air at a constant temperature (±0·5°C) of either 0°, 10° or 20°C. Penetration measurements were taken on the same intact muscle surface throughout storage, and hence muscles were kept under very humid conditions so as to minimize drying. This was done by enclosing the muscles in partially covered dishes containing water, and occasionally wetting the muscle surfaces with saline. In typical cases the evaporative weight loss was found to be about 3½% after 6-days storage at 10°C, which caused no change in consistency of the top few mm of meat.

Initial penetration measurements were made at 24–31 hr post mortem, after having allowed the oxygen penetration to settle down. On five occasions, the muscle sample was bisected, one half exposed to the atmosphere at about 2 hr post mortem and the other at 24 hr post mortem. It was found that on each day during storage, there was no significant difference in penetration, at the 0·05 significance level, between the halves. Penetration measurements were, in every case, for oxygen diffusion in a perpendicular direction to the long axis of the muscle, although there was no evidence that the direction of the fibres relative to the diffusion had any effect on the penetration.

Results

Oxygen penetration was found to increase fairly linearly with time. Eventually the rate of increase in penetration began to fall until finally the penetration ceased to increase and commenced to decrease with time, as putrefaction set in. A typical graph of the variation of penetration with time is shown in Fig. 4. The departure from linearity of the rate of penetration increase, was attributed to the surfaces of the muscles becoming heavily contaminated with oxygen-consuming micro-organisms. The duration of the period of linearity was generally about 40 hr at 20°C, 4 or 5 days at 10°C, and 17–20 days at 0°C. These times are similar to those required for slime to develop, at near 100% r.h., on meat surfaces which are fairly clean initially (Mossel & Ingram, 1955).

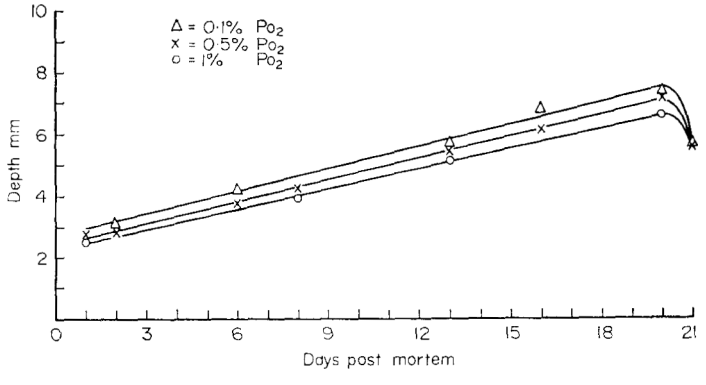

FIG. 4. Variation of oxygen penetration with time for pork *semitendinosus* at 0°C (animal 1).

TABLE 1. Oxygen penetration at 0°C

Muscle sample	Animal number	Oxygen tension	Initial depth (mm)	Intercept a (mm)	Slope b (mm/day)	95% Confidence limits of slope ±	Coefficient of correlation	95% Confidence limits of depth (d) at time (t) from $d = a + bt$
Pork								
Longissimus dorsi	1 (Pietrain)	0·5%	2·6	2·30	0·289	0·009	0·998	0·29
		1%		2·31	0·257	0·026	0·997	0·24
Semitendinosus	1 (Pietrain)	0·1%		2·71	0·243	0·027	0·983	0·72
		0·5%	2·7	2·40	0·235	0·008	0·997	0·27
		1%	2·5	2·24	0·221	0·012	0·998	0·26
Semimembranosus	1 (Pietrain)	0·5%	2·4	2·20	0·274	0·019	0·997	0·37
		1%		2·12	0·265	0·035	0·985	0·62
Beef								
Longissimus dorsi	2	0·5%	3·2	3·03	0·187	0·017	0·985	0·23
		1%		2·76	0·180	0·028	0·991	0·32
Semitendinosus	2	0·5%	3·2	3·02	0·252	0·052	0·969	0·46
		1%	2·9	2·84	0·226	0·048	0·958	0·47
Semitendinosus	3	0·5%			0·280	0·040	0·985	
Sternomandibularis	4	0·5%	2·7	2·65	0·103	0·015	0·955	0·18
		1%	2·5	2·38	0·110	0·025	0·962	0·25
Sternomandibularis	2	0·5%	2·9	2·63	0·214	0·011	0·993	0·19
		1%	2·7					
Mean for 0·5% Po_2			2·8		0·23			
Range for 0·5% Po_2			2·4–3·2		0·10–0·29			

TABLE 2. Oxygen penetration at 10°C

Muscle sample	Animal number	Oxygen tension	Initial depth (mm)	Intercept a (mm)	Slope b (mm/day)	95% Confidence limits of slope ±	Coefficient of correlation	95% Confidence limits of depth (d) at time (t) from $d = a + bt$
Pork								
Longissimus dorsi	5	0·1%	2·5					
		0·5%	2·2	1·54	0·697	0·079	0·986	0·25
		1%	2·0					
Longissimus dorsi	6	0·5%	2·4	2·15	0·307	0·071	0·962	0·38
		1%	2·3	1·98	0·283	0·054	0·974	0·29
Longissimus dorsi	7	0·5%	2·6	2·28	0·309	0·070	0·964	0·39
		1%	2·4	2·09	0·287	0·120	0·940	0·54
Longissimus dorsi	8	0·5%	2·4	1·94	0·495	0·065	0·971	0·23
Longissimus dorsi	9	0·5%	2·1					
		1%	2·0					
Semitendinosus	10	0·1%	2·2					
		0·5%	1·9					
Semimembranosus	7	0·5%	2·3	2·09	0·178	0·062	0·970	0·24
		1%	2·1					
Psoas major	5	0·1%	2·0	1·84	0·194	0·119	0·852	0·26
		0·5%	1·8	1·67	0·133	0·095	0·754	0·26
Beef								
Sternomandibularis	11	0·5%	2·2	1·79	0·425	0·157	0·784	0·59
Sternomandibularis	12	0·5%	1·4					
		1%	1·2	1·08	0·103	0·127	0·520	0·35
Sternomandibularis	13	0·5%	1·8					
		1%	1·6					
Sternomastoideus	14	0·1%	1·9					
		0·5%	1·7					
Mean for 0·5% Po_2			2·1		0·36			
Range for 0·5% Po_2			1·4–2·6		0·13–0·70			

The results at 0°, 10° and 20°C are summarized in Tables 1, 2 and 3 respectively. The initial depths (i.e. at 24–31 hr post mortem) of the 0·1, 0·5 and 1% Po_2 levels are tabulated, together with the characteristics of the corresponding lines of regression of depth (mm) on time (days), namely, intercept (a) at zero time (post mortem); slope (b); 95% confidence limits of slope; coefficient of correlation; 95% confidence limits for the estimation of depth (d) at time (t) from the regression equation, $d = a + bt$. In obtaining values for the 0·1, 0·5 and 1% Po_2 levels, points were considered that were within $\pm 0{\cdot}1\%$ Po_2 of each of these levels. The absolute error in oxygen tension measurements below 1% Po_2 was estimated to be $\pm 0{\cdot}1\%$ Po_2.

Discussion

It can be seen from Tables 1, 2 and 3 that whilst at a particular temperature there are fairly large variations in the slope of the increase of penetration with time between different muscle samples, there are only relatively small variations in initial penetration. For a specific muscle, fairly large variations, particularly in slope can occur between samples taken from different animals, e.g. pork *longissimus dorsi* or beef *sterno-mandibularis* at 10°C (see Table 2). Similar variations occur between different muscles dissected from the same individual, e.g. animal 5 and animal 7 in Table 2. Thus the penetration of oxygen into muscle depends not only on the type of muscle but also varies from one animal to another. The averages of the initial penetration depths at 0°, 10° and 20°C are, respectively, 2·8, 2·1 and 1·2 mm for the 0·5% Po_2 level. It can be seen that on average, for a 10°C increase in temperature, the penetration decreases by roughly one-third.

The oxygen penetration values obtained here using oxygen micro-electrodes, may be compared with oxymyoglobin layer width measurements and also with muscle oxygen consumption rates, bearing in mind however the variability that occurs between different muscles. Oxymyoglobin layer measurements do not yield actual oxygen tension values, since there is some uncertainty as to the precise value of the oxygen tension at the observed boundary. Brooks (1929) measured oxymyoglobin layer widths by pressing a thin slice of muscle between two parallel glass plates and observing the oxymyoglobin layer at the tissue-glass boundary. He obtained the following mean values for beef *flexor* muscles at 18–24 hr post mortem: 2·2 mm at 0°C, 1·6 mm at 5°C and 0·9 mm at 15°C. Brooks obtained approximately linear width increase rates averaging 0·46 mm per day at 0°C. MacDougall (1971, private communication), using a similar device, has obtained oxymyoglobin layer widths for beef *longissimus dorsi* at 1–2°C of about $2\frac{1}{2}$ mm at 2–3 days post mortem, reaching $4\frac{1}{2}$–$7\frac{1}{2}$ mm at 7 days post mortem.

The oxygen tension (C) at any depth (d) in a plane semi-infinite slab of meat consuming oxygen at a constant rate (A), may be calculated from an equation due to Warburg (1923)

TABLE 3. Oxygen penetration at 20°C

Muscle sample	Animal number	Initial depth (mm) of 0·1% Po_2	Initial depth (mm) of 0·5% Po_2	Initial depth (mm) of 1% Po_2	Standard deviation (mm)
Pork					
Longissimus dorsi	15 (Pietrain)	1·5	1·3	1·2	0·05
Longissimus dorsi	16	1·0	1·0		0·05
Semitendinosus	17	1·2	1·1		0·08
Semitendinosus	18	1·5	1·3		0·08
Semitendinosus	16		1·1	0·9	0·08
Semimembranosus	18		1·5	1·4	0·14
Semimembranosus	17	1·4	1·3		0·08
Beef					
Longissimus dorsi	19	1·4	1·3		0·06
Sternomandibularis	19	1·0	1·0		0·06
Sternomandibularis	20	1·6	1·5		0·07
Sternomastoideus	20	1·4	1·4	1·3	0·07
Lamb					
Longissimus dorsi	21	1·1	1·0		0·07
Semitendinosus	21	1·0	1·0		0·08
Mean		1·3	1·2		
Range		1·0–1·6	1·0–1·5		

$$d = \left(\frac{2D\,C_o}{A}\right)^{\frac{1}{2}} - \left(\frac{2DC}{A}\right)^{\frac{1}{2}}$$

where C_0 is the partial pressure of oxygen at the muscle surface and D is the diffusion coefficient of oxygen through muscle. In deriving this equation it was assumed that the consumption rate (A) is independent of oxygen tension. However, Hill (1948) found that A decreases with Po_2 below 0·5 to 2·0 mm Hg (0·07 to 0·26%) for frog's muscle at 15° to 20°C, and if a similar fall-off in oxygen consumption rate occurs in mammalian muscle, Warburg's equation would underestimate the penetration at low oxygen tensions. Calculation of the depth of a given oxygen concentration requires a knowledge of the diffusion coefficient of oxygen in muscle. For the purpose of the following calculation, the value of Krogh (1918) for frog's abdominal wall had to be taken.

Brooks (1936) measured the rate of oxygen consumption by pork at 0°C, from which data a 0·5% Po_2 depth of 1·9 mm at 2–3 days post mortem may be calculated. After 3 weeks storage at 0°C the decrease in oxygen consumption of a single sample corresponded to an increase in penetration of only 0·4 mm. Brooks also measured the oxygen consumption of bacon at 0° and 15°C and calculated that an increase in temperature of 15°C would approximately halve the limiting depth of oxygen penetration. Atkinson *et al.* (1969) measured the oxygen consumption at 15°C of lamb *semimembranosus* stored at 0°C. On making calculations from their data using Warburg's equation, one obtains a 0·5% Po_2 penetration of 0·4 mm at 15°C after 22 hr post mortem storage at 0°C. The decrease in oxygen consumption during the 9 days of observation would correspond to a linear increase in 0·5 Po_2 penetration of 0·05 mm per day of storage at 0°C. Bendall (1971, private communication) has measured oxygen consumption rates between 0° and 37°C and obtained a temperature coefficient of 2·9 per 10°C. This corresponds to a 38% decrease in the limiting penetration per 10°C. increase in temperature. His oxygen consumption rates for beef *l. dorsi* after 48 hr post mortem in a chiller, are equivalent to calculated 0·5% Po_2 penetrations of 1·9 mm at 0°C, 1·2 mm at 10°C and 0·7 mm at 20°C.

Conclusions

An oxygen micro-electrode has been applied successfully to the measurement of the depth of penetration of oxygen into meat, and the following main conclusions reached:

(1) Oxygen penetration increases fairly linearly with time, until heavy surface contamination occurs, when it decreases fairly abruptly.

(2) Fairly large variations can occur between identical muscles taken from different animals and between different muscles dissected from the same animal.

(3) For the muscles observed, the variations of the initial penetrations (24–31 hr

post mortem) and rates of increase in penetration, for 0·5% Po_2, are respectively: 2·4–3·2 mm and 0·1 to 0·3 mm/day at 0°C; 1·4–2·6 mm and 0·1 to 0·7 mm/day at 10°C; and 1·0–1·5 mm at 20°C.

Acknowledgments

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The thermal variation of the density of beef and the determination of its coefficient of cubical expansion*

H. F. T. JARVIS

Summary

The variation of the density of beef muscle with temperature over the range 5–30°C is more than twice that for water. The mean coefficient of cubical expansion over this range is $3{\cdot}75 \times 10^{-4}\,°C^{-1} \pm 1{\cdot}5\%$ compared with $1{\cdot}7 \times 10^{-4}\,°C^{-1}$ for water. Within the limits of experimental accuracy there is evidence of maximum density between 3°C and 5°C. The mean coefficient of cubical expansion of beef fat over the same range is $15{\cdot}3 \times 10^{-4}°C^{-1} \pm 5\%$. The consequent overall variation in carcass density is discussed in relation to carcass composition.

Introduction

Since the early 1940s many investigations have been published in which specific gravity or density of a dressed carcass or joint has been used to determine its fat content. The method has been successfully applied to cattle (e.g. Kraybill, Bitter & Hankins, 1952; Garrett, 1967; Garrett & Hinman, 1969), pigs (e.g. Adam & Smith, 1964; Joblin, 1966) and sheep (e.g. Barton & Kirton, 1956; Kirton & Barton, 1958; Timon & Bichard, 1965). In most cases specific gravity has been determined by the standard method of displacement of water and used as a measure of fat, lean or water content.

Buck, Harrington & Johnson (1962) believe that the wide fluctuations in the muscle/bone ratio of pigs of the same fatness must limit the accuracy of the specific gravity method. Whilst the results of Whiteman, Whatley & Hillier (1953) seemed to contradict this assertion, Adam & Smith (1964) have found that the inclusion of the lean/bone ratio in the prediction equation reduced the residual variance in the calculated percentage of lean. Kline, Ashton & Kasterlic (1955) have shown that there is considerable variation in the specific gravity of a carcass stored in a chill room for varying lengths of time after slaughter. This variation is presumably due to two causes: one,

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* Mr Jarvis died before preparing his results and conclusions for final publication. This task was undertaken by Dr C. A. Miles, also of the Meat Research Institute, Langford, Bristol BS18 7DY.

water loss by evaporation from the surface of the carcass, and the other, changes in carcass temperature. In many papers (see the review by Garrett, 1967) there appears to have been very little attention paid to the variation with temperature of the specific gravity of the carcass. It therefore seems relevant to investigate what possible bearing changes in density due to temperature variation could have on the prediction of carcass composition.

The object of this series of experiments was to determine the change in the density of selected beef samples, both lean and fat, over the temperature range 0–30°C.

Theory

Using the classical method of finding density by weighing a solid sample in air and a liquid at the same temperature, let m = weight of sample air, m_1 = weight of sample in water, ρ = density of the liquid, D = density of the sample, then

$$D = \frac{m}{m - m_1} \rho$$

Where, as in the present studies, the liquid and solid are at slightly different temperatures, there will be a temperature (and therefore density) distribution within (a) the solid and (b) the liquid near the solid–liquid boundary. Neglecting (b), an average density at the mean temperature of the body can be derived from the equation above if the liquid density is taken to be that corresponding to the temperature of the bulk liquid.

Knowing the variation of density, D, with temperature, θ, the coefficient of cubical expansion can be calculated as

$$\gamma = -\frac{1}{D}\frac{dD}{d\theta}$$

If the density is D_1 at θ_1 and D_2 at θ_2, the mean coefficient over a limited range of temperature θ_1 to θ_2 can be written:

$${}_{\theta_1}\gamma_{\theta_2} = \frac{2(D_1 - D_2)}{(\theta_2 - \theta_1)(D_1 + D_2)}$$

Materials and methods

Fat and lean samples were cut from the thick flank of beef animals slaughtered in the licensed abattoir of the Meat Research Institute. The samples were stored in a chill room until required. A two-pan balance with an illuminated microscale reading to 0·1 mg was used, so that the sample, suspended from a pan, could be immersed in a

water bath at various temperatures. The range of the balance was from 0 to 200 g and the samples were of sufficient size to ensure a high degree of accuracy in the determination of their weight. Samples were approximately 5 × 5 × 2 cm, such size being easily available in beef muscle free from fat. This sample was enclosed in a cell constructed of thin copper-coated plastic measuring 5 × 5 × 2·5 cm. Since the density of the sample was not very different from that of water, a brass sinker was included in all weighings (Fig. 1).

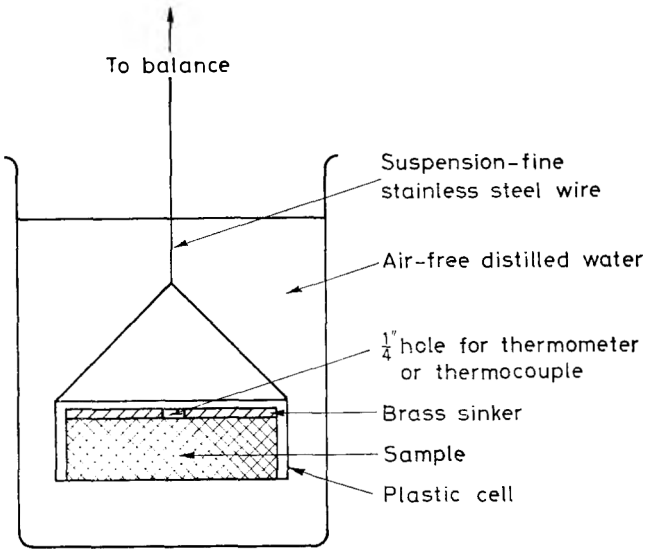

FIG. 1. Experimental arrangement for under-water weighings showing the cell containing the sample.

The following difficulties became apparent from initial experiments:

1. *Dissolved air.* All the distilled water in which the samples were weighed had to be freed from dissolved air by boiling before use, otherwise weighings became inaccurate due to the formation of air bubbles on the surface of the suspended cell and sample.

2. *Use of control bath.* Ideally, the temperature of the sample and the surrounding bath (A) should be taken at the instant of weighing. In practice this was not possible as any disturbance of the suspended system such as would be involved in the taking of its temperature, resulted in serious discrepancies in the weighings. To overcome this difficulty a control bath (B), cell and sample were provided. This bath contained exactly the same weight of water, the cells and brass sinkers were identical and the samples of meat were cut so as to be of equal weight as nearly as possible. It was then assumed that, provided A and B were subjected to the same external conditions, then the temperature difference observed between water and sample in bath B would be

the same in the suspended system A. Baths were stirred gently by hand until just before taking a reading.

In all cases the temperature of the sample was that determined at the centre, in the initial experiments by $\frac{1}{8}$ in. diam. thermistors to $\pm 0{\cdot}1°C$ and subsequently by 38 s.w.g. copper–constantan thermocouples with potentiometer to ± 1 μV. Since the sample was receiving or losing heat from its surface this centre temperature was slightly different from the mean temperature of the whole by about 0·1°C as calculated by classic methods (Carslaw & Jaeger, 1959) which is considered to be within the overall accuracy of the system. As the temperatures of the bath and the centre of the sample differed only by 0·4–0·6°C it was assumed that the centre temperature was sufficiently indicative of the mean sample temperature.

3. *Time factor.* In order to find the temperature of the sample, θ_A, at the instant of weighing it was necessary to know the three associated temperatures: φ_A, the temperature of bath A; φ_B, the temperature of bath B; θ_B, the temperature of the control sample in B. θ_A was then determined from the relation

$$\varphi_A - \theta_A = \varphi_B - \theta_B \qquad (2)$$

The above three temperatures were plotted for the duration of the experiment on a temperature–time graph from which θ_A was found at the instant of weighing. φ_A was often within 0·2°C of φ_B and did not differ by as much as 0·5°C in a typical experiment.

In order that the whole range 3–30°C might be covered in a comparatively short time (3 hr), volumes of 50 ml water were withdrawn from each bath at stated intervals and replaced by the same volume of air-free water at approximately 100°C, the whole of such an operation taking about $\frac{1}{2}$ min. Readings were then continued for a period of about 20 min before further changes of water.

4. *Loss of blood, etc., into solution from the surface of the sample.* Unprotected meat and fat visibly lost constituents and imbibed water in the course of 2 hr immersion. Two ways of overcoming this difficulty were used:

(a) The sample was enclosed in a light cellophane bag so that any juices exuded were confined to the bag. However, it was difficult to ensure that all air bubbles were excluded from the bag containing the sample under test.

(b) It was more satisfactory to coat the sample with a thin layer of polyurethane varnish and allow it to dry. The elastic skin allowed expansion of the sample but prevented interchanges. No significant change in the appearance of the samples, either lean or fat, was observed even after an immersion of 24 hr. Corrections for the weight of the varnish layer were applied to all readings.

Preliminary experiments to find the weight of the empty cell A together with its sinker and suspensions, in water at a series of temperatures, showed that any changes in this weight due to other thermal effects could be neglected.

The two baths A and B containing 800 ml of air-free, distilled water, together with the prepared samples of weights m_A and m_B, were removed from the chill room and placed in position in the apparatus at about 3°C. The suspended sample A and the control sample B were allowed to heat up to about 10°C, the temperatures of the baths and control sample B being taken at intervals of about 5 min. The weight of the suspended sample A was taken as soon as these temperatures were read.

Results

Lean beef

Fig. 2 presents the results of a typical experiment to determine variation in density with temperature of a single, lean beef sample. The density between 3 and 5°C shows

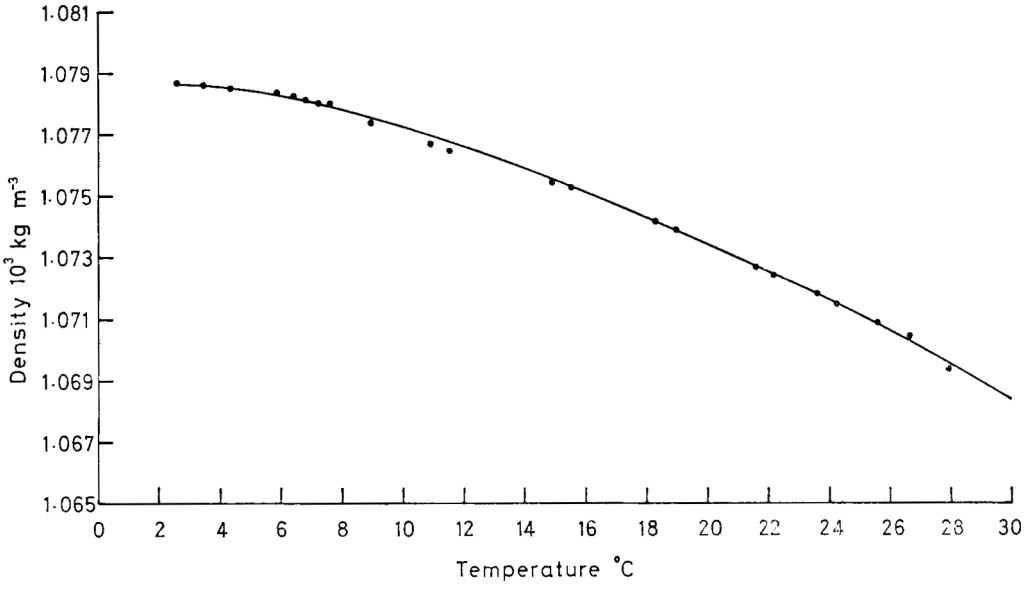

FIG. 2. The results of a typical experiment showing the variation in the density of a lean beef sample with temperature.

very little change, which is consistent with the maximum density of water at 4°C. Although there were indications of a decrease in density below 3°C, readings were unreliable.

Table 1 records the densities of lean beef samples in the temperature range 5–30°C based on experimental results such as those given in Fig. 2. Each sample was excised from a different carcass. Although there were measurable differences in the densities between samples at the same temperature, these differences amounted to no more than 0·3%. From the figures quoted in Table 1 and other similar results it was calcul-

TABLE 1. Densities of lean beef at 5°C intervals in three selected experiments

Temperature (°C)	Density ($kg/m^3 \times 10^{-3}$)		
	Experiment A	Experiment B	Experiment C
5	1·0784	1·0772	1·0765
10	1·0772	1·0762	1·0751
15	1·0756	1·0742	1·0728
20	1·0735	1·0721	1·0705
25	1·0712	1·0696	1·0689
30	1·0684	1·0670	1·0664

ated that the mean coefficient of expansion of lean beef muscle over the range 5–30°C was $3{\cdot}75 \pm 0{\cdot}04 \times 10^{-4}\ °C^{-1}$.

Beef fat

Table 2 records values of the density of fat. Fat densities showed more variation between samples (i.e. between carcasses) than the corresponding figures for muscle. The mean coefficient calculated over the range 5–30°C was $15{\cdot}3 \pm 0{\cdot}8 \times 10^{-4}\ °C^{-1}$, which is approximately four times that of lean beef and nine times that of water. A maximum density was indicated at about 5°C, below which data were unreliable.

TABLE 2. Density of beef fat for three successive experiments

Temperature (°C)	Density ($kg/m^3 \times 10^{-3}$)		
	Experiment D	Experiment E	Experiment F
5	0·9660	0·9580	0·9460
10	0·9581	0·9550	0·9460
15	0·9503	0·9420	0·9400
20	0·9440	0·9362	0·9340
25	0·9350	0·9273	0·9275
30	0·9248	0·9150	0·9212

Discussion

The variation in density of fat and lean meat with temperature is of interest in relation to the determination of specific gravity as a measure of carcass composition. Although the results of this study apply only to beef, temperature is likely to affect the determination of specific gravities of carcasses of other types in much the same way.

There is little mention of the importance of temperature changes in studies of the relation between carcass specific gravity and composition (see Garrett, 1967). In some cases, corrections have been applied to 'weight in water' to take the variation of the density of water with temperature into account (e.g. Adam & Smith, 1964; Joblin, 1966; Holme, Coey & Robinson, 1963.)

The variation in density of lean muscle and fat due to thermal changes is usually overlooked. The present paper shows that the coefficient of cubical expansion of lean muscle has a mean value between 5°C and 30°C, which is at least twice that of water, while that of fat is nine times that value. Fig. 3 illustrates the effect of temperature on the relationship between the specific gravity of a beef carcass, and its composition based on the experimental results presented above. It was assumed that the carcass could be considered as a three component system: fat, lean and bone. For the purpose of this calculation the density of the bone content (constant at 10%) was taken to be $2{\cdot}0 \times 10^3$ kg m^{-3} and independent of temperature.* In view of the small proportion of the carcass volume occupied by bone, errors in Fig. 3 introduced by the neglect of the thermal expansion of the bone content are likely to be small. Even if γ for bone is as high as that of water the error in the coefficient of cubical expansion of the carcass is only of the order of $8{\cdot}5 \times 10^{-6}$ °C^{-1}, i.e. a few per cent of the total coefficient. Referring to Fig. 3, differences in specific gravity due to differences in carcass temperature are smaller at low than at high fat contents. This is due to the coefficient of cubical expansion of beef fat being some five times greater than that of lean beef. Consequently, errors in predicted carcass composition due to experimental temperature fluctuation will be greater the higher the carcass fat content. It can be seen that for comparisons to be made of the specific gravities of carcasses considerable care must be taken to ensure that measurements are all made at the same temperature. As a rough guide which might be useful in experimental design, it can be deduced from Fig. 3 that if the carcass temperature is controlled to ± 1°C, the unexplained fluctuation in % fat due to thermal effects will be less than $\pm 0{\cdot}6$% even for the most obese carcass likely to be encountered.

* Since Fig. 3 was composed, single determinations of the density of beef bones at 18–20°C were made. The results were: os calcis, 1·45; patella, 1·27; astragulus, 1·47; femur, 1·40; tibia, 1·47. As these are lower than was assumed, the curves in Fig. 3 are shifted towards the Y-axis but the conclusions drawn are not radically altered.

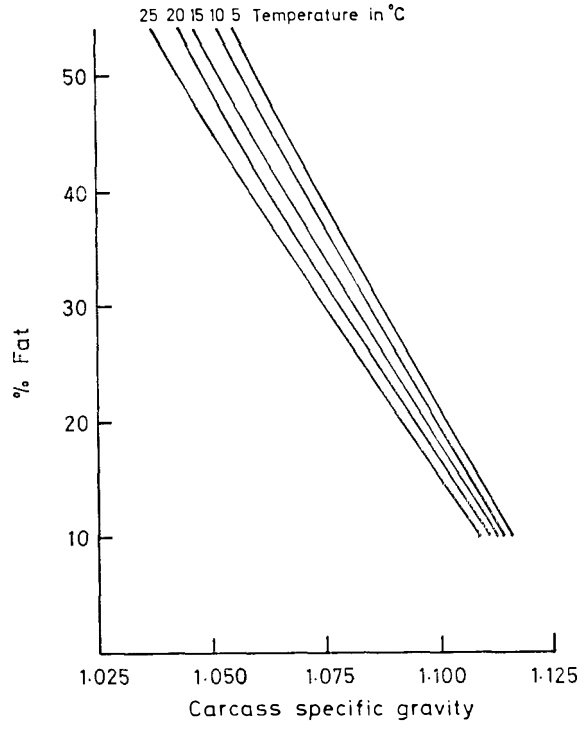

FIG. 3. A computed relationship between carcass specific gravity, composition and temperature. The diagram illustrates the effect of temperature on specific gravity and hence predicted fat content.

Conclusion

The density and specific gravity of fat and lean beef have maximum values at around 5°C. The changes in both due to increasing the temperature above 5°C have been measured under controlled conditions and the coefficients of cubical expansion over the range 5–30°C determined. The results were obtained using samples at atmospheric pressure cut directly from beef rump and no attempt was made to extrude air from the specimen, as in the method of Whitehead (1970) although coating the specimen with polyurethane varnish eliminated fluid losses.

The value of the thermal coefficient of cubical expansion of lean beef in the range 5–30°C of $3{\cdot}75 \times 10^{-4}\ °C^{-1}$ is not in any way anomalous as it corresponds roughly with a mixture of water with 25% organic substances whose coefficients are likely to be in the range $5–8 \times 10^{-4}\ °C^{-1}$. When combined with a value for beef fat of $15{\cdot}3 \times 10^{-4}\ °C^{-1}$, it appears that a beef carcass can vary in specific gravity by as much as 0·01–0·02 over the temperature range likely to be encountered in practice, unless appropriate

precautions are taken. It has been shown that if carcass specific gravity is used as a measure of carcass composition, the neglect of the dependence of specific gravity on temperature may lead to appreciable errors in the predicted composition. As an extreme example, a 10°C change in carcass temperature could alter the predicted % fat by as much as 6%. It is therefore suggested that whilst errors from other sources (e.g. variations in muscle/bone ratio, neglect of voids in the carcass) may be of importance, some of the variation in % fat unexplained by carcass specific gravity measurement may be associated with differences in experimental temperatures.

Acknowledgments

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Some aspects of the quality of carrots on different soil types

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Summary

Carrots are becoming an increasingly important crop in Ireland. High yields can be obtained, particularly on peat soils, but little information is available, as yet, on crop quality.

Three carrot cultivars were grown in peat and mineral soil in 1969 and 1970 and samples of each were harvested three times in 1969 and twice in 1970 at 2-week intervals. Chemical analyses showed that cultivars grown in mineral soil had higher levels of carotene, alcohol insoluble solids (AIS) and dry matter (DM) in all harvests than those grown in peat; values for shear were also generally higher. Contents of reducing sugar for carrots from mineral soil were higher in 1969 but lower in 1970.

Frozen samples from the 1969 experiments were cooked by a standard procedure and were presented to a taste panel. Those grown in mineral soil were rated higher for flavour and softer for texture than peat grown samples. Shear values also showed that both fresh and frozen carrots grown in peat were generally softer when raw, but firmer when cooked than those grown in mineral soils. No taste panels were carried out on samples in 1970, but shear values on cooked carrots showed that the unusual texture change observed in 1969 did not occur to the same extent in 1970.

Introduction

The carrot acreage in Ireland has risen from 1174 acres in 1961 to about 3000 acres in 1970. At the present time over a quarter of the total crop is processed and this figure is likely to increase by a large amount over the next few years. About 70% of the processed crop is dehydrated, the remainder being frozen and canned. Recent developments in production on peat have given yields in excess of 20 tons per acre and the potential for the development of low-cost, large-scale production on peatland is vast.

Because of the higher yields obtained in peat in comparison with mineral soil, it might be expected that quality of carrots grown in peat would be lower than that of

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roots grown in mineral soil. The present study investigates the carotene, reducing sugar and dry matter contents and shear values of three carrot cultivars grown on both peat and mineral soils. These chemical parameters are correlated with organoleptic response. Time of harvest and effect of carrot size on composition were also studied.

Materials and methods

Sampling and harvesting

Samples of carrot cultivars Nantes 1003, Amsterdam 5558 and Sweetheart, grown in peat and mineral soils were each harvested at random on three separate dates at 2-week intervals in the 1969 season. In 1970 the cultivars Nantes 1003, Touchon and Sweetheart were tested and were harvested twice. The number of days from sowing to harvesting in peat and mineral soil was the same for any particular cultivar. There were three field replicates in the experiment. Carrots for analysis were graded into large, medium and small on a weight basis in the 1969 season.

Chemical analysis

Subsamples of ten roots were washed, trimmed and rapidly comminuted in a Boku High Speed Mincer.

Dry matter (DM). 10 g of the chopped material were dried in an oven at 70°C and a vacuum of 560 mmHg to constant weight and the dry matter content calculated.

Carotene content. Carotene was extracted from a 5 g sample of comminuted material with successive portions of a 2 : 3 (v/v) mixture of acetone and petroleum ether (40–60°C). After extraction the extracts were made up to 200 ml and the carotene estimated on a Bausch and Lomb spectrophotometer at 430 mμ.

Reducing sugar and alcohol insoluble solids (AIS). 10 g samples of the comminuted material were canned in 80% aqueous alcohol. At a later date the AIS material was collected by filtration and dried in a vacuum oven at 70°C and 560 mmHg. Purity of the AIS material was tested by further boiling in a fresh portion of 80% alcohol. Reducing sugar was measured on the filtrate by the Schaffer Somogyi Method (Laboratory Method Sheet, 1967). In the 1970 season the AIS content of the roots was not measured.

Hydrolysis of AIS material. Fractionation of AIS material (2g) from both cooked and fresh carrots into pectic substances, hemicelluloses and cellulose was carried out using 0·01 M sodium hydroxide followed by 2 M sodium hydroxide according to the procedure used by Collins (1965) on apple AIS. The fractions were collected by filtration and dried in a vacuum oven at 70°C and 560 mmHg to constant weight.

Shear measurement

Ten roots were washed, trimmed and diced. 50 g samples were sheared in triplicate on a shear press fitted with a 5000 lb proving ring and maximum force reading dial.

In a separate experiment on the effect of the carrot skin on texture, shear values for dice from carrots with skin attached were compared with values for dice from carrots with skin removed. Only peripheral dice which had skin attached were used.

Shear estimations were also made on cooked carrots (skin removed) using 50 g samples. The readings were taken on 'warm' carrots immediately after cooking.

Evaluation of cooked carrots

Because of different harvest dates it was necessary to blast freeze carrots for subsequent taste panel analysis. Samples of the diced, cooked carrots were evaluated for flavour and texture by a trained, six-member taste panel. The dice were all obtained from medium-sized carrots. All samples were cooked by adding 300 g of frozen diced carrot to 570 ml of boiling water in a beaker on a hot plate. Skin was removed from the roots before cooking. After cooking gently for 10 min the samples were collected in strainers and presented for evaluation. The panel members were instructed to rate samples as better or worse than standard in flavour, and firmer or softer than standard in texture, using a five-point hedonic scale. The cultivar Amsterdam, grown in mineral soil, was used as standard (score 0) and also as a coded sample. 50 g samples of the cooked dice were sheared and the results were compared with the taste panel evaluation. Samples from the 1970 trials were not submitted for organoleptic evaluation.

Histological examination

The specimens were prepared by paraffin sectioning according to standard microtechnical methods (Johansen, 1940). Sections were cut at 10–15 μ and stained with safranin–light green (Gurr, 1965).

Results

Evaluation of fresh carrots

The results (Table 1) show that peat grown carrots had significantly lower levels for DM, carotene, reducing sugar, shear and AIS than those grown on mineral soil. All levels, except reducing sugar, increased with successive harvests from roots grown on mineral soil; reducing sugar fell progressively. For peat grown samples only AIS and carotene increased with successive harvests while reducing sugar, DM and shear values increased between the first and second harvests but fell again between the second and third (Table 1).

In 1970 peat grown roots had also lower levels of DM, carotene and shear than those from mineral soil, but reducing sugar contents were slightly higher (Table 2). In contrast to results obtained in 1969, DM levels remained static in peat grown roots and fell in mineral grown roots in the second harvest; shear values also decreased between the first and second harvests.

TABLE 1. Values for DM, carotene, reducing sugar, shear and AIS (means of three cultivars and three field replicates) at three harvest dates on peat and mineral soil (1969)

	Harvest 1			Harvest 2			Harvest 3		
	Peat	'*t*' test	Mineral	Peat	'*t*' test	Mineral	Peat	'*t*' test	Mineral
DM (%)	9·70	***	13·76	10·36	***	14·80	9·66	***	15·18
Carotene (mg/100g fresh weight)	6.82	***	15·46	8·42	***	18·89	10·44	***	20·89
Reducing sugar (%)	2·67	***	3·63	2·70	***	3·18	2·04	***	2·59
Shear (lb)	1226	***	1367	1274	***	1388	1262	***	1408
AIS (%)	3·88	***	5·97	4·14	***	6·09	4·22	***	6·22

*** Significant ($P = 0{\cdot}001$).

It should be noted that data in Tables 1 and 2 are means for three cultivars; however, figures for each individual cultivar were similar to the means in the Tables except for Nantes which had a much lower carotene content in both soil types in 1969 and only carotene increased with successive harvests. In 1970 roots of Nantes from peat soil were slightly firmer than those from mineral soil at the first harvest.

Large carrots grown in peat had higher levels of DM, carotene, shear and AIS and lower levels of reducing sugar than medium or small-sized roots grown in the same soil type. However, only some of the values were significantly different. In contrast, small roots grown in mineral soil had higher levels of DM and shear than medium or large-sized roots.

TABLE 2. Values for DM, carotene, reducing sugar and shear (mean of three cultivars and three field replicates) at two harvest dates on peat and mineral soil (1970)

	Harvest 1			Harvest 2		
	Peat	'*t*' test	Mineral	Peat	'*t*' test	Mineral
DM (%)	10·30	***	15·30	10·32	*	13·81
Carotene (mg/100 g fresh weight)	7·31	***	16·20	8·90	**	16·80
Reducing sugar (%)	3·19	N.S.	2·97	3·05	*	2·62
Shear (lb.)	1333	***	1448	1267	**	1333

* Significant ($P = 0{\cdot}05$). ** Significant ($P = 0{\cdot}01$). *** Significant ($P = 0{\cdot}001$). N.S., Not significant.

Evaluation of cooked carrots

Two sets of taste panels were carried out on cooked carrots in 1969, i.e. Amsterdam and Sweetheart (first harvest) from both soil types (Table 3) and Sweetheart and Nantes (third harvest) from both soil types (Table 4). The results show (Tables 3 and 4) that carrots grown on mineral soil were preferred for flavour while those grown on peat soil had a firmer texture after cooking than the samples from mineral soil. Reasonable correlation was obtained between panel response and sugar and shear values in the panel on the third harvest (Table 4), but in the panel on the first harvest the correlation coefficient between panel flavour response and sugar content was small (Table 3).

TABLE 3. Relationship between panel scores for flavour and texture on cooked carrots[1] (first harvest) and the sugar content and shear values of the raw and cooked samples respectively

Sample	Panel score[2] (flavour)	Reducing sugar (%) raw carrots	Panel score[2] (texture)	Shear (lb) (50 g cooked carrots)
Amsterdam (P)[3]	−1·25	2·22	+0·42	213
Amsterdam (M)	0	3·35	−0·75	138
Sweetheart (P)	−1·33	2·58	+1·25	231
Sweetheart (M)	+0·50	2·62	+0·08	156
Amsterdam (M, coded standard)	+0·17	3·14	−0·17	177
Correlation coefficient (rank)		0·50		0·85
F-test (Panels) df = 1, SE = 0·08	N.S.		N.S.	
F-test (Samples) df = 4, SE = 0·13	***		**	

[1] Carrots frozen before cooking.

[2] Mean score of twelve estimations: two panels × six tasters, scoring system: +2, +1 = better in flavour or firmer in texture than standard; −1, −2 = poorer in flavour or softer in texture than standard; 0 = same as standard in flavour and texture.

[3] P = peat, M = mineral.

Since the texture readings reversed on cooking, i.e. peat grown carrots were softer before cooking, but firmer after cooking than their mineral soil counterparts, another experiment was carried out where fresh carrots were sheared, with and without skin, and were cooked without prior freezing. The texture change again took place (Table 5), and peat grown carrots were firmest after cooking while they were softest before cooking except in the case of Nantes, where peat grown carrots were slightly tougher than the mineral grown roots even before cooking.

TABLE 4. Relationship between panel scores for flavour and texture on cooked carrots[1] (third harvest) and the sugar content and shear values of the raw and cooked samples respectively

Sample	Panel score[2] (flavour)	Reducing sugar (%) raw carrots	Panel score[2] (texture)	Shear (lb) (50 g cooked carrots)
Sweetheart (P)[3]	−1·08	2·16	+1·92	273
Sweetheart (M)	0	2·60	−0·42	126
Nantes (P)	−0·83	2·21	+0·08	183
Nantes (M)	+0·50	2·45	−0·83	90
Amsterdam (M, coded standard)	+0·50	3·14	+0·42	126
Correlation coefficient (rank)	0·83		0·83	
F-test (Panels), df = 1 SE = 0·16	N.S.		N.S.	
F-test (Samples), df = 1, SE = 0·25	***		***	

[1] Carrots frozen before cooking.
[2] Mean score of twelve estimations: two panels × six tasters.
[3] *P* = peat, M = mineral.

TABLE 5. Shear values for carrots[1] cooked directly without freezing and for uncooked carrots with and without skin (1969)

Cultivar and soil type[2]	Shear (lb)			
	Uncooked (skin on) (50 g) I	Uncooked (skin off) (50 g) II	'Skin toughness' (difference) I − II	Cooked (50 g)
Amsterdam (P)	924	894	30	210
Amsterdam (M)	1158	966	192	162
Nantes (P)	1086	1050	36	204
Nantes (M)	1068	990	78	150
Sweetheart (P)	912	912	0	252
Sweetheart (M)	1146	1092	54	168

[1] Carrots of harvest three stored for two weeks at 4–5°C.
[2] P = peat, M = mineral soil.

Carrots grown on mineral soil had tougher skin (Table 5). In the 1970 experiments the 'reversal' in texture on cooking was only observed for the cultivar Touchon (Table 6). Roots of Nantes from peat soil were slightly firmer before cooking but much firmer after cooking than those from mineral soil. In the case of the cultivar Sweetheart no 'reversal' in texture between roots from the two soil types took place on cooking (Table 6).

TABLE 6. Shear values for uncooked and cooked carrots (1970)

Cultivar and soil type[1]	Shear (lb)	
	Uncooked (skin off) (50 g)	Cooked (skin off) (50 g)
Touchon (P)	984	183
Touchon (M)	1026	138
Nantes (P)	999	207
Nantes (M)	993	129
Sweetheart (P)	846	96
Sweetheart (M)	999	164

[1] P = peat, M = mineral soil.

Fractionation of AIS material

Fractionation of AIS material from uncooked carrots on peat and mineral soils gave almost the same levels of pectin, cellulose and hemicellulose when the results were expressed on a percentage basis. However, the recovery figure for peat grown samples (99%) was higher than for those grown in mineral soil (90%). The same pattern of AIS fractionation was obtained for the cooked carrots with a recovery figure of 99% for the samples grown in peat and only 83% for the samples from mineral soil.

Histological examination

Carrots from the 1969 trials grown in mineral soil had a pronounced ring of xylem tissue around the outside of the root. In contrast the samples grown in peat had fewer xylem vessels in the outside but more vessels in the internal part. Vessels in the carrots from peat were also larger than those in roots from mineral soil. No histological examination was made on roots from the 1970 experiments.

Discussion

The results show (Tables 1 and 2) that peat grown carrots were significantly lower in all the constituents measured at each harvest date except for reducing sugar in the

1970 season. When the results for Amsterdam, Sweetheart and Touchon were expressed individually, the pattern was similar, except that levels for some of the parameters were not signficantly lower in the three cultivars. Nantes, however, was exceptional to some extent in both seasons. The large differences in chemical composition between roots from the two soil types could have major implications for processing. Carotene levels are so widely different that any processed product comprising roots from the two soil types might not be acceptable because of colour differences. The lower dry matter content obtained for peat grown roots would also have implications for the dehydration process but might be offset to some extent by the higher yield obtained on the peat soil. Another obvious difference between roots grown on the two soil types was the effect of date of harvest on DM values; in 1969, DM values for roots from mineral soil increased with each successive harvest but for peat soil these values rose between the first and second harvest and fell between the second and third. The differences found in chemical composition may be due to the different rates of growth on the two soil types. The roots grown in peat were larger and had a much better supply of water because of the greater water holding capacity of the peat soil. However, further research into new cultivars for growing in peat is necessary in order to find cultivars which have a quality similar to those grown in mineral soil. This is most important since the peat is an excellent uniform growing medium and provides suitable conditions for mechanical harvesting. In addition, there are many areas of peatland in Ireland suitable for large scale intensive production.

The taste panels for flavour and texture showed that sugar content had a marked bearing on flavour and in general roots with a high sugar content were highly rated. Rank correlations between panel response and sugar and shear values were reasonably good, especially in the panel on the third harvest (Table 4).

The different rates of softening during cooking of roots from the two soil types is noteworthy. The shear values for peat grown samples were significantly lower than those for samples grown in mineral soil, before cooking (Tables 1 and 2); however, in most cases the latter softened more during cooking resulting in the peat grown samples being the firmest. The differences in texture were readily identified by the taste panel (Tables 3 and 4). It was thought that this unusual change in firmness might have been due to differences in skin toughness, since skin was removed in the cooked carrots but not in the fresh ones. The composition of AIS material or the effect of freezing might also have contributed to the texture change. However, the results (Table 5) show that roots from mineral soils had tougher skins, although even without skins they were still tougher than those from peat. The freezing process itself probably caused some of the texture change but since the change also took place in cooked carrots which were not frozen (Table 5) this suggests that other factors are also involved.

The texture change could not be explained on the basis of the AIS since both raw and cooked carrots from the two soil types had approximately the same percentage of

pectins, celluloses and hemicelluloses in the AIS. More detailed studies, however, are required into the nature and location of the pectin.

Samples from the 1970 experiments were also cooked and in the case of the cultivars Touchon and Nantes the roots from peat soil did not soften nearly as much as those from mineral soil during cooking, which endorses the results obtained in 1969. In the case of the cultivar Sweetheart, however, roots from mineral soil were firmest before and after cooking, which contrasts with the 1969 result.

These data suggest that a number of complex factors seem to affect the softening of carrots during cooking. Mineral grown roots may be firmer than peat grown samples because of the more extensive ring of xylem tissue in the former. However, this 'outside' xylem ring might be more susceptible to breakdown during cooking than the internal xylem tissue. Since roots grown in peat seem to contain more internal xylem tissue than those grown in mineral soil this may affect the texture on cooking to some extent. It was not possible to carry out a histological examination of the roots in 1970, hence no information is available concerning the nature of the xylem tissue of the cultivar Sweetheart which did not follow the softening pattern obtained for the other cultivars in 1969 and 1970.

Rainfall may also contribute to the unusual textural behaviour. Both the summers of 1969 and 1970 were dry and carrots on mineral soil were growing under water deficient conditions when compared to peat grown roots since peat is a much better reservoir of moisture. This probably caused the higher dry matter figures in mineral grown roots and to some extent the firmer texture. Availability of water may also have influenced the location of xylem tissue in roots from the two soil types.

Conclusions

Carrot cultivars grown in peat soil had lower levels of DM, carotene, shear and AIS than those grown in mineral soil. This stresses the need for further research on cultivars which are suitable for growing in peat.

Roots grown in mineral soil were in general firmer before cooking but softer after cooking than those grown in peat soil. The phenomenon may be due to differences in the nature of the xylem tissue from roots grown in the two soil types.

Availability of moisture during the growing season may affect the composition, texture and vascular system.

Acknowledgments

We acknowledge the technical assistance of Mr P. Walshe and Miss P. Newburn.

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J. Fd Technol. (1971) **6**, 403–408.

Storage of gooseberries in plastic bags

A. KENNY

Summary

Storage of gooseberries in sealed polythene bags was shown to give useful extension of fresh fruit life without detrimental effects on fruit quality. Quality was assessed by analyses for firmness, colour, acidity and reducing sugars and by tasting of random samples.

At 0–1°C over 90% of the fruit in 7 lb lots in sealed bags was sound after 8 weeks, while in the corresponding 28 lb lots the percentage of sound fruit was in the range 81–88% . Control of the atmosphere in the bags was difficult and this aspect would limit commercial application of the technique. No ethylene was detected in any of the sealed bags.

Introduction

The supply of gooseberries in Britain and Ireland has now outstripped demand, so that new processing and fresh market outlets are needed. A cheap method that would prolong storage life and permit out-of-season sales of the fresh fruit would, therefore, be an asset.

Tomkins (1962, 1965) has reviewed the information on packaging of fruits and vegetables in plastic films. Most films are two to five times more permeable to carbon dioxide (CO_2) than to oxygen (O_2). Tomkins (1965) found that in sealed plastic bags the fall in concentration of O_2 is about twice the rise in concentration of CO_2.

The storage of gooseberries at various temperatures in air and in mixtures of air and CO_2 has been studied by Smith (1966). He found that at 0°C hard green gooseberries could be stored for three to four weeks and suggested that no benefit would be obtained by the additional use of controlled atmosphere.

The present investigation was aimed at determining the usefulness of storage in sealed plastic bags, the necessity of controlling the atmosphere in the bags, and the effects of such storage on fruit quality.

Materials and methods

Gooseberries (cv. Careless) harvested in hard green condition were used in the experiment.

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Packaging

At one day post-harvest the fruit was weighed in 7 lb and 28 lb lots into 400-gauge polythene bags of dimensions 45 cm × 30 cm and 84 cm × 45 cm, respectively.

Storage treatments

Storage was carried out in:

(1) open bags (control treatment);

(2) sealed bags (sealing being done by twist-tieing at the neck);

(3) sealed bags containing sachets of vermiculite which had been soaked in alkaline potassium permanganate (2 N-$KMnO_4$/0·1 N–NaOH) solution for absorption of ethylene; and

(4) sealed bags containing sachets of soda-lime, to absorb carbon dioxide, evacuated and filled with nitrogen to give an initial atmosphere of, approximately, 99% nitrogen + 1% oxygen.

The above treatments were tested at ambient (12–20°C) and at cold store (0–1°C) temperatures.

Control of Atmosphere

A gas sampling port was fitted in a side of each sealed bag. This was done by punching a circular hole in the polythene. A plastic tube of twice the hole diameter was passed through from the inside and the resulting elevated close-fitting lip of polythene was sealed onto the tube with adhesive tape. Closure of the tubing orifice with a suba-seal rubber stopper or a rubber vaccine cap permitted convenient sampling or changing of the atmosphere through a wide bore hypodermic needle.

The sealed bag atmospheres were analysed at frequent intervals for CO_2 and O_2, using an Orsat apparatus, and for ethylene, using the Kitagawa gas detection apparatus with tubes no. 108b (D. A. Pitman Ltd.). Holes were punched in the polythene and bags were also vented as found necessary to maintain concentrations of O_2 not less than 1% and CO_2 not greater than 10%.

Fruit quality assessment

Colour was measured on samples of macerated whole berries using the Hunter Colour Difference Meter with a 2-in diameter specimen port. Firmness was measured as the force (lb) required to shear a 100 g sample of whole berries, using the Allo-Kramer Shear Press (with universal test cell, 5000 lb proving ring, and piston downstroke of 25 sec). Acidity was determined by direct titration to phenol phthalein endpoint of juice expressed from a macerate. Reducing sugar content of the juice was determined by the Shaffer-Somogyi copper reduction method (Laboratory Method Sheet, 1967).

Random samples were tasted by two observers to check for development of off-flavours, such as those of alcoholic fermentation.

Results and discussion

Ambient temperature storage

Storage at 19–21°C gave poor results. The fruit in open bags was rotten after 10 days. In the sealed bags CO_2 accumulated so rapidly that it was difficult to keep its concentration below 30%, and extensive fermentation had set in within two weeks. Thus, storage at ambient temperatures in sealed bags would seem to be of no commercial value.

Cold storage

The fruit was sorted and analysed at *c.* monthly intervals. Sorting results (Table 1) showed that, after 8 weeks; (a) Sealed bags gave better ($p = 0{\cdot}001$) preservation than open bags. (b) Fruit had kept better in 7 lb than in 28 lb lots after 8 weeks in sealed bags; however, the difference was significant ($p = 0{\cdot}05$) only in the case of treatment 3 ($KMnO_4$/NaOH). After 11 weeks the fruit kept better in the 7 lb lots in both open and sealed bags. (c) Treatments 3 ($KMnO_4$/NaOH) and 4 (sodalime/nitrogen) had no advantage over the unmodified sealed bag treatment 2.

TABLE 1. Weight of sound fruit as percentage of total fruit weight

Storage time	Lots	Treatments			
		1 (control)	2	3	4
8 weeks	7 lb	68·4	91·9	91·7	80·9
	28 lb	70·4	81·1	88·1	77·4
		F-test	SEM (Standard Error of Mean)		
	Treatment (T)	***	1·17		
	Lot weight (W)	*	0·83		
	Interaction T × W	*	1·65		
11 weeks	7 lb[1]	26·1	51·3	42·9	60·8

[1] In the 28 lb lots after 11 weeks the fruit was substantially unsound.

Analytical results (Table 2) generally indicated that fruit composition and quality was not greatly affected by sealed bag storage.

After 5 weeks the fruit in open 28 lb bags was less firm ($p = 0{\cdot}05$) than that in sealed 28 lb bags, while in the 7 lb bags the difference was not significant. Differences in

TABLE 2. Analysis of stored gooseberries

Treatment		Greenness (Hunter meter units[1])	Shear value (lb force)	Acidity[2]	Reducing sugar[3]
		Original fruit at 1 day post-harvest			
Bulk sample		10·9	684	16·3	3·69
		After 5 weeks at 0–1°C			
1 (control)	7 lb	8·6	624	15·2	3·74
	28 lb	7·8	456	14·5	3·41
2	7 lb	8·3	627	14·5	3·44
	28 lb	8·7	603	14·3	3·44
3	7 lb	8·5	594	15·0	3·50
	28 lb	8·7	606	14·5	3·55
4	7 lb	7·6	588	15·1	3·76
	28 lb	7·9	605	14·8	3·81
	Treatment (T)	ns	*	ns	ns
F-test	Lot weight (W)	ns	**	**	ns
	Interaction T × W	ns	*	ns	ns
	T	0·21	13·0	0·12	0·07
SEM	W	0·15	9·2	0·08	0·05
	T × W	0·30	18·3	0·17	0·10
		After 8 weeks at 0–1°C			
1 (control)	7 lb	8·8	396	13·1	3·59
	28 lb	7·5	450	12·6	3·65
2	7 lb	8·9	435	12·3	3·35
	28 lb	6·5	378	12·0	3·19
3	7 lb	8·9	414	13·0	3·37
	28 lb	7·5	408	12·6	3·41
4	7 lb	8·4	453	13·0	3·33
	28 lb	5·8	384	12·0	3·15
	T	ns	ns	ns	ns
F-test	W	***	ns	*	ns
	T × W	ns	ns	ns	ns
	T	0·30	13·8	0·17	0·09
SEM	W	0·21	9·8	0·12	0·07
	T	0·42	19·6	0·23	0·13
		After 11 weeks at 0–1°C			
1 (control)	7 lb	5·7	402	8·8	3·28
2	7 lb	5·1	363	9·8	2·89
3	7 lb	6·3	360	9·3	3·17
4	7 lb	4·8	375	8·6	3·25
F-test		ns	*	*	ns
SEM		0·23	5·2	0·13	0·10

[1] Greenness on a scale of 0 to 100.

[2] Ml 0·1N–NaOH per 5·0 ml juice.

[3] As g glucose/100 ml juice.

firmness and in acidity between 7 lb and 28 lb lots were significant ($p = 0{\cdot}001$) for the open bag treatment, but not for sealed bags.

After 8 weeks there were no significant firmness differences, but acidity and greenness were higher ($p = 0{\cdot}05$ and $0{\cdot}001$, respectively) in the 7 lb than in the 28 lb lots with all treatments.

After 11 weeks there was no conclusive quality difference between treatments for the surviving sound fruit in 7 lb bags. Fruit in the open bags was firmer ($p = 0{\cdot}05$) than that in all sealed bags but was, conversely, less acid ($p = 0{\cdot}05$) than that in sealed bags of treatments 2 and 3. The juice expressed from fruit of treatments 2 and 3 was clear and free of floc. This contrasted with the normal cloudiness of the juice from the other treatments, and, presumably, was due to pectin degradation during storage.

Flavour of the fruit, as checked by tasting random samples, did not seem to be adversely affected at any stage by the sealed bag storage.

Control of atmospheric composition in sealed bags

Obviously, precise control of atmospheric composition was not practicable. Control was necessarily confined to attempting to keep the CO_2 concentration as near as possible to 10% by perforating and/or venting the bags in conjunction with frequent measurement with the Orsat apparatus. The latter was important as it was found that even at 0–1°C, CO_2 accumulated quite rapidly in the sealed bags.

With regard to ethylene, no trace of this compound was found in any of the sealed bags, using the Kitagawa tubes No. 108b which have a declared measuring range of 0·5–100 ppm of ethylene. No information on ethylene levels over stored gooseberries could be found in the literature.

Conclusions

These studies indicate that storage in polythene bags could be employed to give useful extensions of gooseberry fresh life. However, practical application of the method would be governed by the availability of a means for determining atmospheric composition.

Further investigation would be necessary to determine optimum conditions of bag size, polythene thickness and lot weight.

Polythene bags have the advantages that (a) they are cheap and readily available, (b) they are light and flexible, (c) they are transparent, and (d) they facilitate control of the enclosed atmosphere.

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A method for measuring the firmness of whole loaves of bread

E. C. BISHOP AND J. J. WREN*

Summary

The firmness of whole loaves of bread, unsliced or sliced, can be measured by using an Instron Universal Testing Machine in a constant deformation method which simulates the purchaser's squeeze-test. A loaf is mounted on a frame and subjected to a small, recoverable compression with a pivoted bar on the oven-spring line. The method is rapid and facilitates the plotting of firming curves or the calculation of rate constants.

Introduction

Many methods have been devised for measuring the firmness and rate of firming of bread. Most of those in common use, for example the Baker Compressimeter (Platt & Powers, 1940), the Panimeter (Hintzer, 1951) and the Cone Indenter (Cornford, Axford & Elton, 1964) are applied to the crumb only. However, the purchaser estimates freshness by squeezing the whole loaf or indeed, if the loaf is sliced and wrapped, the whole package. A whole-loaf 'Squeezometer' was therefore designed by Hill & Dalby (1961) and subsequently modified by Hlynka & Von Eschen (1965). This instrument is quick and relatively easy to use, measuring the deformation produced by a chosen load. The load must be great enough to give a perceptible deformation with a stale loaf but light enough to avoid damaging the structure of a very fresh loaf; this compromise is difficult to achieve.

A more serious disadvantage of the Squeezometer method is that it uses a constant load whereas the customer does not and, in fact, tends to use a constant deformation. Constant-deformation methods are preferable (see e.g. Crossland & Favor, 1950).

Here we describe the use of an Instron Universal Testing Machine (Bourne, Moyer & Hand, 1966; Bourne, 1967; White, 1970) in a constant-deformation method that has been successfully applied to a large number of standard and laboratory-baked loaves. Compression has been designed to simulate the pressure of the consumer's fingertips and thumbtip on or near the oven-spring line as the hand extends over the top of a loaf. The compression is too slight and too rapid to cause a significant permanent deformation, and so measurements can be made at several times during a period of storage. The

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method is described for use with 28 oz round-topped loaves, but is applicable to other sizes and to loaves baked in lidded pans.

Apparatus

1. Instron Universal Testing Machine, table model TMM (Plate I).
2. Steel compressor bar, 75 mm long and 5 mm thick, with rounded lower edge and corners, joined by a central pivot to a vertical rod which is screwed into the load cell (see Plate I and Fig. 1). The pivot allows the bar to move freely in a vertical plane about 15° either way from horizontal.

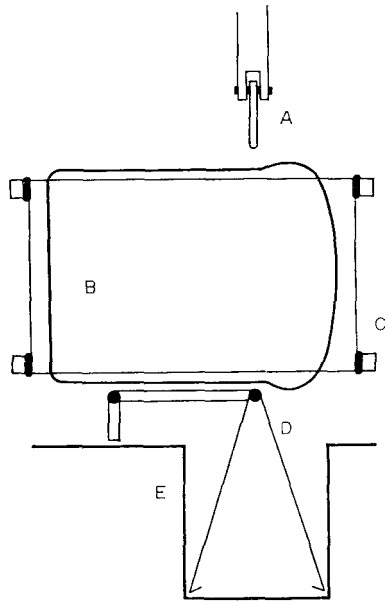

FIG. 1. End view of compressor bar (A), loaf (B), end plate (C), loaf support (D) and well of Instron (E).

3. Loaf support made from sheet aluminium fixed to steel rods (see Plate I and Fig. 1).
4. Two end-plates consisting of oblong plastic sheets, each 160 × 75 mm, with protrusions at the corners on which rubber bands are attached.

Method

1. Invert the CCTM load cell, mount it on the cross-head and attach the compressor bar, and mount the loaf support in the well of the cross-head assembly.
2. Check the chart calibration with a standard weight (2 kg for full-scale deflection).
3. Set the Instron controls:

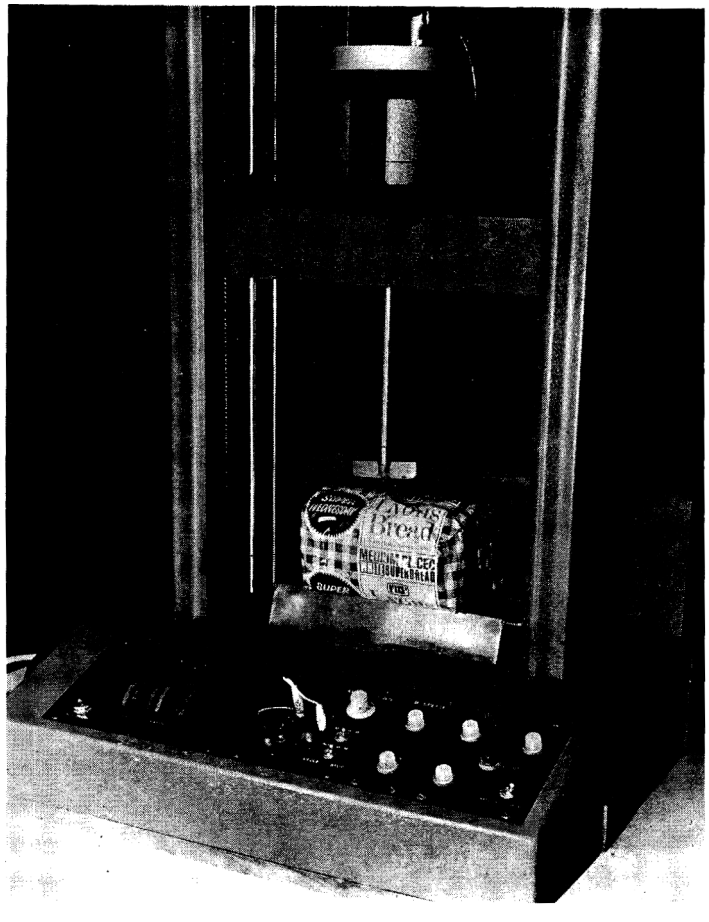

PLATE I. Loaf mounted on loaf support in Instron (recorder not shown) with compressor bar and load cell above.

(*Facing p.* 410)

Cross-head speed *A*	2 cm/min
Cross-head speed *B*	20 cm/min
Chart speed	20 cm/min
Chart control	EXT. STOP
Load cycle left limit	<0
Load cycle right limit	1 chart division
Patchboard: LOAD MAX.	UP at *B* speed
Patchboard: EXTENSION MIN.	DOWN at *A* speed
Max. and min. switches	STOP
Control	AUTO

4. Raise the crosshead to allow just enough room for inserting a loaf and set:

Extension cycle control	MIN. 0·0 cm

5. Mount the loaf between the end-plates and join these with four rubber bands of thickness *ca.* 1/16 in. and unstretched circumference *ca.* 5 in. (This step is omitted for non-sliced loaves or for sliced loaves with tight, secure wrappers.)

6. Lay the loaf on its side, centrally, with the oven-spring line resting on the apex of the loaf support.

7. Start the cross-head DOWN at *A* speed.

8. When it stops (automatically), re-set the controls:

Patchboard: EXTENSION MAX.	UP at *B* speed
Patchboard: EXTENSION MIN.	DOWN at *A* speed
Max. switch	CYCLE
Extension cycle control	MAX. 1·50 cm

9. Start the cross-head DOWN at *B* speed.

10. Read from the chart the peak force reached in the compression cycle.

Discussion

On each of six occasions twelve medium-sliced loaves were taken from a commercial bakery on leaving a wrapping machine; they were tested by the method and then stored and re-tested at daily intervals. The coefficients of variation of the results ranged from 4·4% to 12·7%. These reflect variations inherent in the loaves more than variability in the method, since a measurement repeated after a short recovery period (5 min) usually agreed within 2%.

Such results were compared by plotting firming curves or—more usefully—by computing the constants *A* and *B* in the empirical equation (Willhoft, 1970)

$$F = At^B$$

in which *F* is the force in g and *t* the age in days. The results of a typical experiment, in which the firming of commercial bread made by conventional fermentation was

compared with that of bread made by mechanical development, are given in Table 1.

Results obtained by the Instron method agreed well with results obtained in consumer research (unpublished experiments).

Free pivoting of the compressor bar is important because it allows the bar to 'seat' itself on the loaf at stage 7 of the method, giving a reproducible datum for the compression (stage 9). The 15 mm compression takes less than 5 sec, and one technician can measure the firmness of fifty to sixty loaves of bread in an hour.

TABLE 1. Firming of twenty-four sliced loaves in wax-paper wrapping, half made by conventional fermentation and half by mechanical development

Age	Peak force F (g) (mean ± S.D.)		
t (days)	Conventional fermentation	Mechanical development	
Experimental results			
0·94	1365 ± 85	1270 ± 105	*
1·90	2060 ± 210	1680 ± 120	***
3·08	2565 ± 360	2090 ± 150	***
3·98	2810 ± 470	2320 ± 190	**
4·83	3160 ± 275	2380 ± 225	***
6·00	3230 ± 315	2465 ± 190	***
Computed values (curve $F = At^B$)			
1	1460	1325	
2	2030	1720	
3	2460	2000	
4	2820	2225	
5	3135	2420	
Correlation coefficients			
	0·99	0·99	
Constants			
	$A = 1460$	$A = 1325$	
	$B = 0·47$	$B = 0·37$	
	$AB = 692$	$AB = 496$	(Rate constants)

*Difference significant at the 5% level
**Difference significant at the 1% level
***Difference significant at the 0·1% level

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The role of extract release volume in a rapid method for assessing the microbial quality of pork and beef

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Summary

Experiments with raw pork and beef confirmed the work of other workers in showing that there was a linear relationship between Extract Release Volume (ERV) and numbers of micro-organisms. It is suggested that the ERV could largely replace conventional bacterial counts as a routine quality-control procedure in the food industry. Low ERV measurements, due to the pre-rigor peak, can be eliminated by repeat-testing after 24-hr storage. No useful association between colour and ERV, or total bacteria, was observed but BSS colours for meat regarded as normal by a panel are given.

Introduction

Conventional laboratory methods for determining the microbial quality of raw meat require 1, 2 or even 3 days for completion, depending on the incubation conditions used for cultures. A general impression of quality can be gained from organoleptic measurements; these are usually personal opinions based on whether or not taste, odour, colour, appearance and tactile response are normal. There may be large differences between such opinions but these can be considerably reduced by taking the mean finding of a trained panel of assessors. A disadvantage of organoleptic measurements is, however, that changes which are observed may not be directly related to numbers or types of bacteria present.

What is required is a simple and rapid test to measure objectively some aspect of meat quality which would relate to the quality as a whole. A useful observation towards this end was made by Jay (1964a) who found that the volume of aqueous extracts (ERV) released by beef homogenate fell with the age of the sample. The fall was related to water holding capacity and it was thought to be caused by changes in the meat protein, due to proteolytic bacterial action. A lowering of the ERV value was also found to be accompanied by a rise in the total numbers of bacteria (Jay, 1964b), a rise in pH (Jay, 1964a), increases in total volatile nitrogen, free fatty acid and odour score (Pearson, 1968a,b,c), a rise in meat swelling (Shelef & Jay, 1964b) and an increase in

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the amino-sugar content of spoiling beef (Shelef & Jay, 1969a). The influence of a rising pH on the lowering of ERV was emphasized by Riedel, Burke & Nordin (1967); these workers also noted that if spoilage occurred in the presence of a dominant lactic-acid bacterial flora, no increase in pH occurred and ERV did not reflect spoilage.

Most workers to date have used minced or ground beef in their experiments although similar findings were observed by Shelef & Jay (1969a) with chicken meat and by Price (1965), Borton, Webb & Bratzler (1968) and Reidel *et al.* (1967) with pork.

In the present situation, frequent consignments of raw, boned meat, mostly beef or pork, are received in large pieces for food processing from abattoirs or boning houses. The usual quality-control practice is to perform a simple, visual inspection and replicate microbiological counts. On many occasions, however, the meat has been already processed by the time laboratory results become available and findings are only useful for retrospective analyses. The purpose of the experiments to be described was to investigate the usefulness of the ERV measurement as a rapid quality control procedure in this situation, and to compare the results with those obtained by conventional microbiological techniques and simple measurement of meat colour.

Experimental

Colour

Each meat consignment was inspected soon after delivery by a panel usually comprising three people, one of whom was a qualified veterinary officer. The meat nearly always arrived slightly chilled from animals slaughtered within the previous 24 hr. If delivered in a deep frozen condition, it was left for 2 or 3 hr at room temperature to soften. A judgement was made about the overall colour, whether it was normal, too light or too dark and at the same time it was compared against a complete range of British Standard paint colour cards.

Microbiology

Samples of muscle were aseptically removed to sterile containers. Each sample included at least three square inches of uncut muscle surface. In the laboratory, the samples were transferred to sterile dishes, and sterile 3 by 1 inch microscope slides were pressed firmly for 5 sec against exposed surfaces of the meat. This sampling technique was used by Thomas (1966) when preparing slides for imprinting on to agar surfaces. Each slide was then placed in a screw-capped jar containing 100 ml sterile quarter-strength Ringer solution and the jars shaken violently for 10 sec. Aliquots of the solution were diluted and 'plate' counts performed by pipetting millilitres of solution in to Petri dishes, adding molten, cooled Plate Count Agar (Oxoid), incubating at 32°C for 48 hr and counting cultures containing 30–300 colonies. Results were expressed as organisms per square inch of meat surfaces.

Storage at different temperatures

In order to determine the influence of different storage temperatures on the results, replicate samples of meat were obtained from animals killed only a few hours previously and some were stored at room temperature (+23°C mean), some at domestic refrigeration temperature (+6°C) and the remainder deep frozen (−18°C). All samples were contained in sterile, screw-capped, aluminium cans of approximately 200 ml capacity. Samples were removed at regular intervals and microbiological and ERV tests performed. Deep frozen samples were rapidly thawed out before testing.

E.R.V.

When slide tests had been taken from the meat samples, the ERV was estimated by a method based on a modification by Pearson (1969) of Jay's original procedure. Fifteen grams of meat, chosen to contain a minimal amount of fat, were blended for 2 min at half speed in the cutter unit of an 'Ato-mix' homogenizer (Measuring & Scientific Equipment Ltd, Spencer Street, London, S.W.1) with 60 ml of pH 5·8 buffer solution (Pearson, 1969). The homogenate was immediately filtered in the prescribed manner for 15 min and the volume of filtrate noted.

Results

Colour

No significant relationship was found between colour and ERV, or colour and bacterial population, except for the general observations that deep frozen meat was often darker than normal and that nearly putrid meat tended to be lighter than normal. Pork, regarded by the panel as being of normal colour, often corresponded to BSS colours 1-023 or 1-024, or a blend of both, or to extremes of colours outside this range but with a mean approximately within it. Beef that was considered to be normal usually corresponded to BSS 1-024, 1-025 or a blend of both.

Storage at different temperatures

Typical results of ERV plotted against age at the various temperatures are shown in Figs. 1 and 2. Curves at 6°C are seen to exhibit an initial upward swing or 'pre-rigor peak'—a phenomenon associated with very fresh meat, not related to bacterial numbers (Pearson, 1968c).

Microbiology/ERV

Figs. 3 and 4 show the correlation between ERV and numbers of micro-organisms for both pork and beef. Results include findings from meat tested soon after arrival and also after storage at room temperature and 6°C, but not from samples stored at 18°C

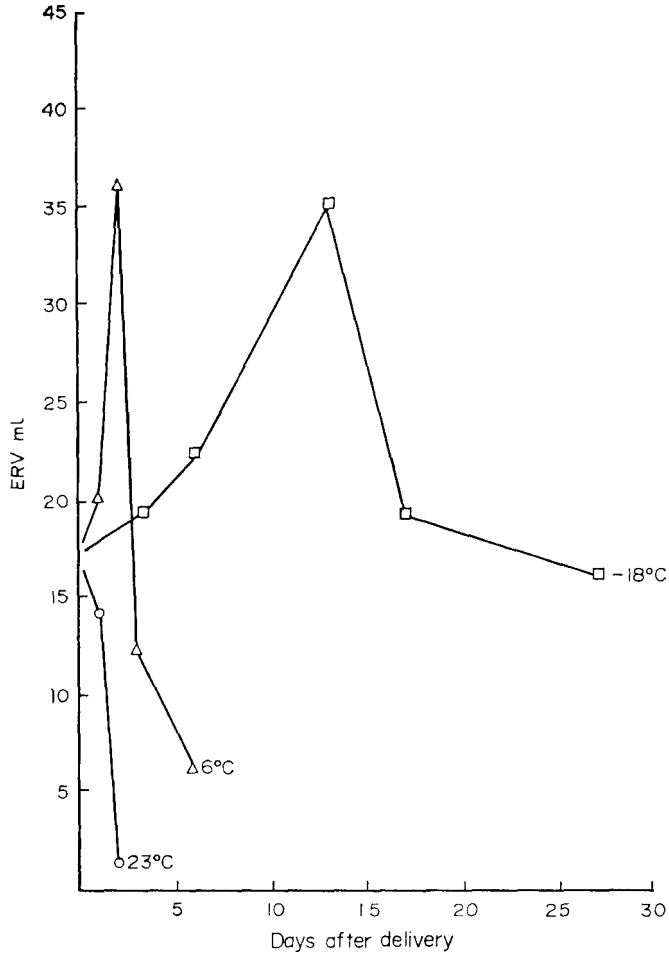

FIG. 1. Extract-release volume (ERV) from pork stored at O, 23°C: △, 6°C and □, −18°C.

below zero. Correlation coefficients were calculated as −0·73 for pork and −0·55 for beef. Standard deviations are 1·10 and 1·17 log organisms/ERV respectively.

Discussion

The present experiments confirmed that there was apparently a linear relationship between ERV and numbers of micro-organisms, although statistically the correlation was not very high, particularly for beef. Reproducibility of the ERV test is considered to be good; Pearson (1969) stated that the coefficient of variation between replicate results (in excess of 10 ml) was about 3% for beef. Microbial counts, however, are

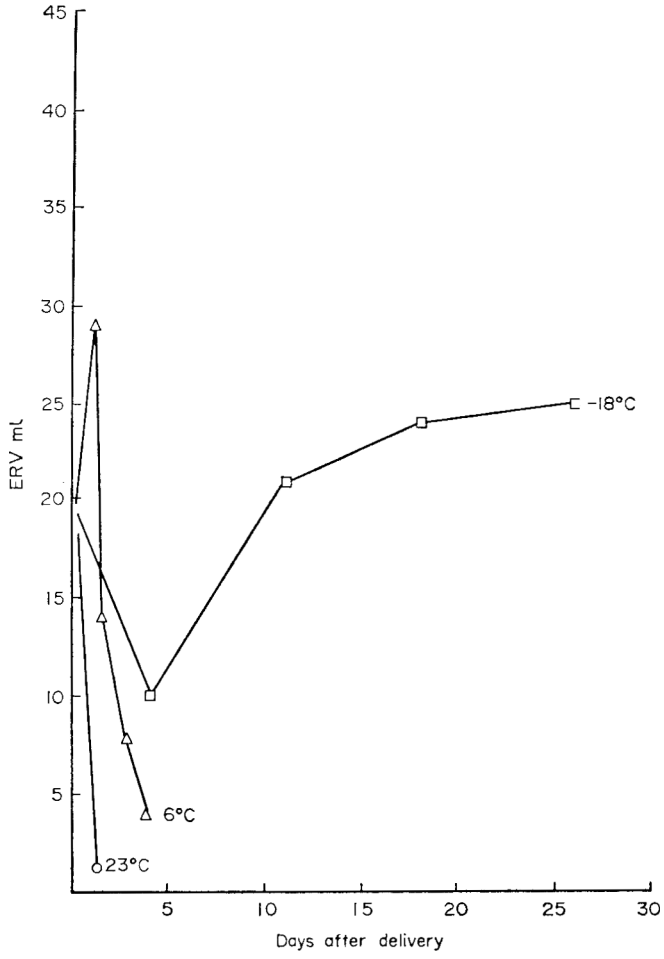

FIG. 2. Extract-release volume (ERV) from beef stored at ○, 23°C: △, 6°C and □, −18°C.

inherently inaccurate; Gibbs & Freame (1965) quoted confidence limits ($P = 0{\cdot}95$) of ± 14 for duplicate colony counts of 100. It is possible that this inaccuracy of microbial method is partly responsible for the rather disappointing statistical findings. Obviously, further research using improved counting techniques is required to determine if the correlation between the two types of test can be improved.

When figures were corrected for differences in technique, the present results compared well with those quoted by Jay (1964b) for beef and Price (1965) for pork. The results obtained by Borton *et al.* (1968) with artificially contaminated pork were slightly higher than those published by the other workers, although they would produce a line roughly parallel to that shown in Fig. 3.

Any attempt to find an 'accept or reject' standard is complicated by the fact that different muscles from the same animal can give different ERV's (up to 25·5% difference, Jay, 1964a, b) and also by the pre-rigor, high peak. Despite these difficulties, most workers seem to agree on a minimum acceptable ERV of 17 ml, based on comparisons with organoleptic measurements. This figure is usually claimed to be the equivalent of approximately log 8·5 micro-organisms per g, which is approximately log 7·1 organisms per sq. in. (Lowis, unpublished). The bacterial numbers corresponding to 17 ml in the present experiments are log 6·3 and 5·7 per sq. in. respectively for pork and beef: the reason why these figures are slightly lower than might have been expected

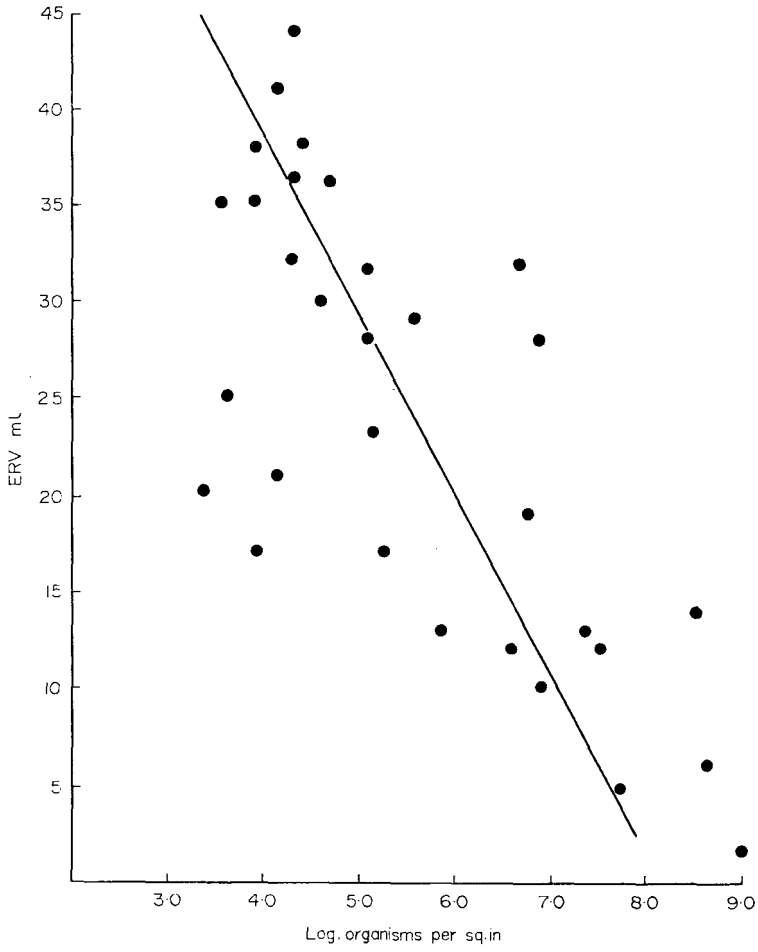

FIG. 3. Correlation between extract-release volume (ERV) and numbers of micro-organisms from pork.

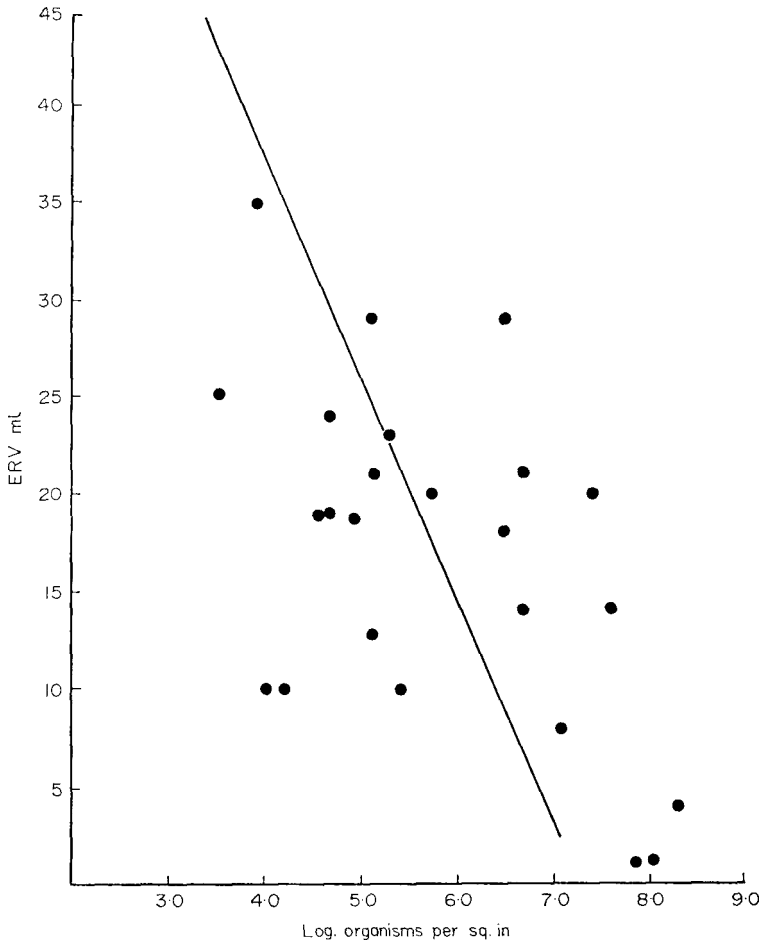

FIG. 4. Correlation between extract-release volume (ERV) and numbers of micro-organisms from beef.

is probably explained by differences in methodology and the degree of error in the bacteriological sampling technique used.

If the ERV is to be used in place of conventional, microbiological techniques, account must be taken of 'false' low results. It is suggested that, if meat on first testing yields an ERV of less than 20 ml, a sample be stored for 24 hr at 6°C and the test repeated. A low, pre-rigor volume will then have increased but a genuine, post-rigor volume will have decreased. This procedure would also help to clarify the phenomenon of a low ERV from naturally high pH meat described by Riedel *et al.* (1967). In the present series of experiments a repeat test, with 24-hr delay would have occurred in less than one quarter of the tests on meat as received. In some instances it would be possible to delay the use of the particular consignment of meat whilst repeat tests are being carried out.

At other times the meat will have been used and the results will only be useful for retrospective analysis.

The storage tests at different temperatures (Figs. 1 and 2) show that microbial proliferation is so rapid at room temperature that there is no early high peak in ERV, as occurs at 6°C. The findings from deep frozen samples are worthy of note in that there is apparently a delayed peak which could correspond to the pre-rigor high point found at 6°C.

It is concluded that the ERV test could be used as an indirect measurement of microbial quality for raw pork and beef, with the proviso that the method may be unreliable for meat stored in vacuum sealed cryovac pouches (Riedel *et al.*, 1967). The dramatic saving of time in presenting at least a provisional quality assessment is of value in the food industry. The confusion over 'false' low results can be overcome by re-testing samples after a further 24-hr storage.

Acknowledgments

Acknowledgment is made to the directors of Ranks Hovis McDougall Foods Limited for permission to carry out this work whilst in their employ, and for permission to publish these findings. I am very grateful to Dr R. Dainty of the A.R.C. Meat Research Institute, Bristol for advice and criticism during the preparation of this paper and to Mr J. Barwood of United Biscuits for help with the statistical analysis.

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Active dried baker's yeast

I. Systems involved in the fermentation of mono- and disaccharides

D. O. BILTCLIFFE

Summary

By choice of nutrients and inhibitors a study of fermentation of mono- and disaccharides by dried baker's yeast has been made. It is concluded that in the development of a maximal fermentation by this yeast at least three systems may be involved.

(a) A system leading to the fermentation of hexose or sucrose in the absence of nutrients other than the sugar.

(b) A system causing an increase in this rate of fermentation of hexose but requiring nutrients.

(c) A system bringing about the induction of maltose fermentation also requiring nutrients.

System (b) probably causes the production of further transport sites. Deterioration of the dried yeast during storage under nitrogen affects the second and third of these systems more than the first.

Introduction

The fermentative behaviour of pressed baker's yeasts in breadmaking and in sugar solutions has been extensively reported (see Bechtel, Geddes & Gilles, 1964, for review). No such detailed reports appear to have been made for active dried baker's yeasts so, as part of a wider scheme of work, an examination in detail of the fermentative behaviour of an active dried baker's yeast has been made.

The first part of these studies concerns the behaviour of the yeast with simple sugars, i.e. fermentable hexoses and disaccharides, and examines the effects of components of the medium on the fermentation of these sugars. As an inhibitor cysteine has been used to indicate the possible nature of some of the changes occurring during a fermentation. Also uranyl ion has been used as an inhibitor to determine the possible role of sugar entry into the yeast cell in these changes.

A consideration arises with dried baker's yeasts which is not pertinent with pressed

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yeasts, namely the effects of deterioration during prolonged storage. Consequently an examination has been undertaken of some of these effects on the fermentation of sugars by samples of yeast which deteriorated whilst hermetically sealed under nitrogen.

Materials and methods

Yeast was a strain of commercial active dried baker's yeast (*Saccharomyces cerevisiae*) obtained from Distillers Company Ltd, prior to 1966. Reference samples have been stored under nitrogen at −20°C and have not changed in activity or viability.

Dried yeast extract was obtained from Difco.

Wheat starch was from British Drug Houses. As obtained this was found to contain a small amount of yeast nutrients so it was thoroughly washed with distilled water, followed by ethanol and drying at room temperature before use.

All other materials were either 'Analar' grade or the best commercially available products used without further purification.

Gas volumes were measured in the apparatus of Burrows & Harrison (1959) at 30°C. This apparatus was modified by the incorporation of a mechanical shaker for agitating the reaction bottles containing liquid media and an extra tube through the bottle stoppers to allow the passage of nitrogen for anaerobic incubations. It was not possible with this apparatus to determine the oxygen uptake under aerobic conditions.

The yeast suspension was mixed with the substrate and other reactants in the reaction vessels (cysteine was included at this stage when required), followed by the starch where used, stirring vigorously with a spatula, then wiping the spatula with a 7 cm filter paper, which was then placed in the reaction vessel.

After stoppering the bottles, agitation was started and nitrogen passed where required. Agitation speed was adjusted to produce maximum fermentation rates (in this case 100 strokes/min of 4 in.). The time allowed for equilibration before closing the burette taps was 13 min.

The dried yeast was reconstituted by dropping into distilled water at 37·8°C, standing for 15 min, then making up to requisite volume with water. Reconstitution temperatures below 37°C bring about progressive loss of activity with declining temperature (Sant & Peterson, 1958).

90 mg dry weight of yeast in a final volume of 15 ml was used in each reaction vessel. The same strain of yeast was used throughout these experiments though the actual batch may differ from Table to Table in the results section.

The quantity of sugars added was 720 mg/reaction vessel or, if mixed, 720 mg of each. 20 g of starch was used in those experiments where it was employed. Amounts in the range 18–26 g did not affect fermentation rates. In the fully nutrient runs yeast extract was present at a final concentration of 4% w/v, ammonium sulphate 0·2% w/v and calcium chloride hexahydrate at 10 mg per reaction vessel.

Increase in the rate of hexose fermentation prior to uranyl inhibition studies was

produced by incubation of yeast suspension with glucose and nutrients for 1 hr at 30°C, using the same quantities as in the gas evolution determinations. The control sample was incubated with plain glucose solution. Yeast cells were recovered by centrifugation, well washed in ice cold water and re-suspended in a solution of glucose and uranyl acetate.

Parallel incubations were conducted for the purpose of determining increases in dry weight of cells during the course of incubation. Following incubation the same washing procedure was followed and the cells transferred to tared dishes to be dried for 17 hr at 105°C. Comparison was made with cells which had been subjected to exposure to the same media, under ice cold conditions to prevent metabolism without the incubation period.

Samples subjected to accelerated storage were stored under nitrogen at 40°C for 5 weeks.

All figures quoted in the tables are for volumes of CO_2 evolved in 3 hr.

Results

The production of CO_2 by yeast from sugars in media with and without other nutrients has been studied under various experimental conditions, i.e. in aqueous solution under initially aerobic and under anaerobic conditions, and in starch paste. Because of the unknown oxygen uptake of the initially aerobic liquid media fermentations and the physical effects of agitation it was felt that the fermentation of flour doughs would be better modelled in starch paste media. The results are summarized in Table 1. Effects of inhibitors have also been examined.

Sugars in the absence of other nutrients

In the absence of nutrients other than the sugar, the volume of CO_2 produced in 3 hr was independent of the conditions used. In each case, with the exception of maltose, CO_2 production accelerates for the first 15–20 min; the rate achieved at the end of this initial period was then maintained for the rest of the incubation (Fig. 1) and was, within the limits of experimental error, identical for these sugars (Table 1).

Maltose gave no significant CO_2 production during the 3 hr incubation without other nutrients.

Sugars and nutrients

In the presence of nutrients, considerably enhanced CO_2 production could be achieved. The 'complete' synthetic mixture of nutrients described by Atkin, Schultz & Frey (1945) led to a more than two-fold increase in the 3 hr CO_2 production on glucose media and further increases could be achieved by the addition of Ca^{++} and of

TABLE 1. Three-hourly production of carbon dioxide from sugars

Substrate	Agitated liquid media		3 hr starch paste media (ml CO_2)
	3 hr aerobic† (net ml CO_2)	3 hr anaerobic (ml CO_2)	
Glucose	31·5	33·0	33·0
Fructose	—	—	33·2
Mannose	—	—	32·5
Sucrose	—	—	32·8
Glucose + 'A'	72·8	—	—
Glucose + 'A' + Ca^{++}	77·6	—	—
Glucose + 'A' + Ca^{++} + NH_4^+	—	—	94·2
Glucose + yeast extract + Ca^{++}	—	—	96·0
Glucose + nutrients*	106·0	123·6	106·5
Fructose + nutrients	—	—	107·2
Sucrose + nutrients	—	—	109·7
Maltose	1·8	—	2·5
Maltose + nutrients	57·0	47·2	47·9
Glucose + maltose + nutrients	—	—	103·4
Nutrients	2·5	2·2	2·8

* Nutrients as described in experimental section.

† Net CO_2 = Total CO_2 output − Oxygen uptake.

'A' is the complete synthetic medium of Atkin *et al.* (1945).

NH_4^+. However, the maximum 3 hr production of CO_2 observed was in media to which yeast extract Ca^{++} and NH_4^+ had been added. In this case, the rate of CO_2 production during this period showed a continuous increase (Fig. 1). This applied also to the sugars fructose and sucrose.

This enhanced rate of CO_2 production was retained when yeast, incubated in a nutrient medium of this type, was centrifuged, washed and re-suspended in a sugar solution with no added nutrients, e.g. yeast recovered from a system at a stage when CO_2 production was 25 ml/hr, continued to produce CO_2 at the same rate when separated and re-suspended in a solution of glucose alone.

Maltose fermentation could be induced by the addition of nutrients to the media, but only after a period of approximately 90 min, during which time CO_2 production was small, did the fermentation become substantial (Fig. 1). Once initiated, CO_2 production continuously increased in rate during the remainder of the incubation period. Again, rates were maintained on transfer of the yeast to maltose solution with no added nutrients. Attempts to produce earlier onset of high fermentation rates by trace amounts of glucose or oxygen were not successful.

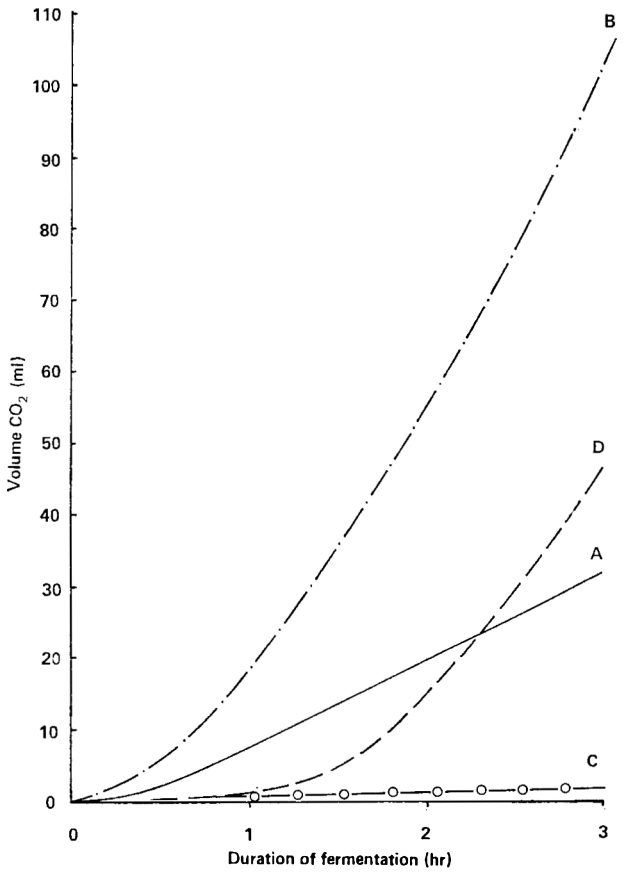

FIG. 1. Total carbon dioxide evolution with time for the fermentation of sugar–starch paste media. A; glucose in the absence of other nutrients: B; glucose in the presence of other nutrients: C; maltose in the absence of other nutrients: D; maltose in the presence of other nutrients.

Inhibition by cysteine

The effect of adding 80 mM cysteine to the media (Table 2) depended upon the presence or absence of nutrients. Evolution of CO_2 during 3 hr incubation in media containing only sugar and starch was only marginally affected by the addition of cysteine; much of the enhancement of CO_2 production produced by nutrients was inhibited by the addition of cysteine. With maltose as substrate the inhibition was such as to reduce the fermentation below the level provided by the fermentation of hexose in the absence of nutrients.

TABLE 2. Effect of 80 mM cysteine on fermentation of glucose and maltose

	3 hr CO_2 (ml)
Glucose + starch	32·5
Glucose + 80 mM cysteine + starch	29·4
Glucose + nutrients + starch	106·6
Glucose + nutrients + 80 mM cysteine + starch	50·6
Maltose + nutrients + starch	48·3
Maltose + nutrients + 80 mM cysteine + starch	18·9

Uranyl inhibition

Uranyl ions reduced CO_2 production from glucose. The extent to which inhibition occurred depended upon the pre-treatment the yeast has received, yeast which had been exposed for 1 hr to a glucose + nutrient liquid medium being less severely inhibited than yeast exposed to a liquid medium containing glucose alone (Table 3). During the nutrient pre-treatment the increase in dry weight of the cells was 2% and budding cells were not present whilst the fermentation rate was doubled. With plain glucose medium no difference in dry weight was apparent.

TABLE 3. Inhibition of fermentation of glucose by uranyl ion in aqueous solution

	Concentration of UO_2				
	*C**	*C*/20	*C*/50	*C*/100	*C*/200
% Inhibition, preincubation with plain glucose	98·4	82·8	51·6	42·7	0
% Inhibition, preincubation with glucose + nutrients	93·6	43·5	0	0	0

* $C = 5 \times 10^{-3}$ M UO_2^{++}

Accelerated storage effects

Storage reduced the amount of CO_2 produced by the yeast from glucose in a nutrient medium, but had little, if any, effect on that produced where glucose was the only constituent of the medium. The ability to develop maltose-fermenting activity in nutrient media was also impared by storage at elevated temperature (Table 4).

TABLE 4. The effect of accelerated storage on the fermentation of glucose and maltose

	Before storage 3 hr CO_2 (ml)	After storage 3 hr CO_2 (ml)	% decline
Glucose + starch	32·7	31·8	2·8
Glucose + nutrients + starch	94·6	79·0	16·5*: 23·6†
Maltose + nutrients + starch	55·8	45·8	17·9

* Calculated on total loss of glucose fermenting ability.

† Calculated on the reduction in the portion of glucose fermentation which required the presence of nutrients.

Discussion

These studies examine the extent to which sugars can be fermented by dried baker's yeast and the dependence of this on the nature of the sugar and on the presence of certain nutrients and inhibitors. Not surprisingly, generally the pattern of fermentative behaviour is similar to that observed by many workers using pressed yeasts, but some differences are apparent.

Sugars alone were not well fermented either in aqueous systems or in starch pastes; the latter may provide conditions which in some degree more closely resemble those existing in flour doughs during bread manufacture than are provided by liquid media.

Under the various conditions employed, the production of CO_2 from media containing only sugars was constant and relatively low with glucose; with maltose there was virtually no fermentation. This low level of hexose fermentation, which will be referred to as the basal level, may be the result of the conditions of high aerobiosis and low substrate concentration used during manufacture. Under these manufacturing conditions the level of carbohydrate intake adjusts itself to the energy requirements of the cell. This is a much lower rate of intake than is required to maintain the same rate of energy supply under anaerobic conditions and is capable only of supporting a relatively low rate of fermentation.

Suitable nutrients raised the rate of glucose fermentation and initiated fermentation of maltose. The fact that, once established, the fermentation of maltose and the enhanced rate of glucose fermentation were retained in the absence of further supplies of nutrient, indicates the synthesis of new cell components. Further, the lack of effect of cysteine on the basal constitutive system as opposed to the nutrient requiring systems supports this contention and also possibly provides a method of distinguishing between constitutive systems and those requiring synthesis.

The nutritional requirements of this yeast are clearly complex, exceeding those

satisfied by the complete synthetic medium of Atkin *et al.* (1945) or this medium supplemented with nitrogen and calcium.

Hexose fermentation by yeast is limited by the rate of hexose transport across the cell membrane (Rothstein, 1954; Hurwitz & Rothstein, 1951). These authors demonstrated that the level of glycolytic enzymes within the cell is sufficient to sustain a rate much greater than that observed, i.e. the basal level is limited by the rate of hexose transport. Rothstein (1965) states that uranyl ion is a specific inhibitor of hexose transport. Table 3 shows the effect of uranyl ion on CO_2 evolution from an aqueous solution of glucose by yeast which had been pre-incubated for 1 hr with glucose complex nutrients and Ca^{++} as compared with yeast which had only been in contact with a plain glucose solution for the same period. It is seen that the concentration of uranyl ion required for a similar degree of inhibition was much higher in the case of the yeast pre-incubated in the complex medium. This implies that uranyl binding sites were formed during pre-incubation in the presence of nutrients, but not in their absence. The increase in the number of uranyl bonding sites is clearly much greater than would be accounted for by the 2% increase in dry weight during pre-incubation with nutrients if merely general growth of the cells were taking place. This change in uranyl inhibition must be due to a membrane change (since the membrane is impermeable to the uranyl ion) and this implies a change in transport characteristics—as indeed is indicated in the present work.

Maltose fermentation in the absence of nutrients was negligible, but could be developed to a substantial degree in the presence of suitable nutrients after a period of about $1\frac{1}{2}$ hr. In the present work it was not possible to reduce the length of the period before substantial maltose fermentation occurred to less than $1\frac{1}{2}$ hr, whereas Blish & Sandstedt (1937) succeeded in doing so by the use of malt flour, dried yeast and carrot extracts. However, the factor responsible for inducing immediate CO_2 production from maltose was heat labile, alcohol labile and nondialyzable; these properties suggest that it was enzymic in nature, possibly a glucosidase or, in the case of dried yeast, a maltase, and if this were the case the additive was inducing a change in the substrate rather than in the fermentative activity of the yeast. Whilst this distinction may not be important in the context of maltose utilization by yeast in bread-making, it is significant in terms of yeast biochemistry.

After $1\frac{1}{2}$ hr the rate of CO_2 production from maltose in nutrient media became comparable with that derived from glucose. The time lag indicates the synthesis of new cell components and it is noteworthy that in their studies on strains of *Saccharomyces cerevisiae* Griffin (1969) and De La Fuente & Sols (1962) concluded that adaptation of yeast to maltose substrates led to the formation of maltase and maltose permease.

The development of maltose fermentation was, as was the development of enhanced glucose fermentation, considerably impaired by high cysteine concentrations. In the case of maltose fermentation the inhibition was such as to reduce the 3 hr CO_2 production below the basal level observed for hexose substrates. This supports the sugges-

tion that in this yeast some components of the maltose fermenting system are not constitutive.

The effects of storage on the basal level as compared with the system leading to enhanced fermentation of hexose and the fermentation of maltose indicate that deterioration under nitrogen is not a simple matter of general loss of enzymic activity or co-factors as such (loss of carboxylase activity due to loss of co-carboxylase has been suggested by Chen & Peppler (1956) for material stored under oxygen). Instead some effect which selectively affects particular systems is suggested. Such a selective effect could occur if, for instance, the genetic material governing the synthesis of new materials were particularly subject to degradation, or it could be due to a deterioration of a moiety which causes a failure of delicately balanced control mechanisms for enzyme synthesis; for instance, development of leakiness of the cell membrane could prevent concentration of the calcium ion shown to be important in enhanced glucose fermentation by this yeast.

In general terms the fermentative activity of this yeast reflects the conditions under which it was grown during commercial production, viz. having been grown on media based on cane or beet molasses (Burrows, 1970) its ability to ferment glucose is constitutive, whereas fermentation of maltose is inducible (in this it resembles many pressed yeasts). This difference is of some importance in breadmaking, particularly in the context of modern short-time dough systems; with the yeast studied in these experiments maltose fermentation in bread production by these systems will be slight and presumably it is for this reason that extra glucose or sucrose must be added to the dough. Commercial production of yeast on media which contain maltose (or maltose-producing substrates) might obviate this requirement.

Conclusions

It is concluded that at least three systems are involved in a full fermentation by this yeast. These are:

(a) An initial basal level for the fermentation of glucose, fructose, and mannose, and through the action of invertase, also sucrose. This level is probably determined by the conditions during manufacture.

(b) A system leading to increased fermentation of the above sugars and requiring the synthesis of new cell components, including transport enzymes.

(c) A system leading to induction of maltose fermentation which then proceeds rapidly.

Storage affects the second and third of these more than the first.

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Some studies on the photolytic decomposition stage in the estimation of N-nitrosamines

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Summary

A study of the photolytic decomposition of certain N-nitrosamines has shown that these compounds degrade at different rates in the various solution media employed. The decomposition follows first order kinetics under most conditions and reaction half lives have been measured. Linear correlations were observed between half lifetimes determined with different sources for each media. A suggestion that the criteria of half lives be used to optimize analytical conditions in the quantitative estimation of nitrosamines is discussed.

Introduction

In recent years the toxic and carcinogenic effects of N-nitrosamines have been reported (Magee & Barnes, 1956; Druckrey, Preussmann & Schmal, 1963). It has been established that these compounds may be produced as the result of chemical and bacterial action on secondary amines in the presence of nitrates (Sander, 1968), and hence may present a hazard with foods containing amines and nitrates, such as vegetables. It was also shown that the synthesis may be accomplished in acidic media without bacterial intervention. The risk of nitrosamine contamination has resulted in projects to investigate methods for their specific sensitive and quantitative determination in samples of interest.

Good separation of selected nitrosamines has been obtained by thin layer chromatography (Preussmann *et al.*, 1964; Serfontein & Hurter, 1966; Neurath, Pirman & Dunger, 1964) and by gas chromatography (Foreman, Palframan & Walker, 1970), but they were unable to include all of the isomeric N-nitrosamines which could theoretically be met. The most sensitive colorimetric reaction is that described by Daiber & Preussmann (1964), in which the NO group is cleaved by U.V. light and the nitrite formed identified by the Griess reagent. The reaction can be carried out on silica gel thin-layer plates and also

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in alkaline aqueous solution. Nitrosamines reduce polarographically at between −0·8 to 1·0 volt relative to a saturated calomel electrode, half wave potentials being pH dependent. Polarographic methods, though sensitive to less than 1 ppm (English, 1951; Smales & Wilson, 1948) are not specific since compounds such as unsaturated aldehydes and ketones which are likely to be present in foods also reduce in this region (Heath & Jarvis, 1955; Lyderson & Nagy, 1967).

An improved method has recently been described which involves the differential polarography of sample solutions before and after photolysis (Walters, Johnson & Ray, 1970). The nitrosamine concentration in the presence of light stable contaminants is estimated by difference.

Little is known about the factors which influence the rate of photolytic decomposition of nitrosamines and photolysis times ranging from minutes (Sander, 1968; Mohler & Mayrhofer, 1968) to several hours (Ballweg & Schmahl, 1967; Walters *et al.*, 1970; Chow, 1967; Sander, 1967) have been recommended. The present study was undertaken to obtain data on the conditions that affect the rates of photolysis of nitrosamines in order to be able to specify more precisely analytical procedures, prior to the final polarographic of spectrophotometric measurement. The effect of pH was examined because several reports suggest that the photolytic decomposition requires strongly acidic conditions (Chow, 1964, 1967; Burgess & Lavanish, 1964; Chow & Lee, 1967) in contrast with the work of Sander (1967) and Daiber & Preussmann (1964) who studied the production of nitrite under alkaline conditions. Since pH and also buffer composition affect the polarographic behaviour of nitrosamines photolyses were carried out in the presence of various supporting electrolytes. The separation and clean-up of nitrosamines from foods, tobacco and certain medicants, prior to their determination, may require solvent extraction (Möhler & Mayrhofer, 1968) so that the variation in the photolysis rates in certain common solvents was also examined.

Experimental photolysis procedures

(1) Irradiation was carried out in stoppered 10 mm silica spectrophotometric cells under a 15 watt Ultraviolet lamp (B.T.L.) designed for chromatographic use, fitted with a Chance OX7 filter to absorb visible radiation and transmit the 254 nm line. The cell, its optical faces at 90° to the radiation beam, was placed at the bottom of the lamp housing 12 cm from the filter. Subsequent experiments at 2, 4·5 and 7 cm showed less than 10% variation in effect with alteration in position. The source is of comparable size and close to the cell so that the inverse square law does not apply. The solution spectra were recorded after various time intervals using a Unicam SP.800 spectrophotometer with the appropriate solvent in the reference cell.

(2) Further investigations were carried out by irradiating 100 ml nitrosamine solutions in a 1000 ml Hanovia Photochemical reactor. The sample solution was placed in the bottom of the outer flask, the removal of small sample aliquots does not significantly

alter the sample–source geometry and hence the irradiation conditions. The medium pressure mercury arc emits predominantly at 254, 265, 297, 313 and 366 nm. The region round the tube is purged with nitrogen and water circulated in a jacket between the gas purge space and the irradiation container. Aliquots of the solution were withdrawn at 10-min intervals until spectra failed to show any further change.

To avoid photolytic decomposition by daylight (Sander, 1967), solution flasks were protected with aluminium foil. The U.V. sources were carefully shielded to prevent irradiation of workers.

Solutions containing 10 ppm of each nitrosamine were used, these had suitable absorbances for following the photolysis reactions.

Results

The absorption maxima of the nitrosamines studied, dimethyl nitrosamine (DMN), dibutyl nitrosamine (DBN) and N nitrosopiperidine (NNP), were found to be in the range 225–242 nm, the exact position varying only slightly with solvent. On irradiation less absorbing products formed, having maxima in the range 204–228 nm, 10–20 nm below the maximum absorption of the parent materials. The band at about 230 nm is known to obey Beer's law (Möhler & Mayrhofer, 1968). Plots of log $(A_t - A_{t\infty})$, for each nitrosamine, at the peak wavelength, against photolysis time gave linear relationships indicating first order kinetics. Here A_t is the absorbance at time t. In some cases a short initial period of lower order kinetics was observed. The data (Table 1) for dimethylnitrosamine in 0·2 M hydrochloric acid showed the most curvature of any studied. The reaction half life times shown in Table 2 were evaluated from the linear, first order, portions of plots. The reaction half life times observed with the low and high intensity sources are given in Tables 2(a) and 2(b) respectively. Plots of half lifetimes determined with one source against those determined with the other source show reasonably linear correlations for each medium examined.

The possible effect of concentration on half lives was examined using 20, 5 and 2·5 ppm NNP at pH 4 in 5, 20 and 40 mm cells with the B.T.L. lamp. The half lives were 1·5, 1·2 and 1·35 hr respectively, i.e. constant within the normal coefficient of variation of results of 15%.

Discussion and conclusions

The rate of photolysis of the nitrosamines studied followed first order kinetics. The reaction halflives are characteristic of each nitrosamine but varied with the solution and irradiation conditions; values observed ranged from $\frac{1}{2}$ to $7\frac{1}{2}$ hr with the low intensity source and from $\frac{1}{4}$ to 1 hr with the photochemical reactor. Decomposition was faster at low pH and in methanol, ethanol and in dichloromethane except that dimethylnitrosamine appeared more stable in methanol. The results show that the effect of

TABLE 1. Decomposition of dimethylnitrosamine in a Hanovia photochemical reactor

Solution	0·2 *M* hydrochloric acid		pH 4 (universal buffer)		
Time (min.)	Absorbance	log $(A_t - A_{t\infty})$	Time (min.)	Absorbance	log $(A_t - A_{t\infty})$
0	1·01	$\bar{1}$·87	0	1·04	$\bar{1}$·91
10	0·92	0·81	10	0·94	0·85
20	0·77	0·70	20	0·82	0·77
30	0·63	0·56	30	0·75	0·72
40	0·52	0·40	40	0·69	0·66
50	0·43	0·20	50	0·64	0·61
60	0·36	0·04	60	0·58	0·54
100	0·27	—	100	0·43	0·30
110	0·27	—	240	0·23	—

TABLE 2. Reaction half lifetimes for the photolytic decomposition of N nitrosamines

Solvent	DMN	DBN	NNP
(a) B.T.L. lamp (times in hr)			
pH 4 (universal buffer)*	2·2	0·7	1·2
pH 9·2 (universal buffer)*	3·5	1·5	1·4
0·2 M HCl	4·9	2·7	2·1
0·1 M HCl†	5·5	3·1	2·0
0·5 M Na_2SO_4, 0·5 M $NaHSO_4$‡	7·3	3·6	3·4
Ethanol	1·3	1·5	1·3
Methanol	6·3	1·6	2·2
Hexane	3·7	2·8	3·4
Dichloromethane	2·3	1·3	1·5
(b) Hanovia Photochemical Reactor (times in min)			
0·2 M HCl	25	16	15
pH 4 (universal buffer)	49	17	23
pH 9·2 (universal buffer)	64	28	25

* Johnson & Lindsey (1939).
† Smales & Wilson (1948).
‡ Lyderson & Nagy (1967).

acidity are less significant than previously suggested, and that nitrosamines are not stable (Chow, 1964) nor are decomposition reactions hindered in organic solvents (Mohler & Mayrhofer, 1968). Comparison of life times determined in 0·2 M and 0·1 M hydrochloric acid (pH about 0·7 and 1·0 respectively) with results obtained in buffered solutions at pH 4 and 9·2, showing a depression of the effect of pH, indicates that salt effects may be important.

As stated previously, half lives were determined from linear plots of log $(A_t - A_{t\infty})$ against time. In some cases the first few points were not linear, due to an inner filter effect, shown as a tendency to zero order kinetics which results when significant fractions of incident radiation are absorbed by solute molecules. Chow (1967) previously observed zero order kinetics in the photolysis of concentrated solutions of nitrosamines. At the 10 ppm levels examined linearity was always achieved in less than one half life.

The linear correlations between half lifetimes in various media, determined using different sources, are useful in that they should allow estimation of reaction times with a particular source for a series of nitrosamines, from half lives measured by other workers. The relationship can be established, for ease of measurement, using the slowest photolysed member of the series. The variation in slope of the interrelation graphs between media is probably due to a lack of uniformity in spectral characteristics of the U.V. sources.

Walters *et al.* (1970) irradiate for fixed periods, the disappearance of a polarographic wave in 2 hr in acid conditions and the formation of nitrite under alkaline conditions in 15 min being used to detect nitrosamines. The use of fixed irradiation times to complete reactions is not satisfactory. The term 'complete reaction', particularly in slow reactions, is not in itself precise since concentrations decrease asymptotically to the time axis for all but zero order kinetics. Once the systems under consideration follow first order kinetics, use of half lives to specify periods of irradiation for a required percentage of reaction to occur, is suggested. A known decomposition of over 99%, a good approximation to complete reaction, would occur in seven half lives.

The extraction and isolation stages from various products such as fish (Lyderson & Nagy, 1967), animal tissue (Heath & Jarvis, 1955), meat and cheese (Mohler & Mayrhofer, 1968) have been considered by previous workers and also lipid effects noted (Walters *et al.*, 1970).

Acknowledgments

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Technical note: Dextrose equivalents of maltodextrins and the Lane and Eynon titration

G. G. BIRCH, M. S. A. KHEIRI AND D. C. HUFTON

Although the Lane and Eynon titration (Lane & Eynon, 1923) has been developed to achieve reproducible oxidation of reducing sugars, it is a non-stoicheiometric reaction in which approximately 5·0 equivalents of cupric ions are required to oxidize 1·0 mole of reducing sugar. This indicates that the oxidation of reducing sugar molecules extends much further than the potential aldehyde or keto group, and part of the sugar is, in fact, broken down to substances, which in turn reduce the copper reagent (Heidt & Southam, 1950).

Commerford & Scallett (1969) have recently measured the reducing powers of a homologous series of linear maltodextrins by application of dextrose equivalents (i.e. D.E. or reducing power calculated as dextrose and expressed on a dry weight basis). They found that the theoretical D.E. of a maltodextrin (i.e. molecular weight of dextrose divided by molecular weight of maltodextrin times 100) was always lower than the observed D.E. under certain conditions. We have, therefore, re-examined Commerford and Scallett's results and the products obtained after oxidation of glucose, maltose and maltotriose in the Lane and Eynon titration in an attempt to illuminate the mechanism of the oxidation reaction.

Experimental

Chromatographically pure glucose and maltose were used for the investigation. Chromatographically pure maltotriose was supplied as a gift from Beecham Products (U.K.). The Standard Corn Industries Research Foundation modification of the Lane and Eynon titration (Standard Analytical Methods of the Member Companies of the Corn Industries Research Foundation, 1963) was used for determining the end point of oxidation of all three sugars. After titration the solutions were filtered from cuprous oxide precipitate and the filtrates were in each case examined for total carbohydrate content by the phenol sulphuric acid procedure (Dubois *et al.*, 1956).

Results and conclusion

When the ratio $D.E._{n+1}/D.E._n$ is calculated from Commerford and Scallett's results, the ratio for sequential members of a series of maltodextrins (Table 1) is found to be

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the same as the ratio $M.W._n/M.W._{n+1}$ (i.e. the sequential molecular weight ratio) with the exception of glucose.

TABLE 1

D.P. (Degree of polymerization)	D.E. (Commerford and Scallet values)	M.W. (Molecular weight)	$M.W._n/M.W._{n+1}$	$D.E._{n+1}/D.E._n$
1	100·0	180	0·526	0·580
2	58·0	342	0·678	0·681
3	39·5	504	0·756	0·754
4	29·8	666	0·804	0·812
5	24·2	828	0·836	0·859
6	20·8	990	—	—

This is not surprising as it is now well recognized that D-glucose differs from homologous members of the maltodextrin series in many physical and chemical properties (e.g. anomalous paper chromatographic mobility and low reducing power), due to the absence of the glycosidic linkage. However, with this one exception Table 1 indicates that a stoicheiometric relationship does exist between reducing power (D.E.) and degree of polymerization (D.P.) up to at least maltohexaose. This conclusion in turn suggests that although oxidation of the reducing sugar is not confined to the potential aldehyde or keto group it is nevertheless confined to the reducing residue. In other words no 'peeling action' occurs along the chain of glucose residues in a maltodextrin. We have now attempted to confirm this final suggestion by investigating the products of oxidation (by the Lane & Eynon titration) of glucose, maltose and maltotriose, by assessing destruction of carbohydrate moiety. If oxidation had been confined to the reducing residue in each molecule, 1·0 equivalent of maltose should yield 1·0 equivalent glucose as total residual glycosidic carbohydrate at the end point of the titration (Birch, 1963). On the same basis, 1·0 equivalent of maltotriose should yield 2·0 equivalents of glucose, as total residual glycosidic carbohydrate. Total carbohydrate assay, after oxidation, however, at a number of different concentrations yielded only 0·88 equivalents of glucose from maltose and 1·60 equivalents of glucose from maltotriose, thus indicating that the oxidations had in each case proceeded beyond the reducing residue. The products of oxidation of glucose, when tested, did not interfere with the analysis.

Although this result disproves the hypothesis that the oxidation of reducing sugars is confined to the reducing residue, presumably oxidation does in fact proceed beyond the first glycosidic linkage of homologous maltodextrins in an analogous manner. This is of significance in the commercial analysis of glucose syrups in calculating deviations from random conversion (Birch, Green & Coulson, 1970).

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Book Reviews

Metabolic Aspects of Food Safety. Ed. by FRANCIS J. C. ROE.
Oxford and Edinburgh: Blackwell Scientific Publications, 1970. Pp 612. £7·00.

This volume provides its readers with the contributions presented by the participants at the Second Nuffield Food Safety Conference held at Cambridge in 1969. It is dedicated to the memory of Dr Alastair Frazer, who, 'but for his untimely death, would have been its principal editor'. It includes, in fact, the paper which would have been read by this great medical scientist on that occasion in which he makes a plea for the need for more biochemical information in the field of food safety evaluation. In summary, Dr Frazer proposed six points concerning the studies needed in the investigation of the safety-in-use of a food additive; these include the adequate specification of the additive, its interaction with the food itself, the changes that it may undergo in the gastro-intestinal tract, the chemical modifications that it may undergo in the body and the biological half-life of metabolites formed, together with the need for biochemical studies in various species or strains of animals and in human subjects to enhance the value of animal toxicity tests. To judge by the reported stimulating and thought-provoking discussion that ensued, this paper set the tone for the rest of the contributions which ranged widely in subject and content.

As befitted a conference concerned with the evaluation of food safety, the various aspects of absorption from the gastro-intestinal tract were well featured, with particular reference to the factors influencing the transport of food additives from the lumen of the intestine into the blood stream. From this stage, attention was directed towards the transport and fate of substances absorbed, followed logically by the consideration of renal function tests in laboratory animals and in man in the assessment of the biological actions of food additives. Of necessity, analytical procedures are essential for the specification of such additives and the limitations of such methods receive due attention amongst the papers presented. A major portion of the conference dealt with liver toxicity and carcinogenesis and a number of papers were concerned with such aspects as the assessment of liver function in man with special reference to hepatotoxicity, the significance of liver tumour induction in animals and the effects of carcinogens on the structure and activity of liver cells etc. Emphasis was also placed upon the status of test animals in the evaluation of food additives and upon their ability to induce liver enzymes relevant to their metabolism along with the significance of the intestinal flora in their appraisal.

From the foregoing incomplete list of the topics discussed, it will be apparent that this book will not find its niche on the shelves of every food technologist. The conference, of which it reports both the individual contributions and the subsequent very enlightened discussion, was designed for the specialist involved in the future requirements

for the assessment of food additives and components to which man could be subjected over a lifespan. As such, the book represents a valuable reference volume surveying in great detail the many aspects which must be considered by the innovator of any future food additive designed to assist the technologist. Whilst the style of each chapter is that of the individual author, all should be comprehensible to the forward-looking scientist who wishes to acquaint himself with the complexities of the issues involved. The topics involved are well illustrated in diagrammatic form and relevant references to the literature are provided to facilitate more detailed inspection of the experimental results received. Of particular interest is the discussion which follows each contribution, in which the critical appraisal of the specialist audience is clearly apparent. In view of the obvious impact of the dietary pattern upon the health of a nation, it is comforting to realize from reading this book the depth to which the actions of food components whether deliberately added or adventitious can be investigated. In praising the Nuffield Foundation for its support of work on food safety, one eminent participant concluded 'it will be seen that food additives do not constitute a very serious toxic hazard because of the way in which they are selected and tested'; this volume performs an invaluable function in allowing the reader personally to examine the evidence upon which that statement is based.

C. L. WALTERS

Micro-organisms: Function, Form and Environment. Ed. by L. E. HAWKER and A. H. LINTON.
London: Edward Arnold Ltd., 1971. Pp. 724. £6·00.

In 1960, a small team of authors from the University of Bristol produced *An Introduction to the Biology of Micro-organisms*, which was at the time unusual amongst microbiology texts in that it dealt with the whole range of micro-organisms including viruses, protozoa, the micro-fungi and micro-algae without undue emphasis either on the bacteria or on the medical aspects of the subject. This general biological approach, together with the lucid treatment and modest price of the work, rapidly established it as one of the standard introductory texts for the growing number of university degree courses in microbiology and related subjects.

The field of endeavour of this succeeding volume—which is in no way a second edition—remains the same, to provide a general survey for the student and research worker of the whole subject of microbiology, but the advances of recent years have necessitated a substantial increase in both size and price. A general volume, even of this bulk, can never replace the specialized texts on such subjects as immunology and the ecological, genetic and applied aspects of microbiology which are essential for the advanced student and for the research worker; it nevertheless seems likely that Professor Hawker and Dr Linton have compiled what will be regarded as one of the best treatments of the subject in a single volume for some years to come.

There is a chapter on the microbiology of food and beverages, which introduces the different methods of food preservation and the part played by micro-organisms in the production of various foods and beverages; the approach is biological rather than technical. Another chapter contains a section on fermentations for the industrial production of micro-organisms which, though dealing with bakers' yeast, surprisingly omits to mention recent efforts towards the production of microbial protein as a dietary supplement. Food safety, food-borne diseases and food poisonings are also briefly discussed.

No less than twenty-two authors (all from Bristol) have contributed, and some inequalities are therefore perhaps to be anticipated; experts will inevitably find flaws. For example, the chapter on Numerical Taxonomy describes for the coding of quantitative data two early methods which are clumsy and inexact; cluster analysis by three methods is described, but principal component analysis is not discussed. The section on Artificial Passive Immunity mentions neither tetanus antitoxin nor purified gamma-globulin preparations.

There are a number of errors in the formulae included in the chapters on the biochemistry of micro-organisms. Thus the formula given for amylase (poly-α-1:4 glucose) in fact represents poly-α-1:4 galactose, there is the occasional pentavalent carbon atom and divalent nitrogen atom and some compounds are missing a functional group. Table 8.1 quite wrongly suggests that Gram-negative bacteria contain far more muramic acid than do Gram-positive species and the structure of N-acetyl muramic acid is misrepresented in Fig. 1.22.

Irritations such as these are probably inevitable in a work of such complexity; they can be corrected in subsequent editions and do not materially detract from the value of what seems certain to become a standard text for university students as well as a most useful reference guide to microbiology for the research worker.

J. E. Smith

International Commission for Uniform Methods of Sugar Analysis. Fifteenth Session, London 1970—Proceedings.
London: I.C.U.M.S.A., 1971. Pp. xi + 338. £4·00.

How can one review a 'Proceedings'? The outline is laid down by the Committee, the content is determined by the participants, and the presentation is largely direct reportage. Nevertheless, the Fifteenth I.C.U.M.S.A. Proceedings has broken new ground in several directions, and must be regarded as an improvement in all previous productions, except perhaps by the hardier dyed-in-the-wool reactionaries. For the first time in its history the format has been changed, and the result is a book of conventional page size (9 in. × 6 in.), bound in substantial board covers with gold blocked

title on the spine, and is a volume which should take its place on the bookshelves of all sugar chemists. The traditional photograph of 'Delegates and Ladies' seemed somewhat compressed in its new form, but has lost nothing in clarity.

One innovation which is long overdue, is the inclusion of the Subject number and title on the upper edges of each pair of pages. The Index is adequate but could well be expanded and there are several peculiar irrationalities.

The entry 'European Economic Community, see E.E.C.' is surely superfluous (for everyone must be familiar with the initials E.E.C.) especially as the single reference under this heading is 'E.E.C. points system for white sugar'. This is not cross indexed under 'Points system' but is included under 'White Sugar'. 'Brunswick colour standards' is listed as a topic on pages 226, 275 and 285. In fact page 226 ignores Brunswick and this seems to be an index error for the subject is introduced on page 274 which appears under the reference 'Colour type of white sugar'. 'EDTA in chemical determination of sucrose' (p. 68) is not mentioned under 'Sucrose, determination by chemical methods'.

Very few 'names' are included as prime references, for example Karl Fischer can be found under 'Water' (incidentally 'Moisture content' does not appear in the Index) but not under 'Fischer'. On the other hand 'Muller's solution: iron content' has the cross reference 'Iron: content of Muller's solution'.

The total of 203 recommendations finally adopted gives some indication of the attention to detail being devoted by analysts throughout the world to sugar chemistry. To some readers, the most significant chapter may well be Subject 29—Starch Hydrolysis Products. A conflict of interests has existed for many years, but at last sweet reason prevails, and we can look forward to a useful collaboration between delegates from the starch-hydrolysis, beet- and cane-sugar industries.

This well-produced volume is an essential supplement to 'I.C.U.M.S.A. Methods of Analysis' (edited by de Whalley, 1964, published by Elsevier) and includes innumerable references to the literature.

R. BUTLER

The Yeasts. Vol. 2. Ed. by A. H. ROSE and J. S. HARRISON.
London and New York: Academic Press, 1971. Pp. 571. £8·50.

A review of Volume 3 of this series of books noted that, so far as some food scientists and technologists are concerned, there are some gaps in the information presented which may make it difficult for the uninitiated to easily comprehend some of the important statements linking practical aspects to fundamental biochemical and physiological data. It was anticipated that these gaps would be filled when Volume 2 was published; this has proved to be the case.

As for Volumes 1 and 3, the editors chose their contributors wisely by selecting

experts having the ability to sift the available information and then present the material in a manner that should satisfy both those who are and those who are not familiar with the subject. All Chapters of the book are more than regurgitations of extracts from the literature; the material, as assembled, both illustrate the developments related to each topic and focus attention on controversial problems.

The large number of references provided are, by themselves, a unique collection, but it has to be regretted that titles of papers were not provided.

The extensive author and subject indexes, covering some 54 pages, enables the readers seeking specific information to find their way easily through the mass of information provided.

It is difficult to pin-point the parts of this volume which are most likely to be of value to readers of this Journal. It is possible that all who have any interest in cell biochemistry and physiology will find some material of value. This is because the very considerable amount of information collated about this group of organisms has rarely been equalled for other groups, hence the readers by analogy may develop ideas applicable to their special biological interest.

For those in the food industry who found Volume 3 to be of value, the most interesting Chapters are likely to be those dealing with nutrition and solute uptake; influence of temperature on growth and metabolism; energy yielding metabolism; kinetics and energetics of growth.

Regarding nutrition, as is to be expected, the Chapter deals individually with nutritional aspects of sources of carbon, nitrogen, minerals and growth factors, but the applied scientist may well find particularly interesting the section on natural and industrial sources. The section on solute uptake presents modern concepts of transport reactions in a digestible form. A short section on the outflow of compounds is included and this is a topic which is often neglected in text-books.

The Section on energy-yielding metabolism includes the usual ground covered by reviews on this topic but brings the reader as up to date as can be done by a textbook. For the food scientist the importance of this section is that, (a) it is an excellent general review, (b) it complements the section on nutrition, and (c) it provides background information to many of the points raised in practical considerations in Volume 3.

The section dealing with the effect of temperature on growth is relatively short but it has assembled the meagre amount of information on this topic, thus making readily available data of value to many in the food industry.

It is becoming increasingly important for many workers in the food industry to have a working knowledge of aspects of the kinetics and energetics of reproduction of microorganisms, but all too often the complexity of the problem is not appreciated. The Chapter on this topic illustrates this complexity but at the same time explains the problems in a lucid manner, taking the reader through ninety-two formulae in a logical sequence. When the reader has mastered the notation for the formulae (seventy-five

symbols are defined), the Chapter is not too difficult for a beginner to understand. The explanation of design of model systems indicates the trends in kinetic studies and shows the potential value of such an exercise.

E. O. MORRIS

Gustation and Olfaction. Ed. by G. OHLOFF and A. F. THOMAS.
London: Academic Press, 1971. Pp. x + 275. £4·00.

This book is a record of the proceedings of an International Symposium held at Geneva in June 1970. Besides a summary of the discussion and references for each of the twenty papers, there are comprehensive author and subject indexes.

The papers, given by scientists from many countries, report recent investigations into the mechanisms of odour and flavour detection, discrimination, sensitivity and specificity. The experiments reported and cited refer to investigations with humans, animals and insects, and are discussed in the light of modern theories of sensory perception.

Some 680 names are listed in the author index, and these, together with the papers and discussions, provide a comprehensive coverage of recent developments in the endeavour to understand this very difficult subject.

H. GOODALL

Food and the Law—A Symposium. Ed. by R. L. JOSEPH.
Dublin: Republic of Ireland Branch of the Institute of Food Science & Technology, 1971. Pp. 62. £2·50.

This slim but well-packed volume contains the proceedings of a Symposium held in Dublin on 22nd and 23rd April 1970 by the Republic of Ireland Branch of the Institute of Food Science and Technology.

A review of a publication of this kind must inevitably be in large measure a review of the content of the Symposium itself. Additionally, the prospective reader will wish to know how effectively the proceedings have been edited and presented (one can call to mind some rather horrid instances in the past); and whether the lapse of time between the Symposium and the publication has rendered any of the content obsolete.

To dispose of the two latter items first, Dr Joseph and his Editorial Board are to be congratulated on absolutely first-class editing and presentation. The text is, by present-day standards, remarkably free from error. The reviewer detected a mere two minor typographical errors which will mislead no-one; and 'Claims of Misleading Descriptions' (p. 45) should of course read 'Claims and Misleading Descriptions'. Furthermore, perhaps by a lucky choice of topics and timing, nothing has occurred since the

Symposium to render obsolete any of the fundamental principles discussed therein. There have been developments in the details of national food legislations, and progress in both Codex Alimentarius and E.E.C. work on elaboration of standards, but basically along the lines described or foreshadowed at the Symposium. Much of what was said about the principles and practice of U.K. food legislation may, of course, become obsolete fairly rapidly when Britain enters the E.E.C., but it remains valid at the time of writing.

Food legislation comprises one of the three major groups of constraints within which food manufacture/marketing has to operate (the other two being consumer preference and the current state of technology). It is not surprising that meetings and symposia on food legislation topics have been numerous and generally well-attended. Nor, in consequence, is it surprising that some of the information, interpretation and opinion here given has been previously presented elsewhere, both by some of the present authors and by others. It is, perhaps, difficult to be original on the subject of legislation!

The book contains a foreword by John A. O.Keefe, O.B.E., the Introductory Address by Erskine Childers, T.D., Táiniste and Minister of Health; General Principles of Law by Liam Hamilton, S.C.; Food Legislation in Britain by Leo G. Hanson; Food Legislation in the European Economic Community by Egon Gaerner; Domestic and International Legislation—An American View by Hermann P. Binger; Food Legislation in Sweden by George Brännland; Consumer Protection and Food Law in Japan by Joji Sasaki; Food Law and the International Company by James Brett and Victor Staniforth; and Food Standards for the World—The Codex Alementarius by Graham Kermode.

The Symposium was thus predominantly international in character. If, from time to time, legislative matters and problems were considered from the viewpoint of Ireland as a food-producing and food-exporting country, this is understandable and in no way detracts from the value of the material to the non-Irish participant or reader.

A two-day Symposium on so wide and important a subject inevitably imposes limitations on the selection of topics and the depth to which any topic can be explored. The programme organizers are therefore 'on a hiding to nothing' with carping critics who might produce a list of relevant topics omitted, and/or complain of inadequate depth of treatment of topics which were included. By and large, in this instance, the organizers are to be congratulated on a well-planned programme, and (despite the reviewer's earlier remarks) even managed to include some unusual material, in the Swedish and Japanese contributions. Nevertheless, although there were passing references to the need for food legislation to be dynamic rather than static, this theme was not developed. It would have been useful to include (perhaps by restricting time devoted to some of the more familiar and previously well-aired material) a discussion on the process and machinery by which food legislation may be made to move with the

times more efficiently and less cumbersomely, a topic which has so far been rather neglected at meetings and symposia, but which is widely discussed informally among food technologists who chafe under 'the law's delay', and which will become increasingly important with the accelerated development of food technology.

Strangely, in a Symposium on 'Food and the Law', the voice of the Public Analyst was not to be heard (unless, perhaps, disguised by the pseudonym 'Q' among the questions and answers given at the end of some of the papers).

All in all, it is a useful and well-produced book, worth every bit of £2·50 to anyone concerned with the technology, manufacture or sale of food.

J. R. BLANCHFIELD

Freeze-drying of Foods (CRC Monoscience Series). By C. JUDSON KING. London: Butterworths, 1971. Pp. 86. £5·00.

The stated purpose of this work is twofold: 'to give a general overview of the status of knowledge and techniques for freeze-drying as used for foods, and to update previous reviews of this field by giving a more intensive coverage of the progress of the last ten years'. The author seems to fulfil these two objectives admirably.

Following a brief introduction in a section entitled Physical Mechanism of Freeze-drying, the evidence for and against the postulation of a sharp sublimation front is presented. In general the evidence supports the use of this theory for design and analysis of freeze-drying processes. Under the title Drying Rates, the Uniformly Retreating Ice Front (URIF) Model for drying rate calculations is outlined and work done on its application reviewed. The weight of evidence suggests that it is a useful model. A good discussion of the factors affecting the thermal conductivity of freeze-dried foods and a table of values compiled from the literature is included. The literature on mass transport of water vapour through the dry layer in 'solid' foods and frozen liquid foods is then reviewed and the section ends with a discussion of the factors contributing to low terminal drying rates. The next section headed Influence of Freezing Conditions covers the literature on the effects of freezing rate on crystal size and void size and subsequent drying for both 'solid' and liquid foods. The problems of recrystallization and keeping liquids frozen during drying are dealt with. Of special interest, is the summary of the influence of freezing rate on the quality of freeze-dried coffee.

The next major section is headed Quality Factors as Influenced by Processing Conditions and includes a detailed coverage of aroma retention. The influence of solids content is underlined and it appears that this effect is more satisfactorily explained by a selective diffusion rather than a selective adsorption concept. Other topics included in this section are: the physical and chemical state of water in frozen and freeze-dried foods, chemical and biochemical reactions during drying and storage

(lipid oxidation, non-enzymatic browning and other reactions), shrinkage, rehydration ratio, water-holding capacity and residual moisture content.

Under the title Processing Approaches the main processing problems are outlined; conventional and continuous processes, vacuum systems and conduction heating systems are discussed. Other developments also reviewed include atmospheric freeze-drying, the use of increased pressure in the drying chamber, carrier gases, fluidized beds and pressure cycling techniques. Microwave heating, its advantages and limitations, and developments in freeze-drying liquid foods are also reviewed.

The final major section, Quality Control, contains discussions of the problems of maintaining uniform and maximum drying rates and end point determination.

Overall this comprehensive review work should prove of great value to students of and researchers into freeze-drying. Its usefulness, to persons involved more in the commercial side of this process, might have been enhanced by an elaboration of the small section on market and product status so as to give a fuller picture of the position of freeze-drying in the wider field of food processing. However this may be outside the intended scope of the work.

J. G. BRENNAN

Proteins as Human Food. Ed. by R. A. LAWRIE.
London: Butterworths, 1970. Pp. xvii + 525. £7·50.

Unlike so many books of multiple authorship this one is particularly well put together with the minimum amount of overlap. The balance of protein content has been well thought out: supply and demand; preservation and processing; animal sources and plant sources; the unconventional, embracing single cell and algae. Why soya proteins should editorially be regarded as unconventional is not clear. The reader will also have organoleptic aspects of protein on cooking as well as catering but perhaps unique for this type of publication is the coverage of protein assimilation. Taking this last aspect first we learn that the metabolic machine needs to be highly adaptive. The liver is intensely bombarded with amino acids from protein digestion following each meal containing protein (Munro), but only about 10% is available for general systemic distribution with some 60% lost as urea. Indigestion of disproportionate amounts of amino acids appear in certain instances to be hazardous (Harper). Assimilation to a low protein diet can make the subject ill-prepared to cope with a subsequent surplus of a few individual amino acids. Unfortunately it appears that the work on these effects reported here is based on rat feeding and hence is not automatically applicable to our subject 'Proteins as Human Food'. The method of protein evaluation for nutritional purposes comes in for constructive review and criticism (Lewis and Boorman) while a comprehensive gamut of abnormalities in protein metabolism (Carson) takes us through disorders in the digestion by enzyme

deficiency, disturbances in intestinal transport of amino acids of which at least five group systems are recognized, then on to include protein intolerance. It will not be possible in a review article to cover all the papers albeit all the authors are well recognized specialists. For example the 'politics' and 'statistics' are covered by Autret, Tuck, Hollingsworth and Pirie. British readers will be consoled to know that the present protein availability in the United Kingdom is satisfactory. To get some peoples to eat proteins, however, requires propaganda and skilful presentation. Can this subject not be the subject of a follow-up 'Easter School'? All the authors are to be commended on highly readable communications backed with down-to-earth information. Even the miniscule amount of mathematical treatment is not out of place. Professor Lawrie is to be congratulated on providing student and specialist alike with a volume that they will need to refer to for some years to come.

A. COURTS

Synthetic Foods. By MAGNUS PYKE.
London: John Murray, 1970. Pp. viii + 145. £2·50.

The thesis of Magnus Pyke's book is 'to demonstrate that food can be made by the artificial means of chemical synthesis'. He makes it clear that there are certain biological syntheses that are strictly speaking outside the terms of the book but are included for historical perspective.

The two references indicating the effect of the discovery and exploitation of detergents on improving supplies of edible fat and the saving in grain by the chemical synthesis of industrial alcohol, show the reader the impact of non-food technology on food resources without being arguments against synthetics as such. The author looks to the petro-chemical industry for the raw material of many of the synthetic processes. Given that nuclear power overcomes some of the envisaged long-term pollution problems and reduces our need for fossil fuel, and cars go electric, then a move towards food production by the petro-chemical industry is a distinct possibility.

After so many recent books and papers stating there is little hope for food production outside the traditional photosynthetic agricultural combination this book is refreshing. The author wisely draws attention to the rise in importance of synthetic fibres in competition with natural fibres over the past 100 years and suggests the time scale for synthetic foods may well be considerably shorter. A thought provoking, thoroughly readable volume in the Pyke tradition which, once started, is difficult to put down.

PAUL WIX

Inhibition and Destruction of the Microbial Cell. Ed. by W. B. HUGO. London: Academic Press, 1971. Pp. 819. £12·00.

The Editor has brought together a number of contributions on a variety of different aspects of the control and destruction of viruses, bacteria and fungi. The chapters in the first half of the book are written from the point of view of specific physical and chemical agencies, excluding antibiotics, used in the control of micro-organisms. They include heat, low temperature, radiation, increased solute concentrations, toxic gases and groups of chemicals used as disinfectants and antiseptics. The second half of the book looks at the inhibition and destruction of particular groups of micro-organisms. The Editor is himself aware that such an approach would inevitably lead to duplication, but there is a worthwhile bonus in the convenience with which the book can be used as a work of reference. This second part of the book includes chapters on *Pseudomonas aeruginosa*, the Gram-positive cocci, the Enterobacteriaceae, Mycobacterium and viruses as well as a detailed account of the destruction of bacterial spores and a chapter on the inhibition and destruction of moulds and yeasts. This latter is mainly concerned with antifungal antibiotics and synthetic fungicides active against human, animal and plant pathogens. The control of fungi involved in industrial and food spoilage is hardly dealt with in the book under review but to have included a satisfactory coverage of these organisms would have made too large a book.

The final chapter in the book concerns the influence of inoculum history on the response of micro-organisms to inhibitory and destructive agents. It is so easy in many laboratory experiments to forget that the nature of the inoculum forms an important part of the experiment and this last chapter should prove to be a useful review to all microbiologists.

Each chapter is followed by an extensive bibliography and the book contains both a complete author index to the bibliographies and a good subject index. Indeed, the subject index helped to solve a small problem in nomenclature which arises from the duplication already referred to. Fig. 1 of Chapter 4 and Fig. 14 of Chapter 11 are identical, being from the same source, but one is described as referring to experiments with *B. subtilis* and the other to *B. globigii*. The index provides the information that *Bacillus globigii* = *B. subtilis* var *niger* thus reconciling the information in the two chapters!

This is essentially a reference book but there is collected together much information which will be of value to a wide range of people interested in micro-organisms, especially those involved with medical, public health and food microbiology.

MAURICE O. MOSS

Books received

Ecology and Physiology of Parasites. Ed. by A. M. FALLIS.
London: Adam Hilger Ltd, 1971. Pp. x + 258. £7·40.

Chemistry of Natural Products, Seventh International Symposium IUPAC Organic Chemistry Division. Ed. by M. N. KOLOSOV.
London: Butterworths, 1971. Pp. vii + 304. £7·80.

Flavour Technology. By NICHOLAS PINTAURO.
New Jersey: Noyes Data Corporation, 1971. Pp. vii + 228. $35.

Poultry Processing. By G. H. WEISS.
New Jersey: Noyes Data Corporation, 1971. Pp. iii + 168. $24.

Vegetable Processing. By MILTON GUTTERSON.
New Jersey: Noyes Data Corporation, 1971. Pp. viii + 335. $36.

Catering with Meat. Ed. by MISS JOAN DANDO.
London: Maitland & Sons Ltd, 1971. Pp. 113.

Chemistry in Industry: Food and Drugs. By PETER TOOLEY.
London: John Murray, 1971. Pp. 280. £2·85 (Paperback edition £1·85).

Analysing Catering Operations. By GORDON CUTCLIFFE in collaboration with DAVID STRANK.
London & Maidenhead: Edward Arnold (Publishers) Ltd, 1971. Pp. vi + 90. £1·60.

Ergonomics: Functional Design for the Catering Industry. By R. H. D. STRANK.
London & Maidenhead: Edward Arnold (Publishers) Ltd, 1971. Pp. v + 56. 90p.

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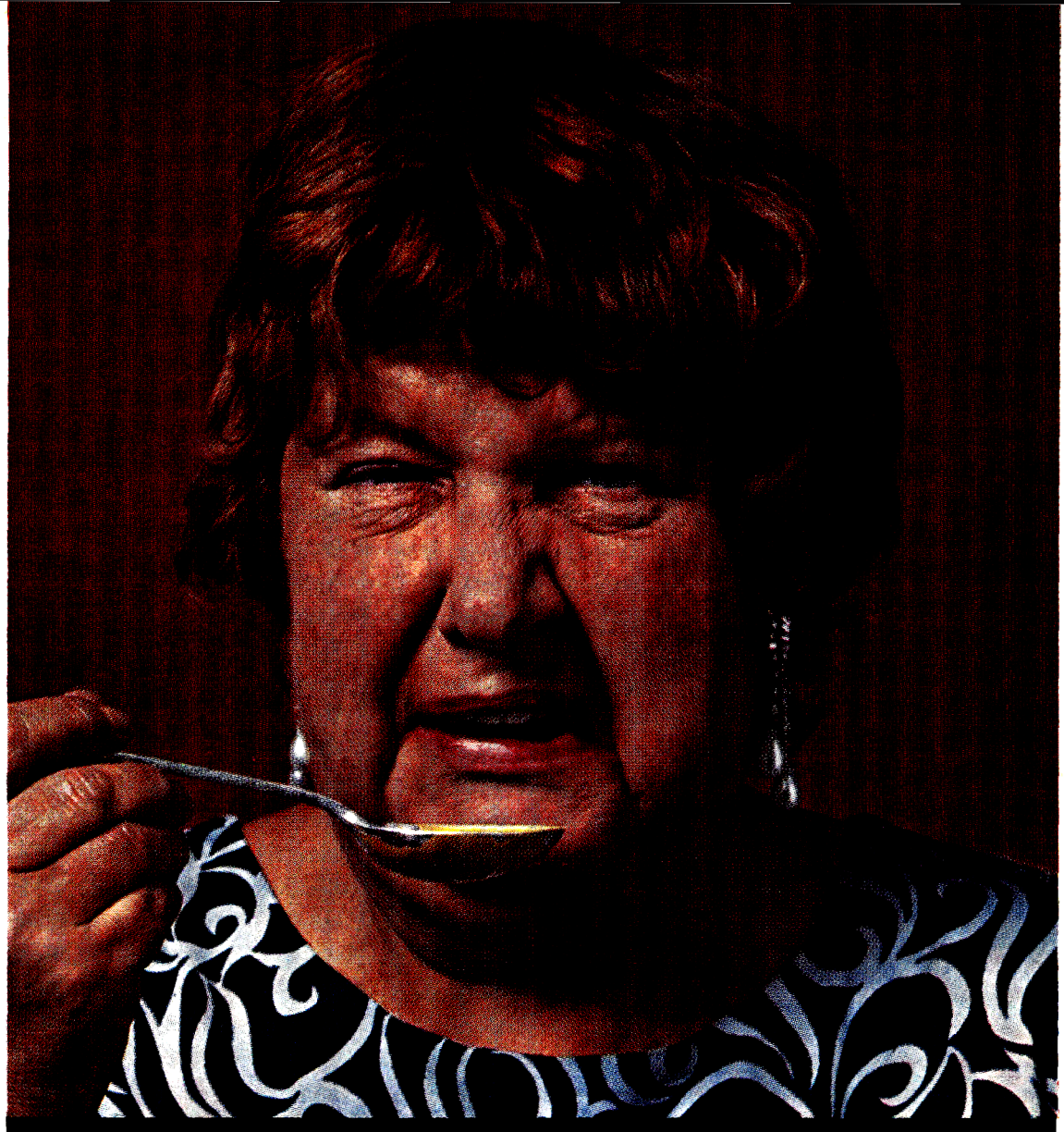

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JOURNAL OF FOOD TECHNOLOGY: NOTICE TO CONTRIBUTORS

The Journal of Food Technology publishes original contributions to knowledge of food science and technology and also review articles in the same field. Papers are accepted on the understanding that they have not been, and will not be, published elsewhere in whole, or in part, without the Editor's permission. Papers accepted become the copyright of the Journal.

Typescripts (two complete copies) should be sent to the Editor, Dr E. H. Steiner, British Food Manufacturing Industries Research Association, Randalls Road, Leatherhead, Surrey. Papers should be typewritten on one side of the paper only, with a 1½ inch margin, and the lines should be double-spaced. In addition to the title of the paper there should be a 'running title' (for page headings) of not more than 45 letters (including spaces). The paper should bear the name of the author(s) and of the laboratory or research institute where the work has been carried out. The full postal address of the principal author should be given as a footnote. (The proofs will be sent to this author and address unless otherwise indicated). The Editor reserves the right to make literary corrections.

Arrangement. Papers should normally be divided into: (a) Summary, brief, self-contained and embodying the main conclusions; (b) Introduction; (c) Materials and methods; (d) Results, as concise as possible (both tables and figures illustrating the same data will rarely be permitted); (e) Discussion and conclusions; (f) Acknowledgments; (g) References.

References. Only papers closely related to the author's work should be included; exhaustive lists should be avoided. References should be made by giving the author's surname, with the year of publication in parentheses. When reference is made to a work by three authors all names should be given when cited for the first time, and thereafter only the first name, adding **et al.**, e.g. Smith **et al.** (1958). The '**et al.**' form should always be used for works by four or more authors. If several papers by the same author and from the same year are cited, a, b, c, etc., should be put after the year of publication, e.g. Smith **et al.** (1958a). All references should be brought together at the end of the paper in alphabetical order. References to articles and papers should mention (a) name(s) of the author(s); (b) year of publication in parentheses; (c) title of journal, underlined, abbreviated according to the *World List of Scientific Publications*, 4th edn and supplements; (d) volume number; number of first page of article. References to books and monographs should include (a) name(s) and initials of author(s) or editor(s); year of publication in parentheses; (b) title, underlined; (c) edition; (d) page referred to; (e) publisher; (f) place.

Standard usage. The *Concise Oxford English Dictionary* is used as a reference for all spelling and hyphenation. Verbs which contain the suffix ize (ise) and their derivatives should be spelt with the z. Statistics and measurements should always be given in figures, i.e. 10 min, 20 hr, 5 ml, except where the number begins the sentence. When the number does *not* refer to a unit of measurement, it is spelt out except where the number is greater than one hundred.

Abbreviations. Abbreviations for some of the commoner units are given below. The abbreviation for the plural of a unit is the same as that for the singular unless confusion is likely to arise.

gram(s)	g	second(s)	sec
kilogram(s)	kg	cubic millimetre(s)	mm^3
milligram(s)		millimetre(s)	mm
(10^{-3}g)	mg	centremetre(s)	cm
microgram(s)		litre(s)	l
(10^{-6} g)	μg	millimetre(s)	ml
nanogram(s)		pound(s)	lb
(10^{-6} g)	ng	gallon(s)	gal
hour(s)	hr	milliequivalent	mEq
minute(s)	min	R_F values	R_F

Figures. In the text these should be given Arabic numbers, e.g. Fig. 3. They should be marked on the backs with the name(s) of the author(s) and the title of the paper. Where there is any possible doubt as to the orientation of a figure the top should be marked with an arrow. Each figure must bear a reference number corresponding to a similar number in the text. Photographs and photomicrographs should be unmounted glossy prints and should not be retouched. Line diagrams should be on separate sheets; they should be drawn with black Indian ink on white paper and should be about four times the area of the final reproduction. Lines and lettering should be of sufficient thickness and size to stand reduction to one-half or one-third. Letters and numbers must be written lightly in pencil. Whenever possible, the originals of line diagrams, prepared as described above, should be submitted and not photographs. The legends of all the figures should be typed together on a single sheet of paper headed 'Legends to Figures'.

Tables. There should be as few tables as possible and these should include only essential data; the data should not be crowded together. The main heading should be in capitals with an Arabic number, e.g. TABLE 2. Each table must have a caption in small letters. Vertical lines should not be used.

Page proofs will be submitted to the contributors for minor corrections and should be returned to the Editor within 3 days. Major alterations to the text cannot be accepted.

Offprints. Fifty offprints will be issued free with each paper but additional copies may be purchased if ordered on the printed card which will be sent to the senior author with the proofs.

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