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Nutritional effects of food processing

A. E. BENDER

Introduction

The preparation and preservation of foods involve a great number of different processes, most of which have an effect, not always deleterious, on nutritional value. Losses, when they do occur, vary enormously with the conditions and it is not possible to generalize about the effects of even such an obviously destructive agent as heat. Although heat usually causes some destruction of vitamins and protein, under certain conditions heat can maintain nutritive value by inactivating enzymes that would otherwise cause damage. Heat may even improve nutritive value by liberating nutrients from otherwise unusable complexes. The improvement in the nutritive value of soya after mild heat treatment has long been known (Longenecker, Martin & Sarett, 1964; Van Buren *et al.*, 1964).

Perspective

Such nutritional losses as occur must be viewed in perspective, as they may be of great or little importance. For example, considerable destruction of vitamin C accompanies heating of milk and its exposure to light, but as the amount initially present in milk is small, and as milk is not regarded by nutritionists as a significant dietary source of the vitamin, such losses are not important. On the other hand, half of the average vitamin C intake in Great Britain in winter comes from potatoes, and losses in processing could be serious to individuals subsisting on a marginal intake of the vitamin.

Again, it has been shown recently (Vipond, Robertson & Tapsfield, 1964) that the meat of broiler chickens contains less vitamin B1 than does the meat of free-range chickens, but as chicken meat supplies less than 1% of the vitamin B1 of the average British diet such a difference has no importance.

Another factor that must be taken into account is the dependence of the consumer on the particular food under discussion. An infant may be relying largely or even solely on a particular manufactured food which must therefore contain an adequate supply of the required nutrients; an elderly person may be living on a strictly limited number of foods, and so processing losses in one of these few may become important to

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him. On the other hand, the average adult in the well-fed communities is consuming possibly a hundred different foods and so it may be of little importance if any one of these suffers nutrient damage—unless that food happens to be the major source of a nutrient as in the case of the potato mentioned above.

To assist in putting some of these factors into perspective the contributions made by different foods to the average British intake of nutrients are shown in Table 1 (taken from Domestic Food Consumption and Expenditure,1965).

TABLE 1. Major sources of nutrients in the average British diet: Per cent of the total daily intake obtained from various foods

	Protein	Vit. A	Vit. B1	Vit. B2	Nicotinic acid	Vit. C	Vit. D
Milk (all forms)	19·0	11·6	12·8	38·1	3·3	9·3	7·6
Cheese	4·2	3·8	0·2	3·6	0·4	—	1·5
Meats	26·1	23·5	24·5	19·7	39·3	1·3	0·9
Fish	4·7	0·3	0·9	1·9	3·7	—	25·4
Butter	—	16·4	—	—	—	—	11·4
Margarine	—	9·1	—	—	—	—	33·6
Potatoes	4·8	—	14·5	7·2	13·8	32·7	—
Other vegetables	4·0	19·8	6·8	3·6	4·1	18·2	—
Fruit (incl. tomatoes)	1·0	5·2	3·3	2·0	2·8	35·0	—
Cereals (incl. bread and cakes)	29·5	1·4	32·7	7·8	28·5	—	—

From Domestic Food and Expenditure: 1963; H.M.S.O., 1965.

The effects of processing vary with, among many other factors, the type of food, the duration and severity of the process and the size and condition of the portions of food. It has even been shown that the rate of loss of vitamin C from the same vegetable differs with different varieties (Holmes, Spelman & Whetherbee, 1949; Hopp & Merrow, 1963). That is why some experimental reports appear to be contradictory. However, a brief survey of the properties of the nutrients offers some guide to their behaviour under a variety of circumstances.

The most sensitive of the nutrients are the vitamins; several, but not all, may suffer considerable loss on processing. Proteins mostly do not suffer damage unless they are severely heated or stored for long periods. Losses of carbohydrate and mineral salts occur in wet processing by leaching but can usually be ignored.

Vitamins

Losses of vitamins in fresh foods can occur through careless handling and they can be lessened by cooling to reduce enzyme activity (but not enough to freeze the cells), by avoiding bruising and by reducing the time between harvest and distribution. This applies to carotene as well as to vitamin C. For example Ezell & Wilcox (1962) found as much as 75% loss of carotene in kale and collards in 4 days at 70°F under conditions of rapid wilting. At 50°F there was 20% loss on slow wilting and 30% on rapid wilting. When wilting was prevented and kale stored at 32°F there was a 25% loss of carotene in 4 weeks. The same loss could occur in 1 day at 70°F. The greatest losses during processing are caused by the leaching out of water-soluble vitamins in operations such as washing and blanching (Adam, Horner & Stanworth, 1942; Lee, 1958). Obviously the smaller the state of sub-division of the food the more is leached out, and the amount lost also depends on the type of food, and the time and temperature of the water treatment. Steam blanching causes smaller losses than hot-water treatment. For example in one experiment the loss of soluble sugars was 7% in 3 min in hot water but only 3% by steam treatment. The losses in peas were three times as great as in beans (Horner, 1936–37). Losses from small pieces of carrots, swedes and sprouts were 32–50% of vitamin C and from large pieces 22–33%.

Under adverse conditions losses of vitamins during blanching can amount to 70%; under favourable conditions they can be as little as 5–10% (Harris & von Loesecke, 1960). Once the food has been treated the subsequent losses are usually small if it is kept cool, and, in the case of dry foods, if the moisture content is kept low.

The exclusion of air during storage is beneficial (Harris & von Loesecke, 1960), for example dried cabbage stored in nitrogen or carbon dioxide lost only one third of the vitamin C lost when stored in air. The exclusion of air also inhibits oxidative rancidity and protects vitamins A, D and E, as well as C and biotin, which are destroyed by peroxides.

Vitamin A and carotene

Both vitamin A and carotene are stable to mild cooking and processing but are destroyed at high temperatures in the presence of oxygen (Harris & von Loesecke, 1960). The fact that vitamin A can be distilled in the molecular still illustrates its resistance to heat in the absence of air.

Both vitamin A and carotene are oxidized by fat peroxides and are therefore destroyed under conditions which also favour the oxidation of fats including the presence of traces of copper and, to a lesser extent, of iron. They are protected by the antioxidants naturally present in many foods, but being long-chain compounds containing conjugated double bonds both these substances are inherently unstable when pure. Thus when synthetic vitamin A and carotene are added to foods for enrichment they require

protection. Not being water-soluble they are not extracted into cooking water. They are unstable to acid but can withstand boiling in strong alkali, as, for example, during the saponification used as a means of extraction before analysis.

Isomerization during canning can lead to losses, but these are very small—e.g. in canned carrots losses were found to be 7–12% in 4 years storage (Weckel *et al.*, 1962). However, in dehydrated carrots there is a degradation of carotene causing an unpleasant flavour (Falconer *et al.*, 1964). An increase in carotene content has been reported in stored fresh carrots (Brown, 1949). About 60% of the carotene is retained in carrots dried by explosive puffing and also in ordinary air drying (compared with blanched fresh carrots) and 80% is retained in freeze-dried carrots. There was no significant loss on reconstituting and cooking these foods (DellaMonica & McDowell, 1965).

The stability of vitamin A is indicated by a recent report showing that braised liver, with an internal temperature of 170°F, retained 90–100% of the vitamin (Kizlaitis, Deibel & Siedler, 1964).

Maqsood, Haque & Kham (1963) reported the losses of vitamin A in enriched ghee and vanaspati. Frying at 200°C caused a 40% loss in 5 min, 60% in 10 min and 70% in 15 min. Boiling in water caused 16% loss in 30 min, 40% in 1 hr and 70% in 2 hr.

There is some loss of vitamin A added as a supplement to powdered foods. Rao *et al.* (1964) and Chandrasekhara *et al.* (1964) stored similar protein-rich infant foods in sealed tins at 37°C and observed a loss of 25% of the vitamin A after 9 months, as well as 15% of the vitamin B1 and 30% of the C.

*Vitamin B*1

Vitamin B1 is one of the more labile of the vitamins and can suffer considerable damage during food processing. As are all the water-soluble vitamins, B1 is mostly lost from foods by leaching, and losses are greater with finely divided foods immersed in large volumes of water for long periods. It is stable to acid, even at boiling point and up to 120°C, but unstable at neutrality and at alkaline pH. Thus there can be much loss during baking if alkaline baking powders are used. Even the mild alkalinity of many natural waters can result in the destruction of much vitamin B1, for example in boiled rice (Roy & Rao, 1963). Distilled water caused a negligible loss, cooking in tap water caused 8–10% loss, and cooking in well water up to 36% loss. When large volumes of water are used, e.g. 10–15 volumes as in the preparation of rice gruel, a common practice in India, losses can be as high as 80%. This is due to alkalinity not to leaching as the same preparation in distilled water loses only 5% of the B1.

Thiamine is unstable to oxygen, is destroyed by sulphur dioxide, but unaffected by light. The baking of bread can result in the loss of 15–30% of the thiamine, mostly in the crust, but after baking it is stable (Zaehringer & Personius, 1949). Toasting,

for periods of 30–70 sec, has been shown to result in a loss of 10–30% of the thiamine present (Downs & Meckel, 1943).

Cereals stored as whole grain can suffer a loss of thiamine depending on the moisture content. In one series of observations there was a 30% loss in 5 months storage at the relatively high moisture content of 17%, 12% loss at the normal moisture content of 12%, and no loss after 1 year storage when the moisture content had been reduced to 6%. It appears to be more stable in rice, which showed complete stability for periods up to 2 years (Bayfield & O'Donnell, 1945; Cuendet *et al.*, 1954).

Thiamine-fortified white flour can lose up to about 10% of its thiamine under favourable storage conditions. Fortification is usually carried out with thiamine hydrochloride but the mononitrate is more stable and its rate of destruction in flour is only half that of the hydrochloride (Hollenbeck & Obermeyer, 1952).

The losses in meat during cooking vary with the size of the cut, fat content, and other factors, but average about 15–40% on broiling, 40–50% on frying, 30–60% on roasting and 50–75% on canning (Harris & von Loesecke, 1960). (See also Tables 8, 9 and 10.)

There are no losses of vitamins B1 and B2 on chilling poultry and the time it is stored chilled has no effect on the retention of these vitamins when subsequently cooked (Pudelkewicz *et al.*, 1963; Rowe, Mountney & Prudent, 1963).

Fish loses up to 50% of its thiamine on boiling and 75% on canning. Even eggs lose some on cooking—one report gives the loss as 9% when scrambled and 29% when boiled (Lane, Johnson & Williams, 1942; Everson & Souders, 1957).

Milk, which is not an important dietary source of B1, loses 10% when spray-dried, 20–30% when roller-dried, 3–20% on pasteurization and 30–50% on sterilization (Harris & von Loesecke, 1960; Kon, 1960).

Vitamin B2

Vitamin B2 is stable to oxygen and to acid conditions but unstable to light and to alkali. Heat alone is not harmful so that foods may be fortified with added B2 without loss so long as the foods are neutral or acidic. Representative losses are shown in Tables 5 and 9.

Its sensitivity to light leads to the destruction of both vitamin B2 and the vitamin C in milk. About 50% of the B2 can be destroyed in 2 hr by exposure to bright sunlight, and 20% on a dull day. The vitamin B2 is converted into lumiflavine and this destroys the vitamin C (Harris & von Loesecke, 1960).

In recent times it has become common practice to offer milk for sale in cartons in supermarkets and the effect of its exposure to fluorescent lamps in this situation has been thoroughly investigated (Dunkley, Franklin & Pangborn, 1962). These authors found that milk in paper cartons stored near the fluorescent lamps lost 24% more vitamin C (and had a more intense 'light flavour') than cartons that were partially

shielded. The losses of vitamin C in clear glass bottles were 2·2 mg/l/hr, and in fibreboard cartons 1·3 mg/l/hr. The minimum time for detectable development of flavour was 20 min for milk packed in clear glass bottles, 5 hr in amber glass and 1–15 hr in the various types of fibreboard cartons.

The destruction of vitamins B2 and C was directly related to the radiant power emitted between wavelengths of 400 and 550 μ, but this relation did not hold for the development of 'light flavour'.

Although the vitamin C of milk is not usually considered to constitute a significant portion of the total vitamin C content of the diet, the accompanying development of undesirable flavour may be a considerable detriment and the authors discuss methods of reducing the losses.

Nicotinic acid

Nicotinic acid is one of the most stable of the B vitamins, being unaffected by light, heat, oxygen, acid or alkali. The only losses are incurred by leaching into the processing water.

In many cereals nicotinic acid occurs in bound and biologically unavailable form. Clegg (1963) showed that 77% of the nicotinic acid of commercial flour is present in the bound form and the amount bound fell linearly with increasing pH during baking, being 70% at pH 6·8 and zero at pH 9·6. Apart from the pH effect there is a partial release of nicotinic acid by lengthening baking time.

It has been observed many times that the treatment of maize with lime produces better growth in animals (Kodicek *et al.*, 1956; Squibb *et al.*, 1959) and the effect is believed to be due either to the liberation of bound nicotinic acid or to an increase in the availability of amino acids. The same effect as lime treatment can be produced by heat and pressure (Bressani, Castillo & Guzman, 1962).

The amount liberated by heat treatment can vary considerably in different foods (Rajalakshmi, Nanavaty & Gumashta, 1964). In roasted maize the free nicotinic acid increased about 30 times, peas three times, Bengal gram by about one third and rice by only one tenth. Pressure cooking of maize for 5 min at 15 lb or baking into chappaties increased the free nicotinic acid.

Vitamin C

Vitamin C is one of the most readily destroyed of the vitamins and its retention is often used as an index of the severity of processing and of storage conditions. If it is well retained then it is unlikely that there has been any serious loss of other nutrients.

It is oxidized in air under alkaline but not acid conditions and the oxidation is powerfully catalysed by traces of copper. For the estimation of vitamin C in foods the tissue is usually macerated under metaphosphoric acid which both inactivates the oxidizing enzyme and complexes with any copper present.

The greatest losses of vitamin C are due to leaching into the processing water. The amount lost depends, among other factors, on the amount of water rather than the time of treatment. For example, vegetables covered with water may lose 80% of the ascorbic acid, half-covered 60%, and quarter-covered 40%. The amount destroyed may be quite small compared with the leaching losses. For example, a sample of cabbage cooked in 10 volumes of water lost 10% by destruction and 80% into the cooking water; 10% was retained in the food. Representative losses are shown in Tables 2, 8 and 9.

TABLE 2. Percentage retention of vitamin C during processing

POTATOES, peeled and boiled	50–70
boiled without peeling	60–80
baked in skin	60–80
fried	65–75

(Stored potatoes lose about one sixth of the vitamin C content per month)

From *The Composition of Foods* by R. A. McCance and E. M. Widdowson (1960) H.M.S.O.

PEAS: percentage retention after the stages indicated

Fresh		Frozen		Canned		Air-dried		Freeze-dried	
—		Blanching	75	Blanching	70	Blanching	75	Blanching	75
—		Freezing	75	Canning	63	Drying	45	Drying	70
		Thawing	71						
Cooking	44	Cooking	39	Heating	36	Cooking	25	Cooking	35

After Mapson (1956).

The vitamin C content of fruits and vegetables is a useful index of freshness as it is slowly lost during storage, particularly if the food has been bruised during handling, and at elevated temperatures. Bruising and wilting allow the ascorbic acid oxidase to come into contact with its substrate and destroy the vitamin.

Destruction starts as soon as the crop is taken from the ground. For example, kale can lose 1·5% of its vitamin C per hour, and about one third in 24 hr. Losses are reduced by factors that reduce wilting such as high humidity and cool storage conditions, but freezing may burst the cells and bring the enzyme into contact with the vitamin. This is an instance where rapid heat applied at the right time can inactivate the destructive agent and protect the vitamin. This is often achieved by blanching before, say, drying or freezing, but some vitamin C is lost into the processing water (Adam *et al.*, 1942). It has been shown that vegetables blanched by electronic heating, sufficient to inactivate the enzymes, show no loss of vitamin C (Proctor & Goldblith,

1948; Moyer & Stotz, 1947) but the method may not be superior to steam blanching (Hard & Ross, 1956).

Vitamin C keeps remarkably well in frozen foods, apart from the losses incurred in the preliminary blanching process. For example, Noble & Gordon (1964) showed that broccoli and beans, cooked fresh, retained 50% of their vitamin C, while the blanched product, stored for 6 months at 0°F, retained 30% of the vitamin after cooking. For asparagus, sprouts and cauliflower the figures were 60% and 40% respectively. (See also Table 8.)

Vitamin C is extremely stable when protected from air as when in cans or bottles. There may be an immediate loss due to dissolved and headspace air. In cans the residual oxygen is normally used up in the electrochemical process of corrosion and so disappears rapidly in plain cans and much more slowly in lacquered ones. In consequence there is greater loss of vitamin C in lacquered cans than in plain ones (Adam, 1941). In bottles the residual oxygen is all available to oxidize the vitamin C. In theory 3·3 mg of ascorbic acid are destroyed by 1 ml of air (Bender, 1958a). After the residual oxygen has been removed in this way there may be no further loss of the vitamin for periods of many months. It is common practice in the United States to allow a loss of 8–15 mg of vitamin C per 4 oz of fruit juice to cover processing and 12 months storage—this is an absolute amount irrespective of the concentration of the vitamin.

Some preparations, however, exhibit great instability. For example, Noel & Robberstad (1963) found that orange juice stored at 40°F lost 5–30% of the vitamin C in 16 days, while apple juice lost 50% in 4–8 days and 95% in 16 days. Pelletier & Morrison (1965) reported so great a lability of vitamin C in a number of orange-flavoured drinks (as distinct from natural fruit juices) that they stated that many fruit drinks on the market in Canada are unsatisfactory and not under proper control. Thirteen out of twenty liquid preparations examined were below label potency and the vitamin content fell rapidly when they were opened. The authors concluded that the fruit-flavoured drinks were either low in vitamin content when manufactured or subjected to excessively long storage periods. Pineapple juice was found to be up to 55 months in the distribution channels with consequent vitamin loss (Darroch & Gortner, 1965).

Sulphur dioxide is useful in protecting the vitamin and Chmielnicka (1965) showed that 80 mg per cent inhibited the peroxidase in fresh tomato pulp and protected the vitamin C.

In an examination of hospital diets, Platt, Eddy & Pellett (1963) showed that when peeled potatoes were soaked in water overnight they lost 45–60% of their vitamin C content. The authors point out that this loss is due to the damage caused by mechanical peeling as leaching does not occur if the tissues are undamaged. Hand peeling followed by 14 hr soaking in water caused an average loss of 9% of the vitamin C, machine peeling for 1 min followed by soaking caused an 18% loss, and if the peeling machine was operated for 3 min it caused 46% loss.

Vitamin D

Vitamin D is extremely stable and there is little or no loss through processing and storage. However, when vitamin D-enriched milk is dried there is loss of 25–30% of the vitamin.

Folic acid and pyridoxine

In general, the vitamins already discussed, A, B1, B2, nicotinic acid, C and D, are those of most importance to the nutritionist, as dietary shortages still exist, even if they are rare in the Western world. The other vitamins are regarded as being of medical rather than nutritional interest as dietary deficiencies of them are so rare. However, folic acid and pyridoxine may possibly need to be considered among the vitamins of nutritional interest.

There is evidence of a deficiency of pyridoxine (vitamin B6) in pregnancy as indicated by the inability of some individuals to metabolise completely a test load of tryptophan until extra pyridoxine had been administered (Hunt, 1957; McGanity *et al.*, 1949; Ranke *et al.*, 1958). The United States Food and Drug Administration has questioned the adequacy of the pyridoxine content of American diets and discussed the possibility of adding it to foods (Harris, 1959).

In considering this last point, Bunting (1965) examined the stability of vitamin B6 when added to foods. Maize meal, stored for 1 year at 100°F and 100% RH, retained 90–95% of the added vitamin; when the maize was baked into bread recovery was 100%. Macaroni retained 100% of the added vitamin on storage but lost 50% into the water when the macaroni was cooked.

It has been shown in several foods that pyridoxine is not stable to heat (Woodring & Storvick, 1960) and that, for example, 45–70% can be lost during the evaporation of milk, with further losses on storage (Davies, Gregory & Henry, 1959).

There is recent evidence that many pregnant women suffer from anaemia due to a shortage of folic acid (Review, *British Medical Journal*, 1964). This is possibly due to the increased demands of the foetus for folic acid for blood formation. One clinical report stating that in the last week of pregnancy 60% of the subjects examined had low blood levels of folic acid indicates that the intake of this vitamin must be safeguarded, at least in pregnant women.

Folic acid is stable to heat in an acid medium but is rapidly destroyed in alkaline or neutral conditions. It is believed that about half is lost in cooking (Bannerjee & Chatterjea, 1964).

Mineral salts

The loss of minerals by leaching into the processing water is not a matter of nutritional importance. The reverse process in which the foods absorb minerals from the water

may be of greater significance in certain instances. It can be a serious problem in the preparation of low-sodium foods for dietetic purposes. In one series of observations the sodium content of peas, which was initially 1·7 mg/100 g, was increased to 12 mg/100 g. For this reason water containing more than 10 mg of sodium per 100 ml is considered unsuitable for canning low-sodium foods.

Calcium can also be absorbed by foods from hard water (Horner, 1936–37) but the amounts involved are not great enough to be nutritionally significant.

Proteins

Damage to proteins may be of two main types, (a) destruction of amino acids and (b) combination of part of one or more of the amino acids in a linkage that is not hydrolysed during digestion. There can also be small losses of soluble proteins leached into the processing water but this mostly affects vegetables, which are not an important source of protein in Western diets.

High temperatures such as those involved in roasting are necessary before the destruction of amino acids occurs, and since only the surface layers of the food reach such temperatures losses from this cause are small. The combination of amino acids into an unusable form is the more significant cause of loss. Combination can occur between the amino group of the amino acid and reducing groups present in the foodstuff, between the amino group and carbonyl compounds formed by oxidation of fats, and carbon-nitrogen links can be formed between proteins (Lea, 1958; Ellis, 1959).

These linkages cannot be hydrolysed by the digestive enzymes—hence the term 'unavailable amino acids'—but they are hydrolysed by the acid treatment that precedes the usual chemical analysis for amino acid content. Chemical analysis may therefore provide misleading information as it will yield the total amount of each amino acid present, some of which may not be biologically available. It becomes necessary, therefore, to use biological methods (Bender, 1958b).

Maillard reaction

The combination of lysine, i.e. non-enzymic browning or the Maillard reaction, has been the one most thoroughly studied but other amino acids are also affected, in some instances to a greater extent than lysine (Iriarte & Barnes, 1966; Ford, 1962). Lysine becomes combined by the reaction between its epsilon amino group projecting from the protein chain and reducing substances, such as glucose, present in the food. The loss of available lysine can occur with relatively small amounts of reducing substances. For example dried egg, containing 83% protein and 3% glucose, will deteriorate both in nutritional value and in flavour during prolonged storage. If this small amount of glucose is removed (by treatment with yeast or glucose oxidase) the egg can be stored without harm (Harris & von Loesecke, 1960).

The first stages in the Maillard reaction result in the formation of a colourless compound; the brown coloration is a later reaction. Thus it is possible for food to deteriorate during processing or storage without becoming discoloured. Lea & Hannan (1949) demonstrated a marked loss of biological activity of a casein–glucose mixture at room temperature without the development of a brown colour. Carpenter *et al.* (1957) heated cod fillets for 729 hr at 85°C and found them little darker than after 27 hr but much lower in nutritive value. The addition of 5% glucose produced a much darker colour after 27 hr heating compared with 729 hr without added glucose, but both samples had the same nutritive value. The reaction can also cause unpalatability in foods that nevertheless retain their full nutritive value. Regier & Tappel (1956) damaged freeze-dried beef to such an extent as to render it completely unacceptable, yet there was no loss in nutritive value as determined by tests on chicks.

Sulphur dioxide can sometimes prevent the formation of the brown colour but not the fall in nutritive value. In dried fruits, where the main reaction is between carbonyl and amino groups (Harper & Tappel, 1957), sulphur dioxide protects the vitamin C but has only a limited effect on the development of the brown colour (Draudt & Huang, 1966).

The conditions for protein damage include the presence of moisture, the presence of reducing substances and the application of heat or prolonged storage. It is possible to subject dry proteins to a high temperature without detectable damage. Miller (1956) heated dry fish for 24 hr at 105°C without nutritional damage. With 9% water added the net protein utilization fell after 24 hr heating from 72 to 43. Lea & Hannan (1949) showed that there is a critical moisture content at which damage is most severe, namely 70% relative humidity. Carpenter *et al.* (1962) showed with herring press cake that the maximum damage by binding of lysine occurred at moisture contents between 4 and 12% and that methionine, arginine and tryptophan were also bound.

The effect of reducing substances was clearly shown by Halevy & Guggenheim (1953). Wheat gluten heated alone was undamaged with a biological value of 55. When heated with glucose the value fell to 18. When the damaged material was supplemented with lysine the BV rose to 63, indicating that lysine was the main seat of damage. Another example is that of a cake mix composed of flour, egg, yeast and lactalbumin (Block *et al.*, 1946). The unheated mixture had a protein efficiency ratio of 3·5; when baked for 15–20 min at 200°C the value fell to 2·4; toasting for 40–60 min at 130°C to produce a rusk further reduced the PER to 0·8. The damage was solely to lysine since the addition of this amino acid to the rusk restored the PER to its original value. It should be noted that some workers have measured nutritive value as Biological Value, BV (per cent of absorbed nitrogen retained), or Net Protein Utilization, NPU (per cent ingested nitrogen retained), others have measured the Protein Efficiency Ratio, PER (weight gain per gram of protein eaten). The last measure ranges from zero to about 4·5 for 'perfect' proteins (Bender, 1958b).

Mild heating is generally harmless. For example, casein may be unaffected by subjection to temperatures of 100–110°C. Heating for 8 hr at 120°C has been shown to cause the same degree of damage as 2 hr heating at 130°C, and at a given temperature the amount of damage is proportional to the time of heating (Greaves, Morgan & Lovern, 1938). Blood plasma, containing less than 0·1% carbohydrate, is stable to heating for 27 hr at 85°C but loses 26% of the available lysine at 115°C. This destruction is believed to be due to protein–protein interaction (Carpenter *et al.*, 1962).

Underwood, Lento & Willits (1959) showed that amino acids react at different rates under various conditions. In model systems consisting of amino acids heated with glucose no brown colour was produced at pH 5; at pH 6 only the alpha-omega diamino acids gave a brown colour: at pH 6·5 omega amino acids of four carbon chain length gave a colour: at pH 6·8 omega-amino acids of chain length shorter than four carbon atoms became reactive: and at pH 7 all the amino acids tested were reactive (including aminobutyric, norvaline, ornithine and norleucine).

Damage to cereal proteins

Most cereals, even before processing, are limited in their nutritive value by their lysine content and it is the lysine that suffers damage. Bread, for example, suffers a loss of 10–15% of its available lysine during baking, with a further loss of 5% on staling. Toasting causes a loss of 5–10% (Rosenberg & Rohdenburg, 1951).

TABLE 3. Per cent availability of amino acids in wheat, flour and bread (biological assay)

	Wheat	Flour	Bread
Methionine	79	85	99
Tryptophan	88	88	97
Isoleucine	73	91	70
Phenylalanine	76	90	77
Valine	76	78	76
Leucine	94	75	100
Threonine	90	100	100

From Hepburn *et al.* (1966).

The availability of seven of the essential amino acids in wheat, flour and bread was measured biologically by Hepburn, Calhoun & Bradley (1966). The results (Table 3) indicate that in most instances the availability was greater in bread than in the original wheat.

Supplementation of the protein of flour with skim milk in a high-protein biscuit prepared for use in Uganda provided the conditions for nutritional loss (Clegg, 1960). Baking reduced the available lysine from 4 g to 2 g per 16 g nitrogen due to linkage with the lactose of the skim milk. When skim milk was replaced by casein there was no loss of available lysine.

As bread is limited by lysine suggestions have been made that synthetic lysine should be added to improve the nutritive value. Biological assay (Jansen, Ehle & Hause, 1964) shows that lysine is lost progressively during baking at times from 0 to 50 min. Thirty minutes baking at 450°F causes a loss of 30% of the added lysine. The loss of added lysine is no greater than that of the lysine naturally present in the wheat; losses are increased by using milk powder as a supplement. A chemical assay showed a 9–13% loss of lysine in biscuits baked without sugar at 450°F for 20 min (Clark *et al.*, 1959).

Breakfast cereals are subjected to a variety of treatments. The heating, rolling and flaking processes do not affect the protein, it is the process of 'explosion' puffing that reduces its nutritive value. Temperatures around 200°C and pressures of 100–200 lb are used and can cause a fall in protein efficiency ratio from 1·6 to 0·3–0·5 (Murlin, Nasset & Marsh, 1938; Stewart, Hensley & Peters, 1943; Kuether & Myers, 1948; Sure, 1951). As these foods do not normally contribute much protein to the diet, and as they are usually eaten with milk which supplements the deficiency of lysine,

TABLE 4. (a) Nutritive value of U.S. breakfast cereals (tested on rats)

	Protein content (%)	Protein efficiency ratio
Quick Oats	17	1·6
Instant Ralston	13·8	1·5
Cerevim	18	1·5
Pablum	15	1·3
Cream of Wheat	11·2	1·2
Cheerios	14·9	1·1
Bran Flakes	9·5	0
Grape Nuts	9·2	−0·3
Kix	7·8	−0·9
Puffed Wheat	13·2	−2·5

From Sure (1951).

TABLE 4 (*Continued*)

(b) Heat damage to oat preparations

	Protein efficiency ratio
Drum dried (boiled 15 min, dried 15 sec, 130°C)	1·6
Rolled oats	1·5
Oven exploded (cooked 1–2 min at 100 lb, dried 1–2 min at 200°C)	1·6
Preparation of 75% oat, 20% corn and rye (boiled, dried, heated 80–100 lb, 190–232°C for 52–62 min, exploded)	0·5
Puffed oats (heated 5 min, 122°C, then live steam 200 lb, 198°C, 2 min, and then puffed)	0·3

From Stewart *et al.* (1943).

(c) Nutritive value of breakfast cereals assayed on man (Egg replacement value)

Pre-cooked oats	77
Granulated wheat and wheat germ	73
Wheat endosperm	85
Torn wheat	66
Flaked wheat	73
Toasted whole wheat	66
Inflated wheat	69

From Murlin *et al.* (1938).

the losses are not important. However, such processes would clearly not be applicable to protein-rich preparations intended to make a substantial contribution to the protein intake.

Protein damage on drying

Foods heated dry or with water generally suffer little damage to the protein; as Lea & Hannan (1949) showed, the critical moisture content at which damage is most severe is 70% RH. Damage can occur, however, while foods are undergoing drying. The temperature and the time during which high temperatures are maintained at the critical moisture content appear to be the controlling factors, as well as the presence of reducing substances which can render the amino acids unavailable.

De Groot (1963) compared the effects of drying in hot air, under vacuum and by accelerated freeze drying (AFD). Under the particular conditions of the experiment hot-air drying caused no damage to the proteins of Lima (butter) beans or chicken. Green beans showed a small fall both in biological value and digestibility on canning but not on drying in hot air, vacuum or by AFD. AFD had no effect on the proteins of sweet corn, beef, fish, fish–maize meal patties, cheese or an egg–milk–fat product.

The fall in the nutritive value of green beans on canning may be explained by the presence of sugar. Powrie & Lamberts (1964) found that haricot beans suffered a considerable loss on canning if glucose was added (BV 33 falling to 15, digestibility 63% falling to 47%) but much less damage if sucrose or a mixture of glucose and sucrose was added.

It must be borne in mind that a biological assay measures only the limiting amino acid and that it is possible to suffer damage to a non-limiting amino acid without this showing up in the normal single biological assay. Biological assay can show damage to non-limiting amino acids if assays are carried out on proteins supplemented with sufficient amino acids to make the damaged one limiting. For example, milk is limited by methionine and has a relative excess of lysine. Slightly overheated milk may suffer damage to the lysine but so long as the methionine is undamaged the biological value is unaffected. Only when the available lysine is reduced so far that it becomes the limiting amino acid in place of methionine does the biological assay show a fall.

Desirable browning

The Maillard reaction can make a positive contribution to food technology by producing desirable colours, flavours and aromas. It is the cause of much of the colour and flavour developed in baking, roasting, frying and toasting, and foods such as biscuits, breakfast cereals, meat extract, and malt extract owe much of their flavour to this reaction (Linko & Johnson, 1963). Although there is some loss of amino acids when these flavours are produced the loss is not usually of much importance and is accepted as a price worth paying. The Maillard reaction can be exploited for particular purposes by the addition of reducing agents to the food. For example, the inclusion of milk powder in a loaf of bread permits more even toasting at a lower temperature because of the presence of lactose. Another example is the production of chicken and meat-like flavours by heating amino acids with pentose sugars and aldehydes.

Cysteine and ribose produce a pork-like flavour, ribose with a mixture of amino acids produces a beef-like flavour, lysine or cystine with furan produces meat-flavour. The specific flavours of breakfast cereals, baked bread, roasted coffee and soya flour are said to be due to the reaction of particular amino acids. Glucose heated with glycine gives the flavour of freshly baked bread, with amino butyric acid it gives a maple syrup flavour and with hydroxyproline, the flavour of potato (Herz & Shallenberger, 1960).

Improvement of protein quality

Although heat often causes nutritional damage, heat treatment of proteins can sometimes produce an improvement in nutritive value. This is true of many peas and beans which contain various toxins and substances such as trypsin inhibitors that lower the nutritive value of the food. Mild heat treatment destroys these substances and results in an increase in biological value. Only when the heating becomes excessive, i.e. temperatures greater than about 120°C, does damage occur (Longenecker *et al.*, 1964; Van Buren *et al.*, 1964; Everson & Heckert, 1944; Liener, 1962; Kakade & Evans, 1965). Hackler *et al.* (1965) showed that available lysine is a useful indicator of quality for overheated samples of soya and trypsin inhibitor retention for underheated samples.

There have been several reports of the beneficial effects of heating wheat (Shyamala & Kennedy, 1962; Hutchinson, Moran & Pace, 1964). The latter workers suggested three possible mechanisms, destruction of a trypsin inhibitor, increased palatability for the experimental animals, and improved digestion through destruction of the capacity of the wheat protein to form gluten. Laporte & Tremolière (1962) showed that raw cereal flours—rice, oats, maize, barley, wheat, rye, buckwheat—inhibited the effect of tryspin on milk powder. Cooking these foods for 4 min at 98°C destroyed the inhibitor in wheat, rice and oats but not in the other foods. Hutchinson *et al.* (1964) showed that steaming barley does not have an improving effect and as barley does not form a gluten these authors suggested that the third mechanism mentioned above is the most likely.

Commodities

Meat

In general, meat, during cooking, loses one third of its content of vitamins B1, B6, B12 and pantothenic acid, and less than one tenth of the vitamin B2 and nicotinic acid. During freezing there is a loss of juice in the drip thaw and this can amount to 10% of the water-soluble ingredients. In canned meat losses are 0–10% B2 and nicotinic acid, 20% biotin, 20–30% pantothenic acid and 20–40% B1 (Harris & von Loesecke, 1960). Meyer, Mysinger & Buckley (1963a) and also Meyer *et al.* (1963) examined beef stored for 3 years at 0°F and the effect of ripening prior to storage for 21 days at 34°F. In theory there should be complete stability of the B vitamins under these conditions but there may be changes due to enzyme action. Earlier reports had shown insignificant losses of thiamine, and inconsistent results with riboflavine. These authors observed a significant increase in the thiamine, 36%, in unripened beef but none in the ripened beef. Riboflavine increased 10% in both. Nicotinic acid decreased significantly, 17%, in unripened beef but there was no change in the ripened meat.

Noble (1965), drawing attention to the rather low intakes of vitamins B1 and B2 in certain sections of the U.S. population, pointed out that meat is so good a source of these vitamins that dependable figures for their content in cooked meat are essential

for diet calculations. The effect of braising various cuts of beef, pork and veal was examined. The cuts of beef and veal were cooked in an oven at 149°C for the lengths of time shown in Table 5. Pieces of chuck weighing 6 lb were cooked for 35 min per lb weight and reached an internal temperature of 85°C. The cuts of pork were cooked

TABLE 5. Retention of vitamin B1 and B2 in different cuts of braised meat

	Weight (lb)	Time of cooking (min/lb)	Vitamin B1 (% retention)	Vitamin B2 (% retention)
Beef				
Short ribs	$4\frac{1}{2}$	30	25	58
Chuck	6	35	23	74
Flank steak	$1\frac{3}{4}$	28	30	72
Round (roast)		27	40	73
Round (steak)		18	40	65
Veal				
Chops			38	73
Round steak			48	76
		Total time		
Pork		of cooking		
Chops		50 min	44	64
Spare ribs		2 hr	26	72
Tenderloin		40 min	57	83

From Noble (1965).

at 175°C but the internal temperature was not reported. Table 5 shows that there was a significant variation of retention of vitamin B1 with different cuts of beef, ranging from 25% for chuck and short ribs to 40% for round. In pork the variation was from 26% retention in spare ribs to 57% in tenderloin. Vitamin B2 was more stable but the range of retention values was as great and there were still significant differences between some of the cuts.

In addition to the losses of vitamins meat can suffer protein damage but only under severe conditions of heating. Samples have shown no loss when roasted in an open pan at 163°C (internal temperature 80°C), when browned in the oven for 30 min and processed in the can for 83 min at 121°C or when corned (Mayfield & Hedrick, 1949). Others have found a loss of 30% in biological value on corning but no further loss after 9 years storage (Bender, in press).

Dvorak & Vognarova (1965) showed the relation between time of heating, temperature and water content and the loss of available lysine in beef. After 3 hr heating there was 90% retention at 70°C, 80% at 121°C, 60% at 140°C and 50% at 160°C. The

addition of glucose before heating had a negligible effect. Smoking and salting with nitrite also reduced the available lysine content. Storage of a freeze-dried product for 1 year at 20°C in air and at 4·8% water content caused nearly 50% loss of available lysine. However, in a range of meats, sausages and hams processed in several ways between 80% and 100% of the lysine was available.

It required 2 hr heating at 120°C to damage pork; there was no fall in nutritive value after 1 hr (Wheeler & Morgan, 1958).

Beuk, Chornock & Rice (1949) showed that autoclaved pork (24 hr at 112°C) lost 44% of the cystine by destruction and 70% became unavailable, while 5% of the lysine was destroyed and 45% was unavailable. The other amino acids showed full retention when analysed after acid hydrolysis but only 35–50% were available. Pork heated in water at 110°C for 24 hr and then dried at 100°C for 16 hr showed a fall in net protein utilization from 76 to 41 (Donoso *et al.*, 1962). The net protein utilization was partially restored to a value of 60 by adding methionine. The available lysine fell by 34% and there was a destruction of 20% lysine, 16% methionine and 44% cystine.

Bender (in press) found that two extremely old samples of canned meat showed a considerable fall in nutritive value entirely due to the non-availability of the amino acids. Veal, canned in 1823 and examined in 1959, had an NPU of 27 but a normal amino acid analysis. The available amino acid content was not measured but the fall of nutritive value is presumably due to unavailability.

Milk

The losses of riboflavine and ascorbic acid on exposure of milk to light have already been described. Boiling milk for a few minutes can destroy 20% of the riboflavine but there is no loss of this vitamin during condensing and drying so long as light is excluded. Pasteurization destroys about 20% of the ascorbic acid and 10% of the B1; spray-drying does the same amount of damage and roller-drying slightly more. Sterilized, evaporated milk loses about 60% of the C and 50% of the B1 (Harris & von Loesecke, 1960; Kon, 1960).

Grudskaja (1965) recently showed that all the vitamin C was lost in milk exposed to sunlight for 2 hr in clear glass bottles; when kept in narrow-necked dark green bottles the loss was only 10% and in dark brown bottles 4%.

In addition to vitamin losses milk can suffer loss of available lysine on heating and on storage (Mauron, 1961). This does not happen to any significant extent in modern methods of drying and storing milk for periods up to about 1 year. It is only when the milk has been sufficiently severely heated to darken in colour (as is deliberately done for the manufacture of milk chocolate) that there is a noticeable fall in nutritive value. Bender (in press) reports, however, that storage, even under excellent conditions, can effect a loss. Dried skim milk had the same NPU as that of fresh milk, namely 74. When stored for 3 years in sealed cans under nitrogen in deep-freeze the NPU, in the

particular sample examined, had fallen to 53. Supplementation with lysine was ineffective and supplementation with methionine restored the value to 73.

Gregory, Henry & Kon (1964) examined the vitamin losses in milk evaporated after processing by various methods, including the addition of nisin which permits the use of less severe heat treatment. Milk held at 113°C for 15 min showed 83% loss of vitamin B12, 38% of B6 and 20% of B1. Milk processed at 105°C for 15 min with added nisin lost 67%, 30% and 19% respectively—only the B12 was better preserved. Milk held at 113°C for 3 min with added nisin showed better preservation of the vitamins, the losses for B12, B6 and B1 being 67%, 23% and 14% respectively. Storage for 12 months at room temperature gave further losses of B1 but not when stored at 4°C. B6, but not B12, was lost to a significant extent on storage at 4°C. There were no losses of biotin, nicotinic acid, pantothenic acid or riboflavine during manufacturing or storage. There was a slight fall in the biological value and digestibility of the protein caused by processing.

The ultra-high-temperature sterilization of milk, employing temperatures of 130–150°C for periods of 2–15 sec, produced inconsistent effects on the vitamins (Gregory & Burton, 1965; Lhussier, Hugot & Biette, 1962). In commercial plants there was no relation between severity of heat treatment and vitamin destruction, and there were inconsistencies between similar plants. All UHT processes had a negligible effect on thiamine, but the losses of B6 and B12 were higher than would occur during pasteurization (Kon, 1960). They were less, however, than would occur by an in-bottle sterilizing process.

In indirectly-heated plants the losses of B6 ranged from zero to 12% but were as high as 35% in directly-heated plants. Losses of B12 were mostly 20–30% in both types of plant. In general, direct-heating plants operate at higher temperatures for a shorter time than do indirect heaters but the authors do not find this a satisfactory explanation for the results obtained. They suggest that the direct mixing with steam causes a greater loss of B6.

It is not clear whether drying of milk has an effect on the essential fatty acids. Pol & Groot (1960) found a fall of 30–40% in spray-dried compared with lyophilized milk but Moore & Williams (1965) found no loss in roller-dried milk.

Fish

In general losses in fish are similar to those in meat. There may be greater losses of vitamin B1 because of the presence of an enzyme, thiaminase, which destroys it.

Smoking fish has little effect on the B vitamins. Salting or smoking of cod has no effect on the availability of the amino acids and has been suggested as a method of preserving fish in the developing countries where other methods are not practicable (Munro & Morrison, 1965).

Gurevic (1966) reported that 50–94% of the iodide of fish may be lost in freezing and

that defrosting and refreezing causes almost complete loss. Salting and hot smoking caused losses of 30% and losses were much less after light salting and cold smoking.

Equipment and methods

The effects of different methods of processing have already been discussed under the headings of the nutrients and commodities, but some of the information available on microwave cooking, aseptic canning and automatic equipment may be usefully discussed separately.

Microwave cooking

With the introduction of new methods of cooking and their potential use on a large scale their effect on nutritional losses is a matter of considerable interest. The effect of electronic cooking by microwave heating has been compared with conventional oven methods by many workers. As vitamin B1 is sensitive to heat it was thought possible that the rapid rise in temperature produced by the almost instantaneous penetration of heat in microwave cooking might be less destructive than the slower rise in temperature by conduction in ovens. In the event the differences were not found to be very marked.

Noble & Gomez (1962) found no significant difference between the two methods in their effect on vitamins B1 and B2 in roast lamb. The retention of B1 was 57% and 54% by microwave and oven cooking respectively, and 75% and 84% for B2, the last difference not being statistically significant. Thomas *et al.* (1949) had found no difference between losses of vitamin B2 and nicotinic acid in meat patties but a slightly better retention of vitamin B1 by microwave cooking. The findings of these authors on roast beef were that conventional methods were slightly superior.

Proctor & Goldblith (1948) showed 100% retention of vitamins B1 and B2 on electronic cooking, compared with 96% and 88% on frying. Fish suffered greater damage on electronic cooking, the retention of the two vitamins being 54% and 87% respectively, compared with 67% and 92% on baking the fish. Baked products showed 80–100% retention of vitamin B1 compared with 50–90% when gas-heated.

Kylen *et al.* (1964), Table 6, found that despite the shorter cooking times needed in the microwave process the weight losses by evaporation and drippings were greater than when cooked conventionally in three of the four types of meat examined. There was no difference in vitamin B1 retention by the two methods of cooking for roast beef, beef loaves and ham loaves; only for pork was there a significantly greater retention of vitamin B1 by microwave cooking—91% compared with 80% retention. Palatability tests revealed that the electronic cooking gave a slightly lower score for colour, texture and flavour.

Apgar *et al.* (1959) showed that although the cooking time was reduced to one-fifth by microwave heating there was no difference in cooking losses in roast meat or patties

TABLE 6. Per cent retention of vitamin B1 in meat cooked by microwave heating (M) and conventional methods (C)

Beef roast	Internal temp. (°F)	Cooking losses evaporation and drippings	Vitamin B1 retention meat and drippings
Beef roast			
C	148	18–20	81–86
M	160	29–39*	70–80
Pork			
C	185	34	80
M	185	37	91*
Beef loaves			
C	185	24	76
M185	185	27*	80
Ham loaves			
C	185	18	91
M	185	28*	87

* Significantly higher than conventional cooking.

From Kylen *et al.* (1964).

TABLE 7. Retention of vitamin B1 in meat cooked at three different temperatures in the conventional oven compared with electronic cooking

Beef round		Retention (%)
Oven cooked		
Temperature 93°C	Inner part	88
	Outer part	77
Temperature 149°C	Inner part	88
	Outer part	102*
Temperature 204°C	Inner part	67
	Outer part	60
Electronic	Inner part	86
	Outer part	67

* Result possibly due to flow of tissue fluids from the centre to the dried outer parts of the meat with local destruction and concentration of water-soluble substances including vitamin B1.

From Lushbough *et al.* (1962).

TABLE 8. Per cent retention of vitamin C in vegetables cooked by conventional methods (C) and microwave cooking (M)

	Cooking method	Time (min)	Vitamin C retention		
			Vegetable	Water	
Broccoli	C	11·7	83	10	
	M	8	79	11	
Cabbage	C	14	69	14	
	M	12	72	11*	
Cauliflower	C	8·5	92	7	
	M	8·5	87	6	
Peas	C	13·9	73	23	
	M	10	74	15*	
Green beans	C	15·8	74	9	
	M	12	78	7*	
Soya beans	C	21·9	79	11	
	M	17·5	76	10	
Spinach	C	7	61	5	
	M	7	56	9	
Frozen broccoli	C	7·4	48	12	Before cooking, 4 months
	M	6	52	9	storage–70% retention, 8 months–60%
Frozen green beans	C	15·8	74	9	Before cooking, 4 months
	M	12	78	7*	storage—62% retention, 8 months—53%
Frozen spinach	C	7	22	10	Before cooking, 4 months
	M	10	26	5*	storage—40% retention, 8 months—36%

* Significantly less than conventional cooking.

From Kylen *et al.* (1961).

but there was a smaller loss in chops electronically cooked. Headley & Jacobson (1960) found slightly greater losses by microwave cooking. This experiment was carried out on eight replicated pairs of legs of lamb and conventional methods required 52 min cooking time per lb and lost 35% by evaporation and drippings, while microwave cooking took only 13 min per lb but lost 43%. Conventional methods scored better on palatability as measured by flavour and juiciness.

Lushbough *et al.* (1962) compared beef round cooked in the oven at different temperatures with microwave cooking. The results in Table 7 show no difference between losses for vitamin B1 at oven temperatures of 93°C and 149°C and microwave cooking although the losses in the very hot oven at 204°C are greater.

The general pattern is that there is little difference between the methods of cooking meat in their effect on destruction of the B vitamins. Apart from any differences in conditions such as time and temperature of cooking and size of meat in the different experiments, such discrepancies as there are may be due to evaporation of water from the surface of the meat followed by a flow of tissue fluids from the centre carrying the water-soluble vitamins with them.

In the case of vitamin C retention in vegetables, Table 8 (Kylen *et al.*, 1961) shows that only in five results out of twenty was there a significant difference between the two methods of cooking—there was a slightly greater amount of vitamin C in the

TABLE 9. Per cent retention of vitamins cooked by microwave heating (M), boiling (B) and pressure cooking (P)

Vegetable	Method	Vitamin C		Vitamin B2		Vitamin B1	
		Solid	Liquid	Solid	Liquid	Solid	Liquid
Broccoli	M	64	23	71	31	76	31
	B	60	25	69	33	75	33
	P	72	6	94	8	90	8
Cabbage	M	59	31	69	19	62	42
	B	42	37	61	35	53	52
	P	71	10	95	2	88	3
Carrots	M	83	15	93	11	91	14
	B	80	10	90	12	88	12
	P	77	13	93	14	85	15
Potatoes	M	31	13			91	10
	B	76	18			83	14
	P	86	5			92	3

Carotene—no loss.
From Thomas *et al.* (1949).

water from conventionally cooked vegetables. There was no difference in the C contents of the vegetables themselves.

Thomas *et al.* (1949) had shown earlier (Table 9) that the losses of vitamins are largely determined by the volume of water and the cooking time and for this reason pressure cooking was superior to either boiling or microwave cooking. They observed no loss of carotene under any of the conditions listed in the table and even an apparent increase of 20–30%, presumably due to analytical error or an increase in the amount released during cooking.

Irradiation

There are many reports of the damage to vitamins caused by ionizing radiation (Groninger, Tappel & Knapp, 1956; Wilson, 1959). Some protection is offered by freezing, possibly due to the slower rate of diffusion of the free radicals.

Karmas, Thompson & Peryam (1962) showed that if pork, a rich source of the radiation-sensitive thiamine, is freeze-dried before irradiation, there is no destruction of the vitamin, but 80% can be lost by treatment with 2 Mrad at room temperature, 24% at −10°C and none at −75°C or at room temperature after freeze drying.

Kennedy (1965) found no loss in the nutritive value of the proteins of animal feeds (dosed with 0·5 and 1·0 Mrad), or with frozen egg at 0·5 and 5·0 Mrad or with wheat at 0·2 Mrad. Doses of 1 Mrad to wheat and wheat gluten produced a small fall in nutritive value.

Mameesh *et al.* (1966) found that 40% of the thiamine of fresh fish or smoked cod was lost by irradiation with 0·3 Mrad and all of it was lost with 3 Mrad. With the larger dose 30% of the riboflavine of fresh cod was lost, and after irradiation and storage the figure was 60%. Nicotinic acid was stable.

Irradiation of fats is known to initiate autoxidation and to eliminate almost completely the induction period (Bradshaw & Truby, 1962; Tonnelat & Flanzy, 1961). On subsequent storage in vacuum there is some recovery of stability. This is due to the formation of free radicals. The system in foodstuffs appears to be more complex—although there is a considerable decrease in stability on irradiation fatty foods show a remarkable recovery on post-irradiation storage. Chiphault & Mizuno (1966) showed that 2 Mrad followed by 20 weeks storage at room temperature resulted in fat stability in foods equivalent to the original unirradiated material. There was a difference between pork and beef fat that the authors were unable to explain in that added antioxidants protected beef fat but not pork from oxidation. Both meats showed a considerable improvement with added antioxidants on storage after irradiation.

Carotene and cryptoxanthin (both vitamin A precursors) were destroyed in sweet corn when irradiated and canned. Irradiation with 0·25 Mrad had no effect on beta-carotene, 0·75 Mrad destroyed 40% and 1·5 Mrad destroyed 54% (Tichenor, Martin & Wells, 1965).

Aseptic canning

Aseptic canning has been compared with conventional canning for its effects on the retention of vitamin B1 (Everson *et al.*, 1964). Tomato juice concentrate retained 100% by both methods of treatment, but for strained beef and strained Lima (butter) beans aseptic canning was superior.

Subsequent losses in storage were not affected by the method of processing. The overall retention after processing and 9 months storage was 45% in beans, 55% in meat and 80% in tomatoes.

TABLE 10. Aseptic *versus* conventional canning: percentage retention of vitamins after processing and 6 months storage

	Tomato juice concentrate	Strained butter beans	Strained beef
Vitamin B1			
Aseptic canning	100	86	93
Conventional canning	100	58	80
Vitamin B6			
Aseptic canning	100	80	80
Conventional canning	100	80	80

From Everson *et al.* (1964).

The retention of vitamin B6 was the same by both methods of treatment (Everson *et al.*, 1964), 90% of the vitamin B6 being retained in beans after processing and 70% after 9 months subsequent storage; in the beef the figures were 95% and 70% and in the tomato juice 100% even after storage.

Automatic equipment

The effect on nutrient losses of cooking in automatically operated large-scale cooking equipment is under investigation (Kunkel, 1966; Werts *et al.*, 1966). For example, potatoes steamed for 28 min and analysed $\frac{1}{2}$–1 hr later lost 27–83% of their vitamin C. White cabbage lost 80% of the C in 20 min steaming and red cabbage 90% in 40 min. Savoys steamed for 14–20 min lost 30%. Losses of calcium and phosphate were not significant but there was some loss of potassium both in steaming in large vessels and in the automatic cooker.

The damaging effect of mechanical peeling on the vitamin C content of potatoes soaked in water after peeling has already been discussed (Platt *et al.*, 1963).

Conclusion

As suggested in the Introduction, it is important to view processing losses in their proper perspective. When the population is well-nourished there is obviously no cause for concern that some foods may be of lower nutritive value than they could be. The difficulty is in defining what we mean by well-nourished and in determining whether or not there are groups of individuals living on sub-optimal amounts of nutrients. There is no direct evidence that an intake of nutrients above the minimum daily requirement confers benefit on the consumer, but the general approach is to make sure by recommending intakes considerably higher than the minimum requirement. Conversely any diminution in nutrient intake may be looked on as a retrograde step.

Moreover, there will always be groups of individuals living on diets restricted in variety so that they tend to lean heavily on a few foods to supply the full complement of nutrients. Again, changes in eating habits may alter the emphasis on the major sources of the nutrients, for example greater reliance on canned foods, or, in future years, on dried or frozen foods or meals eaten away from home. In general, as pointed out elsewhere by the author (Bender, 1965) improved methods of processing intended to retain flavour, colour and texture tend to involve less severe treatment and incidentally, if not designedly, inflict less damage on the nutrients.

An example of the need for caution is contained in the Report on Hospital Feeding (Platt *et al.*, 1963). As shown in Table 1 potatoes serve as the major source of vitamin C in the British diet, yet these authors were able to state categorically that 'Potatoes as served in most hospitals do not make a substantial contribution to the provision of ascorbic acid'—because of processing losses.

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The measurement of cake crumb strength

A. H. ROBSON

Summary. A short survey is given of the various types of instruments which have been and are at present being used to evaluate cake crumb strength.

The Panimeter is an instrument in current use, and it has been designed to measure crumb strength by compressing a sample under conditions of constant stress. Certain modifications in design have been made in order to improve the general performance of the instrument, including complete automation of the test and the provision for making constant strain tests. Elasticity and firmness tests have also been made on margarine and butter with the Panimeter.

A new instrument, the Rotary Cutter, has been built with which attempts have been made to assess crumb strength from the forces developed when a rotating blade cuts a spiral path in a cake sample. A continuous record of crumb strength is obtained by transmitting the forces via a potentiometer and Wheatstone bridge circuit to a pen recorder.

A comparison is made of the results produced by the instruments from tests carried out on sandwich cakes prepared from fats of varying hardness.

Introduction

In the assessment of cake crumb strength the instruments in common use include compressimeters, penetrometers and the more sophisticated type of instrument which simulates the human chewing action. Included in the first category is a compressimeter developed by the British Baking Industries Research Association (1963). In this instrument the sample is compressed between two brass plates, the lower plate being anchored to the baseplate while the upper plate has a platform to which weights can be added. The upper plate and platform assembly are counterbalanced by a suitable weight which hangs over a pulley. To carry out a test under conditions of constant stress, a weight is added to the platform, and a pointer attached to the pulley registers the resultant compression. A test can also be made under constant strain conditions, but the duration of the operation is unduly long, as weights have to be added progressively to the platform at 1 min intervals until the desired compression is reached.

More recently Babb (1965) has developed a compression type of instrument which

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allows the rapid examination of the firmness and compressibility of cakes to be recorded.

In the penetrometer class of instrument, an indenter is described which uses a 90° counterbalanced cone (Cornford, 1963). With the tip of the cone resting on the surface of the sample, a 200 g weight is added and the depth of penetration noted after 1 min. It was claimed that this instrument was more reliable in performance and faster in operation than the compressimeter.

The measurement of tensile stength of cake has been undertaken in various ways. One method consists of clamping a thin sample at one end and applying a gradually increasing force at the other end until breakage occurs (Cornford & Coppock, 1950). Another method is carried out by clamping a sample at one end and rotating the other end until the sample breaks (Griswold, 1962). It is claimed that the angle of break is related to tensile strength.

A test has been reported which attempted to relate cake structure to crumb stickiness (Cornford & Coppock, 1950). In this test a flat brass plate was pressed against the surface of a cake, and after 2 min the force required to remove it was taken as an evaluation of cake structure. This method proved to be unreliable however, as it was too sensitive to minor changes in baking.

A sophisticated instrument known as a Tenderometer has been used to evaluate the textures of a wide variety of foodstuffs (Proctor *et al.*, 1955). This instrument operates in the same way as the human jaws and bites food between a set of teeth in the upper jaw and a plate in the lower jaw. Vertical, side and forward movements are given to the jaws by having them mounted in the mechanical adaptation of a dental articulator. The resistance offered by the foodstuffs is transmitted by strain gauges mounted in three planes to an oscillograph, where the display is photographed by a Polaroid Land camera.

Materials and methods

Original design of the Panimeter

In our investigations into the evaluation of crumb strength, we have carried out a series of tests with a compression instrument which is similar in principle to the B.B.I.R.A. compressimeter. This instrument, the Panimeter, was designed by Hintzer (1949, 1951) for the Instituut voor Graan, Meel en Brod, T.N.O., The Netherlands, and we used it in the form shown in Fig. 1. A cylindrical sample of cake (a) was placed on a pan (1) which hangs from one end of a balance beam (2). A compressive force was applied to the sample by weight (3) which was driven along the beam by its built-in motor. The sample was compressed between the pan and the upper plate (4), the latter being anchored by columns to the base plate. Attached to the right-hand beam was a cord (5) which wound around a pulley (6). An arm was attached to the pulley shaft and carried at its extremity the pen (7) which gave a continuous record of the state of sample compression. The beam was raised into its operating position by

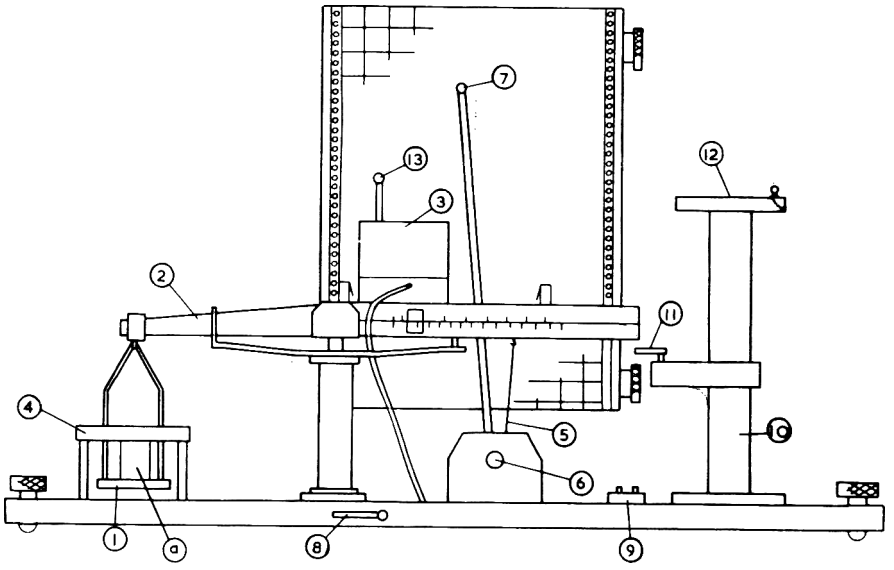

FIG. 1.

the handle (8) which acted in a similar manner to the elevating mechanism of a chemical balance. A cylindrical cake sample, 2·8 cm in length and either 3, 4 or 5 cm in diameter, was placed on the pan, and with the weight at its starting position near the fulcrum, the beam was raised. The weight in this position applied a force of 50 g to the sample, which gave a positive start to the sample compression, and assisted in adjusting the pen to the zero line of the chart. The motor was then switched on at (9) and was driven along the beam by a rack and pinion mechanism to a predetermined position on the beam. The weight was held at this position for 2 min during which time the sample continued to compress. After 2 min, when compression was virtually complete, the motor was reversed and the weight returned to the fulcrum. The actual recovery value was derived from the reading after a further 2 min.

The percentage compression and recovery values of the sample are derived from the readings on the chart after each 2 min pause, as mentioned above. The values are calculated as follows:

$$\% \text{ Compression} = \frac{\text{Maximum Panimeter scale unit } (P)}{1000} \times \frac{16{\cdot}68}{28} \times 100 = 0{\cdot}0594P$$

$$\% \text{ Recovery} = \frac{\text{Maximum Panimeter scale unit} - \text{Minimum Panimeter scale unit}}{\text{Maximum Panimeter scale unit}} \times 100$$

Modifications to the Panimeter

1. The arms of the yoke which support the pan (1) pass through wide slots in the upper plate (4), and we found that due to the lack of guides at these points, the sample, in addition to being compressed also tended to shear horizontally. The shear was eliminated by inserting nylon guides in the upper plate.

2. The operation of the Panimeter test has been made virtually automatic by re-designing the electrical circuit. This modification eliminates human error, particularly in timing the holding periods, and permits the operator meanwhile to prepare the next sample. A push button starts the motor and sends the weight along the beam until a switch is tripped which holds the weight in the desired position on the beam. A cam timer holds the weight here for 2 min, then through a two-relay circuit the motor is reversed and returns the weight to its starting position. The weight is prevented from over-running the fulcrum by a micro-switch, and the recovery curve is traced until the timer switches off the chart drive after 2 min. The major components of the electrical circuit include a Carpenter relay, two mains voltage relays and a cam timer.

Constant strain test with the Panimeter

It has been shown that when the cake sample fractures in a compression test, the compression and recovery values are meaningless. In order therefore to compare a range of cakes under conditions of constant stress, it is essential that the compression range be carefully chosen so that fracture of any one of the samples should be avoided. As this condition is difficult to meet in view of the non-linear behaviour of cake structure, it was felt that results would probably be more reliable under conditions of constant strain.

The Compressimeter is used to compress bread and cake under conditions of constant strain, but the method is time-consuming as weights have to be added by the operator at 1 min intervals until the desired compression is reached. Further disadvantages are that the instrument is non-recording and it is probable that inaccuracies occur when the weights are added by hand. As a replacement for the Compressimeter we have modified the mechanism of the Panimeter as follows, so that it will carry out tests under constant strain conditions.

A brass pillar (10) was built on to the base of the instrument near one end of the beam (see Fig. 1). A light-action leaf switch (11) was connected with a lead screw inside the pillar so that it could be raised or lowered by means of a handwheel (12). A compression test was made by placing the sample on the pan, raising the beam and adjusting the height of the leaf switch so that its contacts were just closed by the end of the beam. The leaf switch was then lowered by a distance which was equivalent to the desired compression of the sample. The weight was then driven along the beam until

the sample reached its predetermined compression when the leaf switch closed (maximum force). When this occurred, the direction of the motor was reversed by a series of relay switches and the weight continued in reverse until the leaf switch re-opened. As a result of the tendency of the sample to undergo further compression, the weight moved backwards in a series of short steps. A pen (13) recorded the movement of the motorized weight, and the value of the stress required to give a predetermined compression was taken from the height of the recording after precisely 5 min (minimum force). By this means a fully automatic procedure for making determinations under conditions of constant strain was achieved.

The application of the Panimeter to margarine and butter testing

The possibility of using the Panimeter as a test for margarine and butter was investigated. With these comparatively rigid materials the percentage compression which can be tolerated without fracture is very low, and in fact the constant stress test proved to be impracticable as the small differences in compression could not be resolved from the recordings.

The constant strain test was, however, more successfully applied and four margarines and one butter were tested at 10, 15 and 20°C at compressions of $2\frac{1}{2}$, 5, 10 and 20%. The soft, most easily spreadable, margarines, could be compressed by 20% at 15°C, and by about 15% at 10°C without fracturing, but in contrast to this high level of plasticity the harder, more brittle, types fractured when compressed to between 5 and 10% even at 20°C. The level of applied force which was initially required to obtain the predetermined compression was found to be proportional to the hardness of the sample; this reading was obtained from the maximum or peak reading of the trace. The weight then reversed to an intermediate position, but if fracture or flow due to extreme softness had occurred then the weight returned to its zero position.

The butter, although as tough as the hardest margarine, fractured in one case only, namely at 20% compression and 10°C. The fact that the butter sample did not readily fracture and also that the final applied stress value was usually relatively high compared with the value given by the margarines, demonstrated the springiness or elasticity which is a well-known characteristic of butter.

Rotary cutter

While the compression type of tests are satisfactory for estimating the crumb strength and tenderness of cakes, we required another test to supplement the results of the Panimeter and which would simulate the destructive mechanism of chewing. Without attempting to imitate human jaw movements, a simple test was evolved which consisted of cutting a spiral path through the cake sample with a blunt two-bladed cutter, and measuring the resultant forces. The prototype instrument is shown

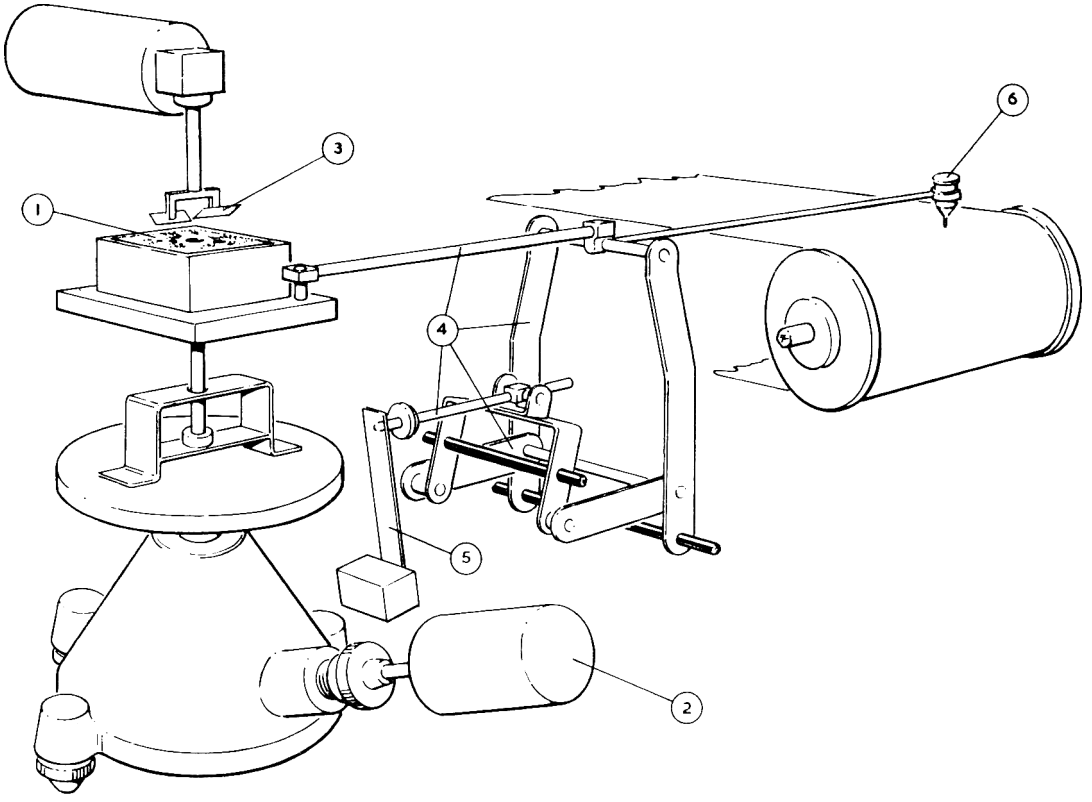

FIG. 2.

in Fig. 2. A rectangular sample (1) and its supporting framework were raised at constant speed by the motor (2). The torque imparted to the sample by rotating the blades (3) counter-clockwise was transmitted to the linkages (4) and was opposed by the leaf spring (5). Thus the resistance offered by the sample to the cutting blades was measured by the deflection of the leafspring and recorded by the pen (6).

From experience gained in the operation of this instrument it became apparent that several modifications were necessary. For example, the centre core of the sample had to be removed with a cork borer otherwise a small compressed dome of cake was formed below the axis of the cutter. This small piece of compressed cake gave an erroneous value to the average height of the trace. Also the crumbs which collected in the cut-away well of the cake produced an artificial drag on the mechanism and this affected the level of the recording. No simple way could be found of removing these residues during the course of the test. Also the relatively large weight of the cake, its holder and the lever mechanism seriously damped the pen oscillations and reduced the

sensitivity of the instrument by a considerable amount. A further factor leading to this insensitivity was the mechanical linkage which produced a high level of frictional drag. The inertia of the cake support, drag of the crumbs and mechanical friction resulted in failure of this instrument to distinguish small differences in texture in, for example, sponge cakes, which have a soft crumb structure.

In order to overcome the above-mentioned disadvantages, a second model was built (Fig. 3). The accumulation of crumbs was avoided by interchanging the positions of the sample holder (1) and blade (2). The cutter blade shaft had a hardened point (3) at its lower end which rested on a small platform (4). This platform was connected to a rack and pinion device (5), which raised the platform and blade at constant speed.

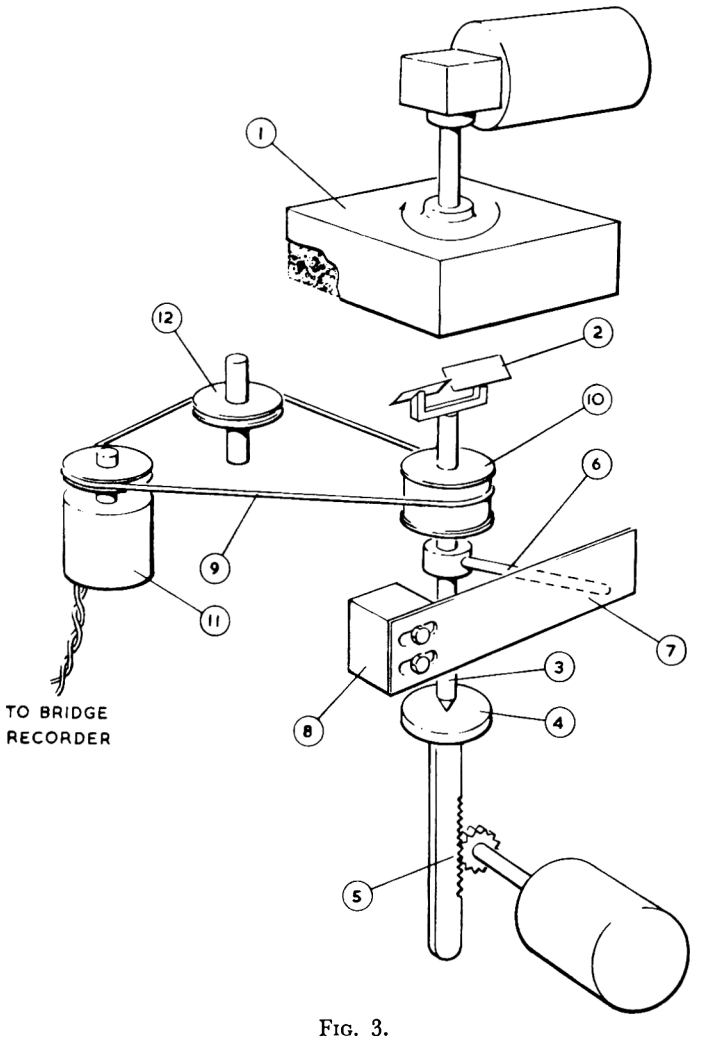

FIG. 3.

A rod with a rounded tip (6) was attached to the shaft of the blade and the rounded end made contact with the surface of a leaf spring (7). The leaf spring anchorage (8) allowed a certain adjustment of the effective length of the spring to be made (i.e. shortened for hard cakes and lengthened for softer ones). The movement of the cutter blade, as governed by the texture of the sample and the rigidity of the spring, was transmitted via a cord loop (9), which wound round a drum on the cutter shaft, then round a pulley which was connected to the shaft of a potentiometer (11) with friction-less bearings. Slack was taken by an idler pulley (12). The potentiometer was connected to a Wheatstone bridge circuit, and the out-of-balance signal was fed into a pen recorder.

It was found that as this modified instrument was low in frictional drag, even the most tender cakes could be tested. Also by having the sample above the cutter blade no interference was experienced from crumbs which fell away into a collector tray. The adjustable spring anchorage had the advantage of allowing a wide variety of cakes to be tested without having to interchange springs.

Cone penetrometer

An improved method for the determination of crumb strength has been claimed by Cornford (1963) who found that the cone indenter gave more meaningful results than the compression type of instrument. One advantage of the indenter or penetrometer is that a large number of determinations can be made on one cake, and it is conceivable that homogeneity of cakes could be determined in this way.

We have made texture determinations with the Hutchinson Cone Penetrometer (40° cone), but difficulty was experienced in determining the level of coarse-crumb cakes. The determination of the surface by means of a thin sheet of aluminium foil was found to be awkward in practice, and we are at present investigating other methods which could automatically locate the tip of the cone at the surface.

Results and discussion

The improvement in performance which was gained by using yoke guides for the sample pan of the Panimeter is shown in Fig. 4 and Table 1. Four samples were taken

TABLE 1. Panimeter, constant stress: applied load 350 g

	Sample	% Compression	% Recovery
With guides	1	39·8	26·9
	2	39·5	26·3
Without guides	3	44·6	28·0
	4	45·1	37·0

from the same cake, and two tests were made with and two without the guides in position. Better duplication of results was obtained when the guides were used, and it can be seen that with the guides absent the shearing component has produced a false, high figure for compression value.

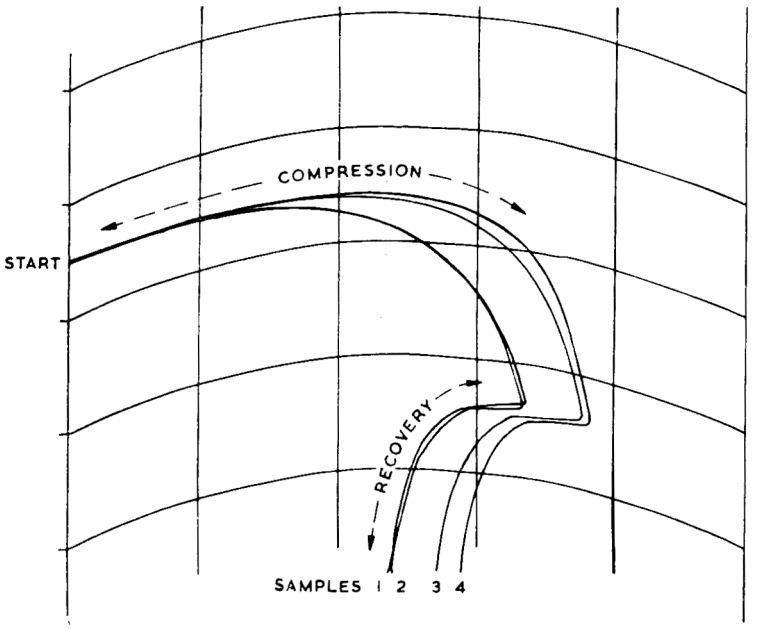

FIG. 4.

Six groups of sandwich cakes, each group being made from fats which were known to give cakes of different crumb strength, were tested with the Panimeter and Rotary Cutter 1 day after baking. The cakes were prepared in a pilot bakery, and having cooled down after baking, were then wrapped and stored overnight at 25°C. The wrappings were removed immediately before testing, the Panimeter samples being taken at a distance of 1 in. from the centre of each cake, with crust and base excluded from the sample. The constant strain determinations were made on 3 cm diameter samples. In the constant stress tests, forces of 400 and 750 g were applied to samples of 3 and 4 cm diameter respectively. Rotary Cutter samples were taken from the cake centres. The results are shown in Table 2.

It can be seen from the 3 cm percentage compression column of constant stress that the samples exhibit the correct order of crumb strength with the exception of sample D. In the 4 cm column, where the compression is greater, the order does not correlate satisfactorily, suggesting that the crumb structure was severely ruptured.

TABLE 2

Sample	Known crumb strength	Panimeter						Rotary Cutter (A.U.G.)
		Constant stress				Constant strain		
		3 cm/400 g		4 cm/750 g		Required force (g) for 10% compression		
		% Compression	% Recovery	% Compression	% Recovery	Maximum	Minimum	
A	Weakest	38	14	43	23	350	250	0·308
B		37	21	51	20	385	290	0·358
C		26	32	47	28	490	340	0·321
D		13	62	32	45	405	280	0·369
E		18	32	33	28	620	390	0·357
F	Toughest	17	38	33	43	670	450	0·348

In the constant strain test both sets of readings were in the order found in the 3 cm constant stress column, again with the exception of sample D.

The Rotary Cutter coefficient was derived by cutting out and weighing the recorder paper between the graph and base line. This coefficient is expressed as 'Area under graph' (A.U.G.). The recorder trace always took the form of an oscillating trace and attempts have been made to discover any relation between texture or other cake property and the difference between maximum and minimum points on the trace. So far no significance has been found in these figures when applied to sandwich cake.

A series of determinations were also made with the Rotary Cutter on cakes having greater crumb strength. A group of madeira cakes were prepared with fats which gave cakes of different crumb strength, and in addition to the Rotary Cutter tests, constant stress Panimeter tests were made as a basis for comparison. The baked products were individually wrapped, stored overnight at 25°C and tested 24 hr after being withdrawn from the oven. The results are shown in Table 3.

TABLE 3.

Sample		Panimeter: constant stress			Rotary Cutter (A.U.G.)
		% Compression			
		Applied force			
		500 g	400 g	300 g	
A	Weak crumb	25	21	20	0·34
B		23	15	7	0·38
C		22	18	8	0·42
D		23	16	10	0·51
E		18	8·5	6·5	0·51
F	Strong crumb	16	9	3·5	0·53

Diameter of Panimeter sample: 3 cm.

The results of this test show that the correct order of crumb strength was selected by the Rotary Cutter compared with the generally indecisive results given by the Panimeter constant stress test. Further tests have confirmed that the Rotary Cutter can be successfully applied to cakes possessing strong crumb strength.

Acknowledgments

The author wishes to thank Unilever Limited for permission to present this paper, and also Mr P. Alexander who organized the preparation of cake samples.

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J. Fd Technol. (1966) **1**, 303–311.

Thermal conductivities of muscles, fats and bones

M. J. MORLEY

Summary. The thermal conductivities of the components of meat were determined by two rapid methods. Values for muscles and fats at temperatures between −19°C and +37°C were obtained using the probe method, and for bones at 0°C, 19°C and 70°C using the thermal comparator method.

Introduction

The heating, cooling and freezing rates of meat are governed by the thermal conductivities of its components. Hitherto, measurements of the thermal conductivities of such materials have been made using slow, steady-state methods, with the exception of Walters & May (1963) who used a non steady-state method. The latter method was rapid, each measurement taking only a few minutes. The rapid methods used in this study have the added advantage that measurements can be made *in situ*.

Measurements were carried out using two different methods; one, the probe method, being applicable to muscle and fat, while the other, the thermal-comparator method, was suitable for bone. The probe method was initially developed by Van der Held & Van Drunen (1949), later developments being made as the method was more widely applied. The thermal comparator method was introduced by Powell (1957). It was later modified into a direct reading form and applied to a wide variety of solids and fluids (Clark & Powell, 1962; Powell & Tye, 1964).

Apparatus and procedure

(a) *Probe method*

The apparatus is illustrated in Fig. 1(a). The probe (Fig. 1(b)) consisted of a 4 in. long 17 swg steel tube, which enclosed a 40 swg constantan heater and a 40 swg copper–constantan thermocouple, the free space on the probe being filled with an epoxy resin ('Araldite'). The heater, being constantan, had negligible variation of resistance over the relevant range of temperature. The probe was inserted into the specimen which was then placed in a constant temperature enclosure. One junction of the thermocouple was situated at the mid-point of the probe length and the other junction in the constant temperature enclosure, suitably shielded from temperature

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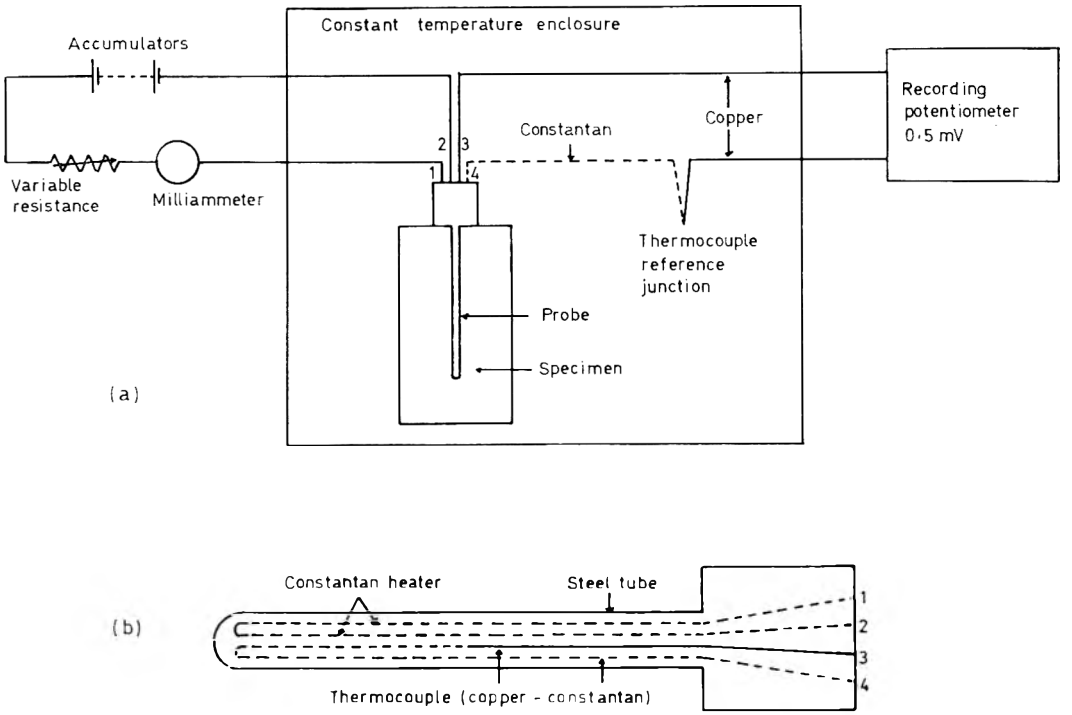

FIG. 1. Probe method. (a) Diagram of apparatus. (b) Details of the probe.

fluctuations. When the specimen had reached equilibrium with enclosure temperature, a constant current was passed into the probe heater and heat flowed radially from the probe into the surrounding specimen. The increase in temperature (θ) at the mid-point of the probe length, with time (t), was recorded by a recording potentiometer for a period of about 5 min. A graph was then plotted of temperature increase (θ) against $\log_e t$ and the gradient (G) of the linear region measured. As an illustration, the graphs for lean meat at 16°C are shown in Fig. 2.

The theory of the probe (de Vries & Peck, 1958) gives:

$$\theta = \frac{I^2R}{4\pi K}\left\{\log_e\left(\frac{1}{x}\right) - 0{\cdot}5772 + x\left[2\left(1 - \frac{aK_1}{a_1K}\right)\left(\log_e\left(\frac{1}{x}\right) - 0{\cdot}5772\right) + 2 - \frac{a}{a_1}\right] + \text{terms in higher powers of } x\right\}$$

where x is $r_0^2/4at$; r_0 the probe radius (cm); I the heater current (amps); R the resistance per unit length of the heater (ohms cm^{-1}); K, K_1 the thermal conductivities of the specimen and probe respectively (Joules sec^{-1} cm^{-1} $°C^{-1}$); a, a_1 the thermal diffusitivies of the specimen and probe respectively (cm^2 sec^{-1}).

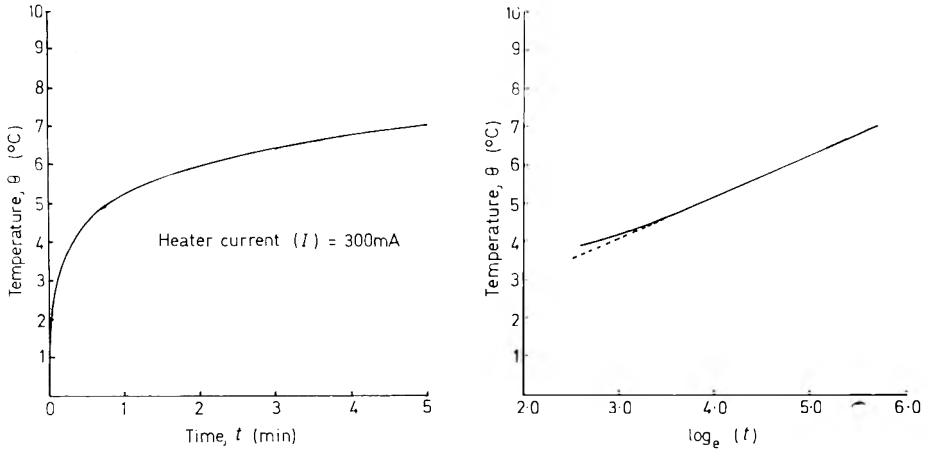

FIG. 2. Probe method graphs for lean meat at 16°C.

After a certain time, x becomes sufficiently small for the equation to reduce to:

$$\theta = \frac{I^2R}{4\pi K}\left\{\log_e\left(\frac{1}{x}\right) - 0{\cdot}5772\right\}$$

Thus the graph of θ against $\log_e t$ becomes a straight line of gradient $G = I^2R/4\pi K$ and hence the thermal conductivity (K) of the specimen is given by:

$$K = \frac{I^2R}{4\pi G}$$

For the specimens examined, the graphs became linear after an interval of between 10 and 50 sec.

In the theory it was assumed that the probe is infinitely long. Finite probe length (l) results in axial heat flow in the probe and specimen, causing a falling off in the gradient G. Blackwell (1956) has shown that the percentage decrease in the gradient is not more than:

$$\frac{100}{e^{(I^2/16at)}\sqrt{(\pi)}}\left\{\frac{4\sqrt{(at)}}{l} + \frac{lr_0{}^2K_1[1-(a/a_1)]}{8K(at)^{3/2}}\left[\log_e\left(\frac{4at}{r_0{}^2}\right) - 0{\cdot}5772\right]\right\}$$

The dimensions of the probe were such that this effect was negligible for measurements on the specimens in this experiment.

The radius of the specimen must be $\geqslant 2{\cdot}6\sqrt{(at_e)}$ (Vos, 1955) to prevent a significant quantity of heat reaching its boundary during the period of the experiment (t_e). The departure from linearity of the graph of θ against $\log_e t$ indicates when the radius of insufficient magnitude. In this experiment, a radius of between $\frac{1}{2}$ and 1 in., depending on the specimen, was found to be adequate.

(b) *Comparator method*

The apparatus is illustrated in Fig. 3(a). The comparator consisted of a cylinder of constantan (volume approximately 50 cm^3) conically formed at one end. Two nichrome wires (28 swg) were attached to the constantan block, one to the upper surface A and the other peened into a hole as close as possible to the tip B, forming a thermocouple

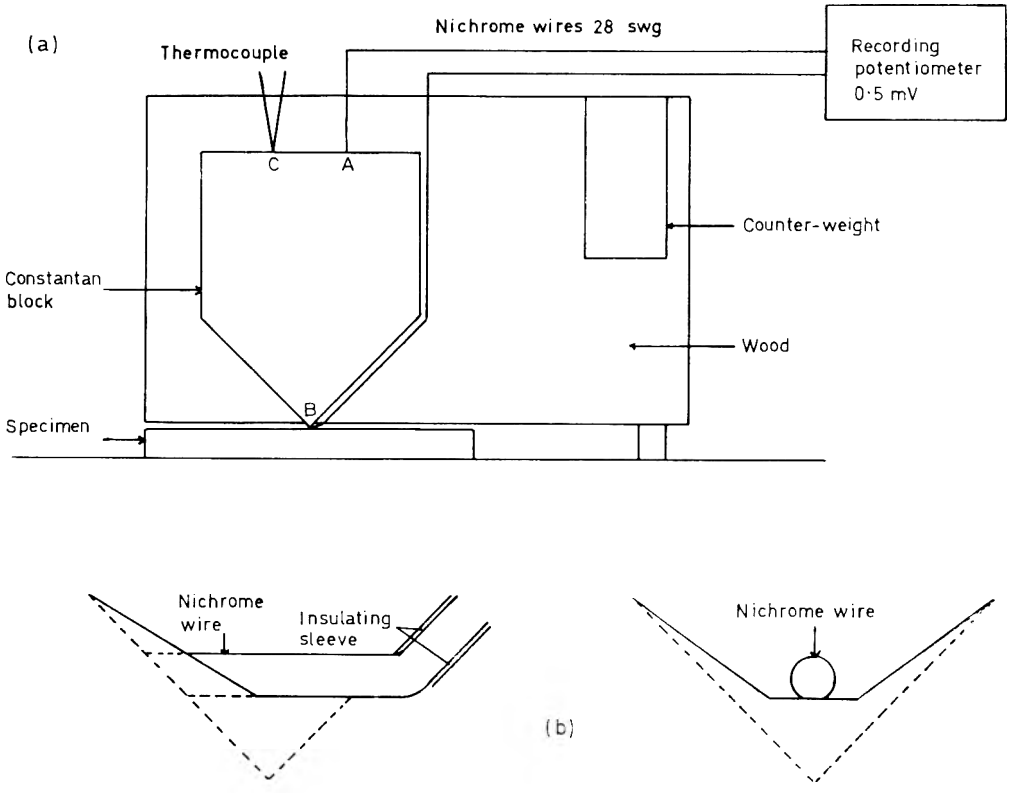

FIG. 3. Comparator method. (a) Diagram of apparatus. (b) Formation of the tip.

indicating the temperature difference between A and B. The tip was then filed down to expose part of the wire and to reduce the area of contact, as illustrated in Fig. 3(b). Another thermocouple junction was inserted at C to measure the temperature of the constantan block. The whole was mounted in wood with only the tip B exposed. The specimen whose thermal conductivity was to be measured was equilibrated in an enclosure controlled at the measurement temperature θ_2. The comparator was equilibrated in an oven at a temperature θ_1, 97°C above θ_2, and was then removed and placed on the specimen as shown in Fig. 3(a). Heat flowed from the tip of the comparator into the specimen and, after a few seconds, a constant temperature was established at the

point of contact. The difference between this contact temperature θ_c and the comparator temperature θ_1, was measured by means of a thermocouple and recording potentiometer. This temperature difference $\theta_1 - \theta_c$ depends upon the thermal conductivity of the specimen (K_2) and the temperature difference between the comparator and the specimen ($\theta_1 - \theta_2$). A calibration graph of $\theta_1 - \theta_c$ against K_2 was plotted for constant ($\theta_1 - \theta_2$), using specimens of known thermal conductivity. Glasses were used for calibration since their thermal conductivities were in the range of interest, and have been extensively studied (Ratcliffe, 1963). The calibration graph was linear over the range of conductivities considered (Fig. 4). Readings were reproducible to within $\pm 1\%$.

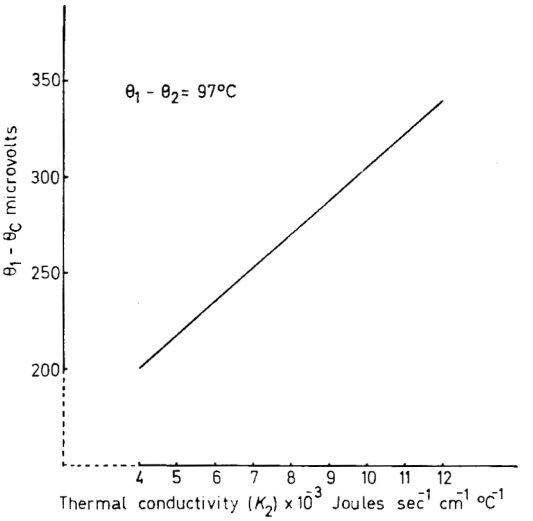

FIG. 4. Calibration graph of the comparator.

An approximate theoretical analysis of the method (Clark & Powell, 1962) gives:

$$\theta_1 - \theta_c = \frac{K_2(\theta_1 - \theta_2)}{K_1 + K_2}$$

where K_1 and K_2 are respectively the thermal conductivities of the comparator and specimen. For maximum sensitivity, $\theta_1 - \theta_2$ must be large and K_1 close to K_2. The type of metal used in the comparator will also depend on its ability to provide a sufficiently sensitive thermocouple with the connecting wires.

The rate of cooling of the comparator due to heat flow into the specimen is approximately:

$$\frac{4K_1K_2\, r(\theta_1 - \theta_2)}{(K_1 + K_2)\, \rho\, cV}$$

where r is the radius of the region of contact and ρ, c, V are respectively the density, specific heat, and volume of the comparator. Thus, to minimize the rate of cooling, r must be small and V large. The comparator also cools by conduction to its surroundings and some degree of insulation was obtained by mounting the device in wood. During the initial $1\frac{1}{2}$ min the temperature of the comparator fell by only 0·5°C, this having negligible effect on the measured temperature difference. The latter, i.e. $\theta_1 - \theta_c$, became constant after $\sim$ 10 sec of contact with the specimen.

The specimen had to be sufficiently thick to prevent a significant quantity of heat reaching its boundaries during the period of measurement. For bone the minimum permissible thickness was found to be approximately 0·1 in.; below this value the measured temperature difference was no longer constant.

Results

The thermal conductivities of ice at -19°C, and water (gelled with approximately 2% gelatin to prevent convection currents interfering with measurement) at 20°C, were determined by the probe in order to test the method. Observed conductivities were compared with reported values obtained by other methods. The figure obtained for the gelled-water ($6{\cdot}19 \times 10^{-3}$ Joules sec^{-1} cm^{-1} $°C^{-1}$) agreed, within experimental error, with the established value for water (Powell, 1958), and with the value obtained by Lentz (1961) for low concentration gelatin gels; similarly the value for ice at -19°C ($24{\cdot}7 \times 10^{-3}$ Joules sec^{-1} cm^{-1} $°C^{-1}$) agreed with that reported by Ratcliffe (1962).

The thermal conductivities of various components of meat were measured by the probe method, giving the values shown in Table 1. It has been shown previously (Lentz, 1961) that the thermal conductivity of muscle depends on the direction of heat flow relative to the fibres, being higher when parallel than when perpendicular to them. In this study, heat flow was always approximately perpendicular to the muscle fibres. At 16°C there was little difference in thermal conductivity between the different muscle samples examined. The results were consistent with reported values for lean meat (Cherneeva, 1956; Lentz, 1961) and were close to the figure for water. The similarity with water was not unexpected due to the high water content (75% approx.) of lean meat, and it was further demonstrated when the temperature was reduced to -19°C. Here the figures tended towards that for ice, but there was considerable variation between the different muscles examined. This variation was also found by other investigators. Cherneeva (1956) obtained a variation from 12·9 to $15{\cdot}7 \times 10^{-3}$ Joules sec^{-1} cm^{-1} $°C^{-1}$ for three different muscles (two beef, one pork); Lentz (1961) obtained values from 11·5 to $14{\cdot}2 \times 10^{-3}$ Joules sec^{-1} cm^{-1} $°C^{-1}$ for four different muscles (one beef, one pork, two turkey), heat flow being perpendicular to the fibres in both cases. These variations in thermal conductivity appear to be unrelated to differences in fat

TABLE 1. Results obtained by the probe method

Specimen	Temperature (°C)	Thermal conductivity $\times 10^{-3}$ Joules sec^{-1} cm^{-1} $°C^{-1}$
Gelatin gel (2% gelatin in water)	20	6·19
Ice	−19	24·7
Beef long. dorsi, lumbar	16	5·15
	−19	13·0
Beef pectoralis	16	4·98
Pork long. dorsi, lumbar	16	5·15
	−19	18·0
Lamb long. dorsi, lumbar	−19	17·7
Beef sirloin fat	16	2·09
	−19	2·68
Beef kidney fat	37	1·88
	16	1·88
	1	1·92
Rendered beef kidney fat	16	1·25
	1	1·25
Pork loin fat	16	1·95
Cancellated bone	16	2·60
(beef femur extremity)	−19	3·30

and water content (Lentz, 1961). At −20°C approximately 87% of the water in mammalian muscle is present as ice (Moran, 1933). A possible explanation for the variations is that they were due to differences in ice crystal size. When muscle is frozen, ice crystals are formed between the fibres causing the latter to be distorted and bunched together; the larger the crystals, the greater the disruption. The continuity of the ice is greater when the crystals are larger and a higher thermal conductivity might be expected. Crystal size depends mainly on the rate of freezing, a rapid rate producing small crystals. The values obtained at −19°C tended to be greater than those reported by Lentz (1961). Since the freezing time in the latter case was approximately 3 hr compared with 12 hr in this study, the difference in values was attributed to the resulting difference in ice crystal size.

The results obtained for beef and pork fats were consistent with other reported values (Lapshin, 1954; Cherneeva, 1956; Lentz, 1961). The lower figure obtained on rendering beef kidney fat was to be expected, due to the lower water content resulting from the rendering process. The thermal conductivity of fat did not vary significantly with temperature between 1°C and 37°C. The increase in conductivity when the temperature was reduced to −19°C was much less pronounced than with lean meat, this being attributed to the lower ice content of the fat.

The probe method was not generally suitable for measurements on bone due to the large specimen size required. Measurements were carried out only on cancellated bone in the extremity of beef femur, where a sufficiently large volume of this tissue was located. Its thermal conductivity was found to be fairly similar to that of fat. The probe could not be used for measurements on the outer region of bone (compact bone) since the available volume of this tissue did not meet the requirements of the method.

Measurements of the thermal conductivity of compact bone were carried out using the comparator, and the results are shown in Table 2. Each value is the mean of

TABLE 2. Results obtained by the thermal-comparator method

Bone	Temperature (°C)	Thermal conductivity $\times 10^{-3}$ Joules sec^{-1} cm^{-1} $°C^{-1}$
Pork humerus	0	5·19
Beef radius	0	5·32
Pork femur	19	5·78
Pork femur (boiled in water for 2 hr approx.)	19	5·69
Beef rib	19	5·44
Beef rib (boiled in water for 2 hr approx.)	19	5·44
Beef radius	19	5·69
Beef radius (boiled in water for 2 hr approx.)	19	5·57
Beef radius (allowed to dry at room temperature and humidity for several months)	19	4·86
Pork femur	70	4·61
Beef femur	70	4·98

several readings taken at different points on the surface of each bone, the variation of these being within $\pm 5\%$ of the mean. Measurements were made only on smooth regions of the bone surface since roughness leads to poor contact and consequently, false readings. At each temperature there was no significant difference in thermal conductivity between the different bones examined. In general their values were rather higher than those for lean meat. The slight increase in thermal conductivity of bone between 0°C and 19°C suggested a higher figure at 70°C. However, the values obtained at 70°C were lower than those at 0°C and this was attributed to the drying of the bone surface, which took place during the 12-hr period of equilibration at 70°C before measurements were commenced. The effect of surface drying was further demonstrated by the reduction in thermal conductivity of beef radius which had been allowed to dry under conditions of room temperature and humidity for several months. Heat

treatment alone had no apparent effect on the thermal conductivity of bone: since values obtained before and after boiling bones in water for approximately 2 hr did not differ significantly.

The results obtained by the two methods of measurement were estimated to be accurate to within $\pm 5\%$.

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The dielectric constant of foods and other materials with high water contents at microwave frequencies

G. P. DE LOOR AND F. W. MEIJBOOM

Summary. Measurements are given on foods and other materials with high water contents between 1·2 and 18 Gc/s. Within the measuring accuracy all results lie on semi-circular Cole–Cole arcs, thus indicating a single relaxation time. This relaxation can be ascribed to the free water available. It is shown that mixtures can be designed which are useful as model systems.

Introduction

Since dielectric heating equipment is beginning to be operated in the microwave region it is of growing importance to know more about the dielectric behaviour of food and related materials at microwave frequencies. As most of the systems to be heated are heterogeneous it is relevant to study such materials from this viewpoint (de Loor, 1965). For a survey of the dielectric properties of heterogeneous mixtures the reader is referred to former work (de Loor, 1956, 1964). Various dielectric losses play an important role in dielectric heating and Fig. 1 shows the frequency range of the

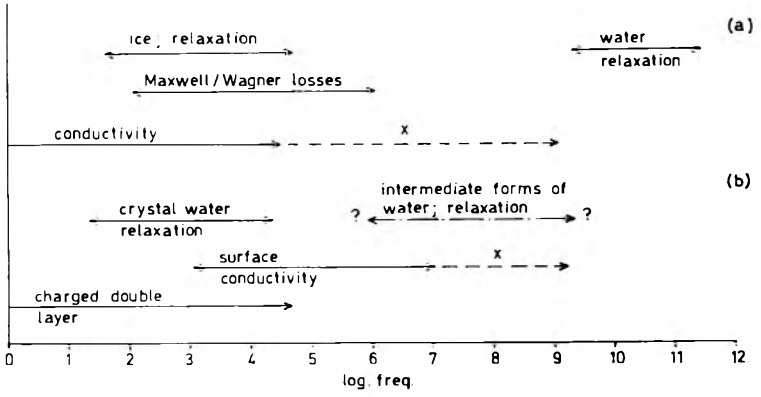

FIG. 1. Origin of the losses in heterogeneous mixtures containing water. (a) No surface effects; (b) losses due to surface effects. x = extension for water containing ions.

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various types of loss which can occur in heterogeneous mixtures containing water. While at the lower frequencies many phenomena come into play in the build-up of the total loss in such mixtures, only a few remain at microwave frequencies. They are the dipolar losses of the free water and, at the lower frequency end of the microwaves, the conductivity losses due to water containing ions.

The investigation reported was intended primarily to check the above approach. For the moment it includes only materials with a very high water content (more than 75% by volume).

Measuring methods

Since each of the materials involved in this study has a very high water content, large values of dielectric constant and dielectric loss had to be measured. This led us to the

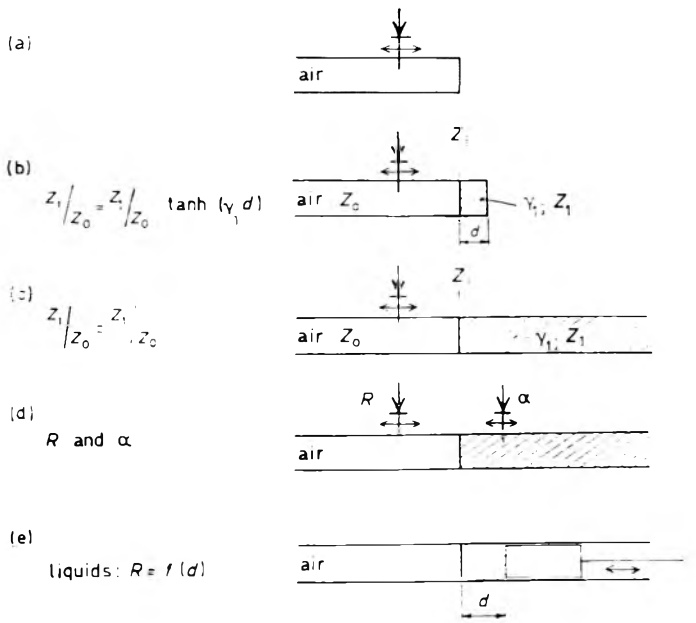

FIG. 2. Measuring techniques used.

series of measuring techniques represented in Fig. 2 of which most are slight adaptations of standard measuring techniques and only one is novel. In all cases we tried to use as many techniques as possible on the same sample and took the average value thus obtained as the final value for the dielectric constant for the frequency used.

Coaxial equipment of our own design (diameter of outer conductor 36 mm, of inner conductor 15·7 mm) was used for the frequency range 2–4 Gc/s and rectangular waveguide equipment between 4 and 18 Gc/s (four bands: waveguides WG 12, 14, 16 and 18).

The symbols used in Fig. 2 have the following meaning: $\mathcal{Z}_0$ is the characteristic impedance of the air-filled waveguide; $\mathcal{Z}_i$ the input impedance at the front face of medium 1; $\mathcal{Z}_1$ the intrinsic impedance of medium 1; γ_1 the propagation constant for medium 1 and d its thickness; α the attenuation of the EM-wave in this same medium and R the reflection coefficient.

Medium 1 is in all cases the sample to be measured.

The first measurement as indicated in Fig. 2(a) is a short-circuit measurement, to fix the plane in which the impedance is determined in one of the subsequent measurements (where appropriate). For such an impedance measurement the procedure described by Roberts & von Hippel (1946) and von Hippel (1952) is used.

The following measurement (Fig. 2b) is of the impedance of a sample of finite thickness ($\mathcal{Z}_i/\mathcal{Z}_0 = \mathcal{Z}_1/\mathcal{Z}_0 \tanh \gamma_1 d$) from which impedance the dielectric constant can be determined as described by Benoit (1949) and Poley (1955a). To obtain reasonable accuracy all samples were measured over a wide range of thickness at each frequency. In the coaxial equipment, for example, the thickness of the 4% agar sample was varied between 4 and 17 mm at 3 Gc/s while at 6 Gc/s (waveguide WG 14) the thickness was varied between 2 and 12 mm. For the other samples measured the thickness range was about the same.

The method was used between 2 and 8 Gc/s and for all materials except liquids.

The third technique (Fig. 2c) is an impedance measurement on a sample of 'infinite' length ($\mathcal{Z}_i/\mathcal{Z}_0 = \mathcal{Z}_1/\mathcal{Z}_0$).

For the materials concerned a few centimetres suffice to obtain an 'infinite' length. Between 4 and 12 Gc/s, where the method was used with all materials except liquids, usually a thickness of 10–15 cm was chosen.

Calculation of the dielectric constant is easy and straightforward in this case. This method, however, is not very accurate and is usually combined either with the foregoing or with the following technique.

The fourth technique (Fig. 2d) depends on a measurement of the reflection coefficient, R, in front of a sample of 'infinite' length and a measurement of the attenuation, α, in the sample. This method gives the loss factor ε'' fairly accurately. It is only used between 8 and 18 kMc/s (two bands), and was applied to all materials except liquids. Since the method is novel, we shall describe it more extensively.

Use is made of an ordinary standing wave detector with a sturdy probe which, when necessary, can be pricked into the material to a predetermined depth. Measuring at a wavelength λ_v (cm) the complex dielectric constant $\tilde{\varepsilon} = \varepsilon' - j\varepsilon''$ is obtained as follows:

The attenuation in the sample in N/cm is:

$$\alpha = \frac{2\pi}{\lambda_v}\sqrt{|E|}\sin \tfrac{1}{2}\,\delta$$

where δ is the loss angle ($\tan \delta = \varepsilon''/\varepsilon'$; $\tilde{\varepsilon} = |\varepsilon| e^{-j\delta}$)

and $|E| = \sqrt{\left\{\varepsilon' - \left(\frac{\lambda_v}{\lambda_c}\right)^2\right\}^2 + \varepsilon''^2} \approx |\varepsilon|$ for the materials involved. λ_c = cut-off-wavelength of the waveguide used.

Then

$$\alpha = 8{\cdot}686 \frac{2\pi}{\lambda_v} \sqrt{|\varepsilon|} \sin \tfrac{1}{2} \delta \qquad (1)$$

where α is now expressed in dB/cm.

To give an impression of the values encountered, the figure obtained for α for the 4% agar mixture at 9375 Mc/s was 31 dB/cm. Furthermore it can be shown that for materials of high dielectric constant the square of the standing-wave-ratio η is:

$$\eta^2 \approx \frac{|E|}{\sqrt{1 - \left(\frac{\lambda_v}{\lambda_c}\right)^2}} \approx \frac{|\varepsilon|}{\sqrt{1 - \left(\frac{\lambda_v}{\lambda_c}\right)^2}} \qquad (2)$$

With the aid of formula (1) and (2) $|\varepsilon|$ and δ and thus ε' and ε'' can be determined.

For the measurement of liquids (Fig. 2e) we use the method given by Poley (1955b) where in front of a liquid column of variable length the reflection coefficient R is determined as a function of the thickness of the column.

From the curve $R = f(d)$ the dielectric constant is calculated.

Samples

Measurements were performed on samples of the following materials (in all cases the percentage by weight of the dry material in water is given).

(a) Agar gels, 4% and 8% in water. All samples were made from the same batch (Ion-agar, nr. 2, from Oxoid, density: 1·80 g/cm^3). Conductivity: 4% agar; 0·85 mMho/cm; 8% agar: 1·70 mMho/cm.

(b) Potato: 'Bintje', solid material: 23%. Average composition: 19% starch, 2% proteins, 2% cell walls. Conductivity, raw: 0·33 mMho/cm; blanched: 2·8 mMho/cm.

(c) Potato liquor; obtained from: Proefstation voor Aardappelverwerking TNO, Groningen and made by rasping and centrifuging of potato. Conserved with Na_2SO_3 and kept at 2°C until the measurement. Starch content 0%. Solid material 5%. Conductivity 11 mMho/cm.

(d) Potato starch. Obtained from: Laboratorium voor Levensmiddelen fabricage

en-techniek, Wageningen. The starch was stabilized with agar to a gel at a temperature so low that no hydration could occur. Final composition of gel: total content of solids 25·8%; agar 2%. Conductivity: 2·3 mMho/cm.

(e) Milk; Solids total: 12%; fat 3%; sugar 4·2%; proteins 3·7%. Conductivity: 4·1 mMho/cm.

Results

In Figs. 3–5 the results of measurements on the samples mentioned above are represented in the form of Cole–Cole arcs (Cole & Cole, 1941) after correcting the values of ε'' for d.c. conductivity. All measurements are compared with the results obtained for water as reported in the literature (Poley, 1955b; Hasted, 1961). Within the measuring accuracy all points lie on semi-circles. Similar measurements by Cook (1951, 1952) and England (1950) on blood, muscle and skin, plotted in Fig. 6, show the same behaviour.

The value for $\varepsilon_{m\infty}$, the intersection with the ε'-axis at the high-frequency end, is taken as 5·5, i.e. that for water (Hasted, 1961). The intersection at the low-frequency end, ε_{ms}, is easily determined from the plots.

For 4% and 8% agar solutions no shift in relaxation frequency with regard to that of water is observed. For potato a shift to lower frequencies occurs, the deviations, however, being small.

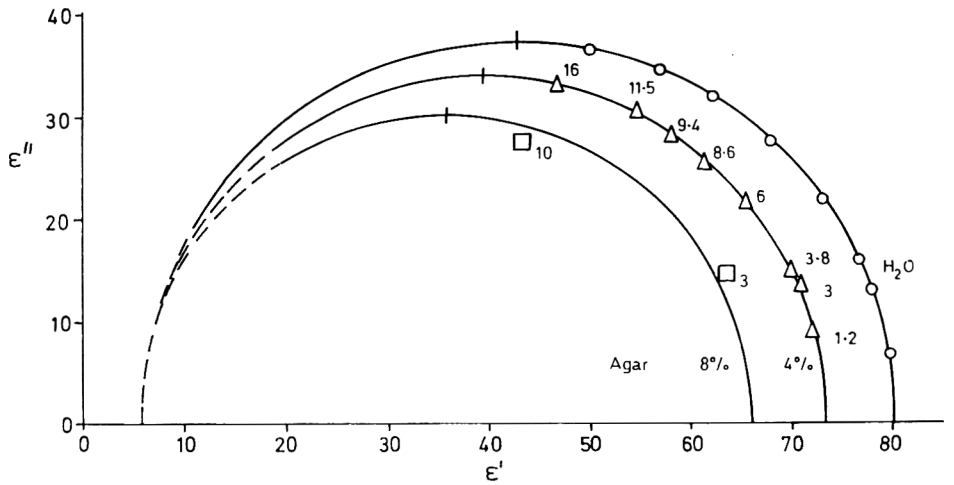

FIG. 3. Measurements on agar, T = 20°C. Frequencies indicated at the measuring points in Gc/s.

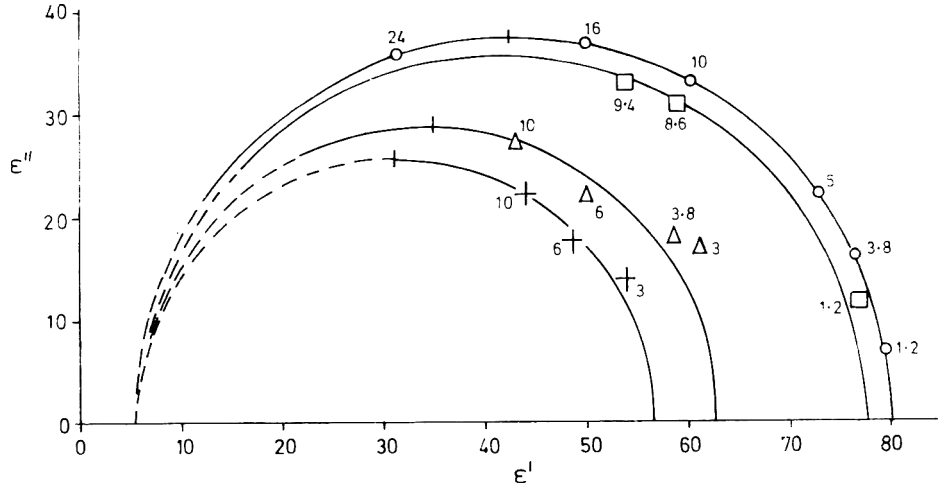

FIG. 4. Measurements on potato, △; potato liquor, □ ; and starch (2% agar + 25% starch), + ; T = 20°C. Frequencies indicated at the measuring points in Gc/s.

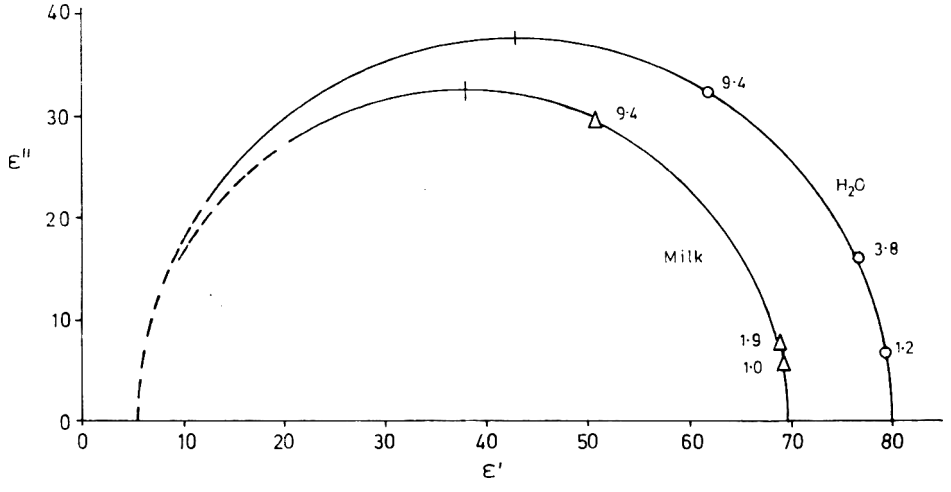

FIG. 5. Measurements on milk, T = 20°C. Frequencies indicated at the measuring points in Gc/s.

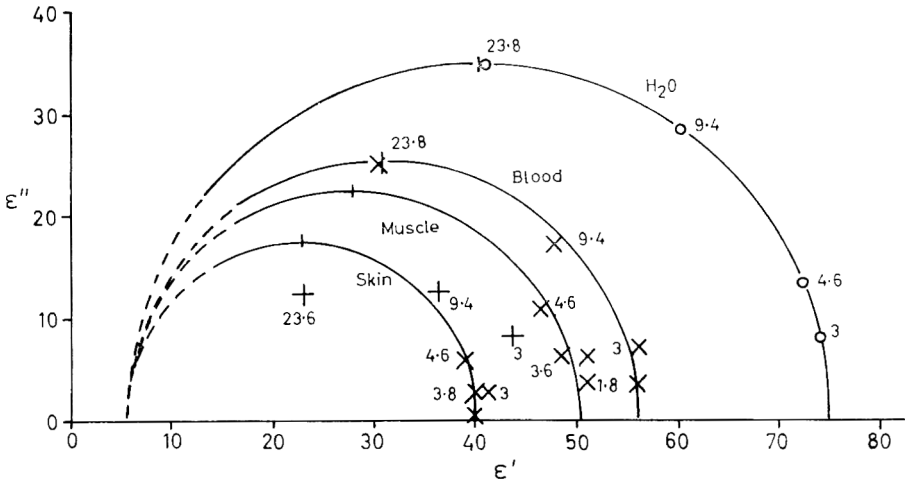

FIG. 6. Measurements on blood, muscle and skin after Cook (1951, 1952) and on skin after England (1950). T = 37°C. Frequencies indicated at the measuring points in Gc/s.

Discussion

All measurements reported concern systems with a very high water content. Since for all materials investigated semi-circles are found for the function $\varepsilon''_m = f(\varepsilon'_m)$ it can be concluded that they all show relaxation in the microwave region and have one relaxation time within the measuring accuracy. This relaxation can be attributed to the relaxation of the free water available (*vide* Fig. 1). This also means that to a first approximation it becomes possible to predict the value of the dielectric constant at different frequencies when ε_{ms} is known with the aid of the formulae (Bottcher, 1952; de Loor, 1956).

$$\varepsilon'_m = \varepsilon_{m\infty} + \frac{\varepsilon_{ms} - \varepsilon_{m\infty}}{1 + \omega^2\tau^2} \tag{3}$$

$$\varepsilon''_{m\ dip} = (\varepsilon_{ms} - \varepsilon_{m\infty}) \frac{\omega\tau}{1 + \omega^2\tau^2} \tag{4}$$

where $\varepsilon_{m\infty} = 5{\cdot}5$ and τ: the relaxation time of water. To find ε''_m at the lower frequencies, to the dipole contribution $\varepsilon''_{m\ dip}$ must be added the conductivity loss $\varepsilon''_{m\sigma}$ ($= 60\lambda \cdot \sigma$ with λ, the wavelength in cm and σ the conductivity in Mho/cm). Thus

$$\varepsilon''_m = \varepsilon''_{m\ dip} + \varepsilon''_{m\sigma}$$

To a first approximation ε_{ms} can be predicted from mixture relations (de Loor, 1956, 1964). The accuracy of the figures obtained in this way is sufficient for many applications.

This makes it possible to design model systems which can be used in experiments with microwave equipment, as for example in the testing of microwave heating ovens. These model systems can be made with a reasonable reproducibility, to simulate the material that is finally to be used in practice.

Agar, with the addition of fillers to vary the dielectric constant and other properties as, for example, specific heat, is a good base material for such purposes.

Since we measured the dielectric constant ε_{ms} of the materials under consideration we can also use the mixture relations already mentioned in the last section to determine the amount of 'bound water', water so tightly bound to its surroundings that it shows no relaxation in the microwave region ('irrotationally bound water'; Buchanan *et al.* (1952) and Hasted (1961)). In the last section we knowingly overlooked the variations in the results due to this 'bound water' as the figures obtained from the mixture relations (with the low-frequency values for the dielectric constant of the components) and equations (3) and (4) are accurate enough for many applications. De Loor (1956, 1964) describes the behaviour of heterogeneous mixtures and gives limits between which the dielectric constant of such a mixture lies for a certain volume-filling-factor v_i when the dielectric constants of the individual components are known.

The determination of the amount of 'bound water' can be effected by the method of Buchannan *et al.* (1952) and Hasted (1961). We shall not venture here to go further than following their procedure and interpretation. For the systems under consideration we then approximate the formulae for the limiting values of the dielectric constant ε_{ms} with

$$\varepsilon_o - \varepsilon_{ms} = \frac{v_i}{1-v_i} (\varepsilon_{ms} - \varepsilon_i) \cdot \beta \tag{5}$$

as upper limit and

$$\varepsilon_o - \varepsilon_{ms} = v_i (\varepsilon_o - \varepsilon_i) \cdot \beta \tag{6}$$

as lower limit, where

$$\beta = \sum_1^3 j \frac{1}{1-A_j}$$

assuming the inclusions to be ellipsoidal with A_1, A_2 and A_3 being the depolarization factors along the main axes of the ellipsoid. ε_o = static dielectric constant of the continuous phase, water in this case, thus $\varepsilon_o = 80$ (at 20°C); and ε_i = static dielectric

constant of the dispersed phase, the organic material, and taken here as 2·5–3. $\beta = 1{\cdot}5$ for spheres, 1·67 for needles and very large for disc-shaped inclusions.

By comparing the measured dielectric constant with that predicted by the above theory, the difference gives the amount of 'bound water', assuming that this 'bound water' has about the same dielectric constant as the organic material at microwave frequencies (compare with ice: $\varepsilon \approx 3{\cdot}2$), a reasonable assumption because a variation in ε_i of, for example, from 2·5 to 5 gives variations in the results which remain within the accuracy of the determination of the 'bound water' as described. In this way Table 1 is obtained.

TABLE 1

	ε_{ms}	β	Theory $v_i(\%)$*	Exp. $v_i(\%)$†	'Bound water' $v_i(\%)$	g/g	g/cm³
Agar 4%	73·5	1·67	5·2	2·2	3	0·75	1·4
Agar 8%	66	1·67	11·4	4·5	7	0·88	1·6
Starch	56·5	1·5	21·3	17·1	4	0·16	0·23
Potato	63	1·5	15·3	15·3	—	—	—
Potato liquor	77·5	1·5	2·3	3	—	—	—
Milk	69·5	1·5	9·4	8	1	—	—

* Mean value of v_i derived from equations (5) and (6).
† v_i determined from density of known constituents.

According to the procedure followed the agar contains much 'bound water': 0·8 g/g or 1·5 g/cm³. The arbitrary choice of $\beta = 1{\cdot}67$ (needles), however, makes this conclusion somewhat uncertain. When a higher value of β must be assumed the calculated amount of 'bound water' will decrease. Looking at the further results we may conclude that the 'bound water' found for the starch probably also is due to water bound to the 2% agar used to stabilize the gel. The figures given by Buchanan *et al.* (1952) and Hasted (1961) for protein hydration (0·2–0·3 g/cm³) suggest that the low figure found for milk is possibly due to protein hydration. When this is assumed we find $\sim 0{\cdot}25$ g/g or $\sim 0{\cdot}2$ g/cm³ for the water bound to the proteins in milk in accordance with their value.

Conclusions

Difficulties associated with the measurement of the dielectric properties of materials with high dielectric constants and losses have been overcome by means of a new technique in which the attenuation of the EM-wave in the material is determined and reasonably high accuracy is obtainable.

The losses of the materials under consideration are primarily determined by the

dipolar losses of the free water and, at the lower frequency end, also by d.c. conductivity.

Though in some cases small shifts are observed in relaxation frequency relative to that of water it is possible to calculate the dielectric constants of these watery materials with a reasonable accuracy with the aid of the available formulae for mixtures because only one relaxation time is involved. It is thus possible to design model systems, and in this respect agar is a very suitable material, particularly since it may be used together with fillers to simulate thermal and other properties in addition.

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The effect of phosphate solutions on the denaturation of frozen cod muscle

R. M. LOVE* AND G. ABEL†

Summary. The effect of treating cod fillets with phosphate solutions prior to freezing was studied. Little influence on subsequent denaturation of the cold stored material was found, as measured by protein extractability in dilute salt solution. However, the treatment invalidated the cell fragility method by producing very low values. Electron microscopy showed that these were the result of the solvent action of the phosphate solutions, which removed some of the protein from the myofibrils and made the cell fragility homogenate correspondingly less dense. Some of the dissolved protein was shown to be deposited on the surface of treated fillets, and it is the resulting film which probably seals the fluids in the fillet and causes the well-known reduction in thaw-drip.

Introduction

In 1962, Mahon patented a process for reducing the fluid which usually exudes from frozen and cold-stored fish after thawing. The process comprised 'Contacting the said fish flesh prior to freezing with a solution of alkali metal chloride and a compound selected from the group consisting of sodium and potassium salts of molecularly dehydrated phosphoric acids having a molar ratio of H_2O to P_2O_5 of from about 1 to 1 to about 2 to 1.' The claim for the effectiveness of the treatment was supported by considerable experimental data which also showed that the most effective additive to the brine solution was sodium tripolyphosphate. Mahon stated that the exact mechanism by which his invention produced its remarkable results was not known.

In the original specification, no claims were made for the treatment apart from a reduction in thaw-drip, but articles in the trade press (Milleville & Leinen, 1962; Anon., 1962) have extended the claims somewhat to include various desirable features from the consumer's point of view.

When the work was repeated in other laboratories, the reduction in fluid exudate was confirmed in a number of instances (Tanikawa, Akiba & Shitamori, 1963; Ohta

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& Nishimoto, 1963; Mahon & Schneider, 1964; MacCallum *et al.*, 1964a, b; Pienaar, 1964; Boyd & Southcott, 1965; Chalker, MacCallum & Idler, 1965) though not in all (Dyer *et al.*, 1964). One report stated that only in fish frozen and thawed twice was there any clear advantage in using polyphosphate (MacCallum *et al.*, 1964b), and another that the reduction in fluid loss was only marked under conditions where it would have been slight anyway (Anon., 1963).

The texture of frozen muscle tissue when eaten after cooking has been reported to be improved by prior treatment with polyphosphate solution (MacCallum *et al.*, 1964a; Klose, Campbell & Hanson, 1963). It is when various criteria of cold-storage deterioration are examined in relation to each other that the picture becomes less clear. During storage of frozen fish muscle at a given temperature, the main structural proteins become increasingly insoluble in dilute salt solution after thawing out (Reay, 1933) according to the time of storage. The development of insolubility (strictly speaking 'reduced extractability') proceeds in a similar fashion to the development of toughness when the cooked product is tasted (Dyer, 1951), and the two criteria are generally considered to be two aspects of the same phenomenon, 'denaturation' (Love, 1966a), fluid exudation being a third. Assuming that a polyphosphate dip really does inhibit fluid loss, it is therefore surprising to find that, while one laboratory reports a concurrent inhibition of the insolubilization of the protein (Ohta & Nishimoto, 1963), two others (Dyer *et al.*, 1964; Anon., 1964) state that there is no effect on the extractability, and two claim that polyphosphate actually decreases it, i.e. causes deterioration (Tanikawa *et al.*, 1963; Nikkilä, Linko & Kuusi, 1964). The behaviour of the muscle as studied by fluid exudation and by protein solubility is evidently not the same, calling into question the fact that a single phenomenon is involved during cold storage.

In view, therefore, of the contradictory state of the published accounts, the following work was carried out in an endeavour to shed more light on the mechanism of phosphate activity.

Materials

The cod used throughout the work were caught by trawl net within a 30 mile radius of Aberdeen. They were gutted at sea and stowed in melting ice for 1 or 2 days before arrival at the laboratory, where they were filleted by a professional filleter and used with the minimum of delay.

The phosphates used were as follows:

Tetrasodium pryophosphate, $Na_4P_2O_7$

Sodium hexametaphosphate $(NaPO_3)_6$

Disodium dihydrogen pyrophosphate $Na_2H_2P_2O_7$.

These three phosphates were British Drug Houses 'Laboratory Grade' reagents.

Sodium tripolyphosphate

This was Albright and Wilson 'Food Grade' material. It will be referred to as 'polyphosphate' in this paper for the sake of brevity.

Methods

Protein extractability was carried out following the procedure of Ironside & Love (1958), and the cell fragility method was used as described by Love & Mackay (1962).

Samples for determination were prepared as follows. The anterior third of each fillet was chopped into five pieces each weighing about 50 g. One sample for determination consisted of ten such pieces, each from a different fish, the intention being to reduce some of the variations resulting from biological factors. The ten pieces were soaked in phosphate solution at 0°C for 2 min, drained, and wrapped together in a sheet of aluminium foil and frozen in air at −30°C. After thawing at room temperature, about 18°C, one cell fragility determination was performed on each piece in the sample and the results were averaged. When protein extractability was being measured, a roughly equal quantity of tissue was removed from each piece, those from the ten pieces in a sample being mixed together and chopped with scissors. Duplicate quantities of 1 g were then taken from the mixture, and the results averaged.

Histological sections of the frozen material were prepared by cutting a 5 mm cube of muscle from a larger piece with a fine-toothed saw at −29°C and vacuum-drying it at the same temperature, essentially as described by Koonz & Ramsbottom (1939). The specimen was then vacuum-embedded in paraffin at 50°C, sectioned at 5 μ, and stained with haematoxylin and eosin, when the spaces formerly occupied by ice crystals showed up as cavities.

Where it was desired to observe the appearance of the tissue before freezing or after thawing, a similar cube was gently cut from the soft muscle with a scalpel and dropped into liquid propane which had been cooled to −190°C in a copper container by partial immersion in liquid air. This procedure fixed the tissue instantaneously, and made ice crystals of minute size which could be ignored when examining the picture. The tissue was then vacuum-dried at −35°C and embedded as before. All cubes had as one of their faces the surface of the fillet exposed to the action of the phosphate solution, and the specimen was orientated for sectioning by placing a spot of Indian ink on the face diametrically opposed to this at the time of excision from the fillet.

Homogenates of treated muscle prepared by Love & Mackay's 'Cell Fragility Method' (1962) were supported on formvar and shadowed with gold–palladium for electron microscopy.

Results and discussion

Since a reduction in the exudation of fluid after thawing had frequently been reported in the literature, but protein extractability not so definitely characterized, it was

decided to measure cold-storage deterioration by another criterion, 'Cell Fragility' (Love & Mackay, 1962). In this technique, a small sample of fish muscle is subjected to mild, accurately controlled homogenization in 1% formaldehyde solution. The muscle of fresh, post rigor mortis fish disintegrates to a suspension of individual cells, which are themselves disintegrated further to myofibrils, resulting in an opaque, homogeneous suspension. Denaturation of the muscle in the cold store causes a progressive strengthening of the bonds holding the myofibrils together, so that larger and larger bundles of myofibrils are seen in the homogenate of the thawed material, until in very tough ('denatured') muscle the cells are not degraded at all, but suspended, relatively few in number, in a crystal clear fluid. The effect of cold storage is therefore to reduce the opacity of the homogenate, and the extent of denaturation can readily be assessed by means of a colorimeter. The quantity measured is of course the scattering of light, not true absorption.

The effects of three phosphates on denaturation, as measured by cell fragility, were studied in the first experiment (polyphosphate was not then available to us). The solutions (9% in water) were neutralized by the addition of acid or alkali, and after the samples of fish had been dipped and frozen they were stored at −9°C in order to obtain rapid denaturation. The fish had been caught on 29 June 1964.

Fig. 1 shows that very low figures, usually indicative of much denaturation (Love

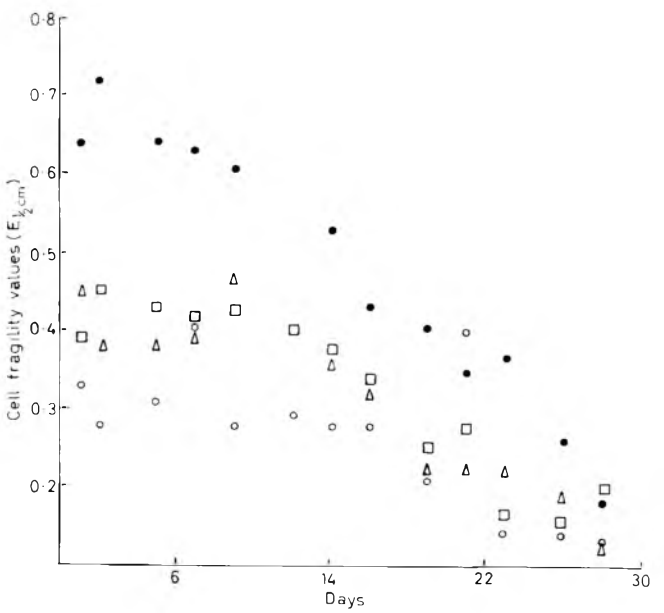

FIG. 1. Cell fragility values (optical density) of samples of cod frozen at −30°C and stored at −9°C for different times after treatment with various phosphate solutions. ●, Control (undipped); △, $Na_2H_2P_2O_7$; □, $Na_4P_2O_7$; ○, $(NaPO_3)_6$.

& Mackay, 1962) resulted even before cold-storage had started. The values from sodium hexametaphosphate were slightly lower than those from the other phosphates, and since all the solutions were neutralized, the difference was not a pH effect. The curve of the controls (undipped) descended smoothly from a high value in the manner already characterized (Love & Mackay, 1962).

When polyphosphate became available, conditions were arranged to be as close as possible to Mahon's original specification (1962).

Pieces taken from fish caught on 16 February 1966 were dipped for 2 min in a chilled 12·5% solution of sodium tripolyphosphate containing 4% NaCl, then drained before freezing. The pH of this solution was 8·05, and that of 12·5% polyphosphate alone, which was used for comparison, was 8·45. The solutions were not neutralized in this experiment.

Apart from more random scatter in the control values, the results (Fig. 2) are

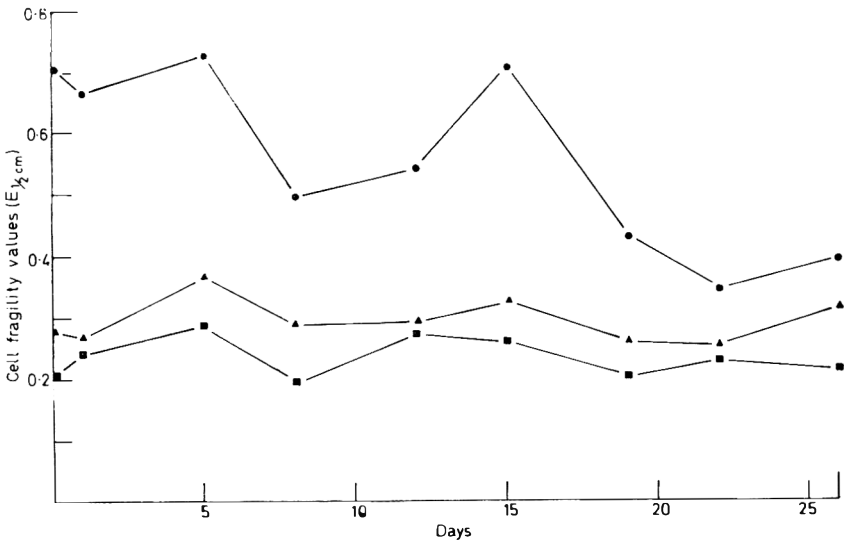

FIG. 2. Cell fragility values (optical density) of samples of cod frozen at −30°C and stored at −9°C for different times after treatment with sodium tripolyphosphate solution. ●, Control (undipped); ■, 12·5% sodium tripolyphosphate; ▲, 12·5% sodium tripolyphosphate containing 4% NaCl.

similar to those of Fig. 1, but the initial values of the samples treated with polyphosphate were now so low that no further decrease occurred during subsequent storage. The values obtained with polyphosphate alone were slightly lower than those with polyphosphate and sodium chloride.

Although the protein extractability of cold-stored fish muscle treated with polyphosphate had been reported in the literature a number of times, it was decided to

carry out a check in the present instance after the unexpected results of Fig. 2 had been obtained. Extractability was measured on the samples stored for 15 and for 26 days at −9°C, and the results are shown in Table 1.

TABLE 1. Extractability in 5% NaCl of protein nitrogen of cod muscle after freezing and storage at −9°C, with and without prior treatment with polyphosphate solution

Treatment	Protein N extracted (% of total protein N)	
	15 days at −9°C	26 days at −9°C
Control (undipped)	62·0	41·8
12·5% polyphosphate	59·4	50·9
12·5% polyphosphate +4% NaCl	60·6	48·2

Values are the mean of duplicate determinations.

It is clear that the effect of polyphosphate on protein extractability was indefinite and slight, and not to be compared with the overwhelming effect on cell fragility values. Here then appeared to be a paradox, since earlier work had indicated that cell fragility values and protein extractability always went hand in hand (Love, 1962a). However, more recently Love & Olley (1965) showed that during cold-storage the reaction rates of denaturation as measured by the two techniques were not the same with species of fish other than cod, and Love *et al.* (1965) showed that the optical density values from the cell fragility test could diminish for reasons other than a side-to-side aggregation of the myofibrils. They found that when fish were kept over 30 days packed in melting ice, the homogenates showed a decrease in optical density: they did not, however, exhibit the aggregation seen after frozen storage but instead became paler or 'misty'. Examination of the homogenates with a microscope showed that the myofibrils had been broken into much shorter lengths, probably by a weakening of the Z-band material.

Examination with an optical microscope in the present instance revealed homogenates of a similar appearance to that of the homogenates of very stale fish reported by Love *et al.* (1965), so they will not be illustrated again here. However, electron micrographs yielded further information.

Plate 1(a) shows part of a homogenate of normal fresh cod muscle, 2 days after catching, optical density 0·85. The two myofibrils seen are very sharply defined and dark-stained. Plate 1(b) shows a similar homogenate (optical density 0·24) of a sample of cod taken from near the surface of a fillet which had been dipped for 2 min in 12·5% polyphosphate containing 4% NaCl. The right-angle breaks can again be seen, but also obvious is the fact that much of the protein has been dissolved out of

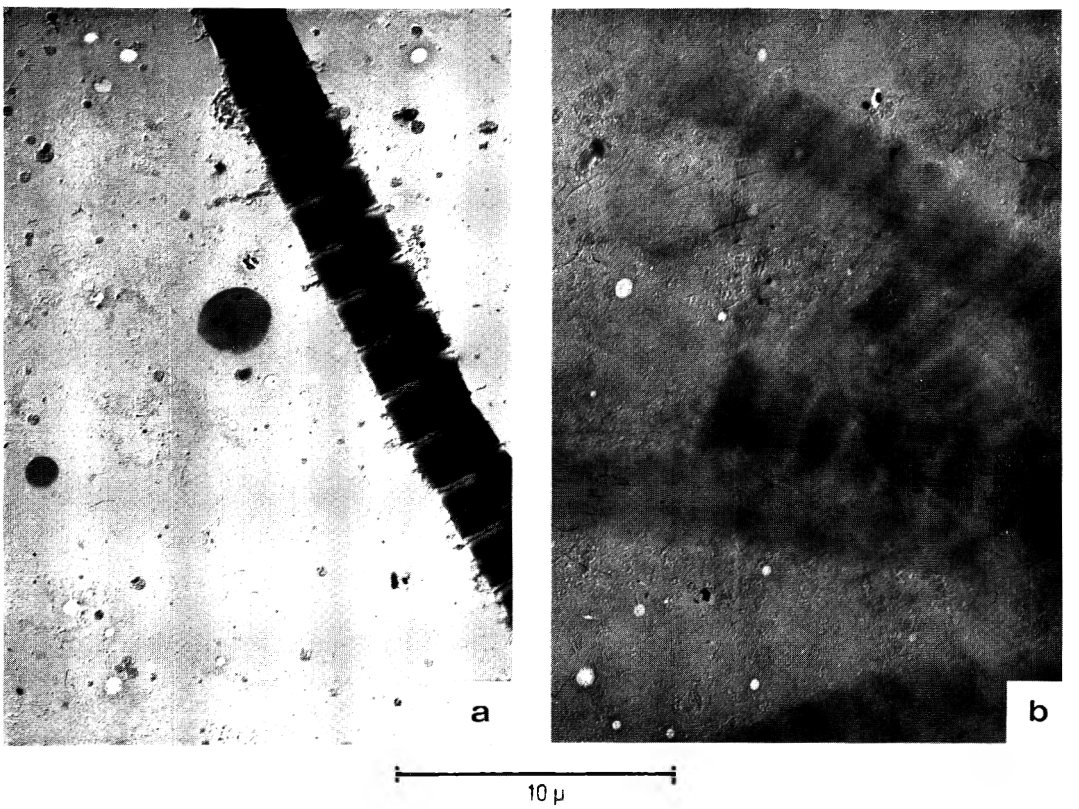

PLATE 1. Electron micrographs of homogenates of cod muscle obtained by the cell fragility apparatus. (a) Control (undipped). (b) After treatment with 12·5% sodium tripolyphosphate containing 4% NaCl.

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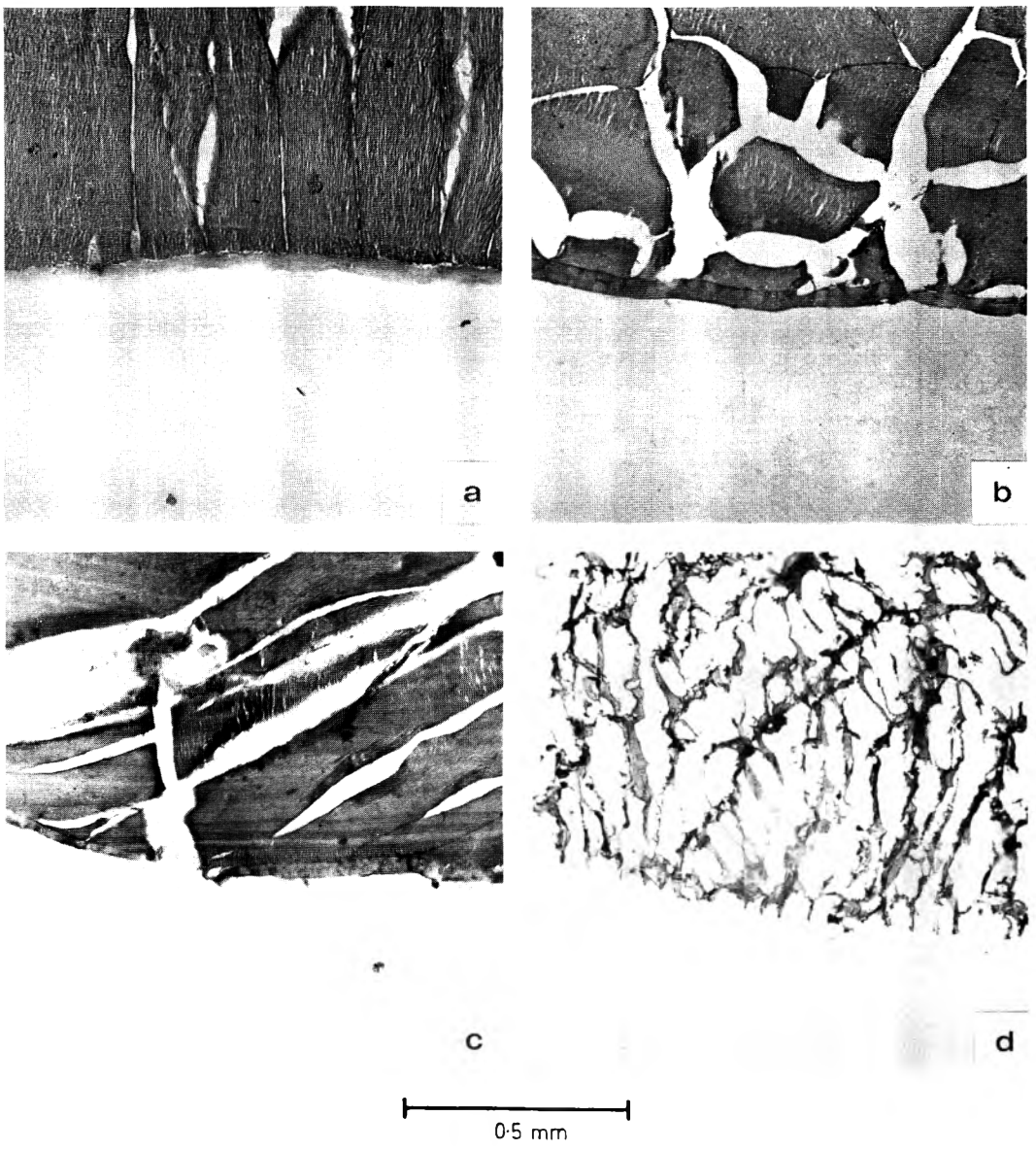

PLATE 2. Sections of cod muscle showing the face of the fillet after dipping in phosphate solution. Fish in (a), (b) and (c) were dipped in, respectively, $(NaPO_3)_6$, $Na_4P_2O_7$ and polyphosphate, drained, frozen in air-blast at $-30°C$ and thawed the following day. The appearance in the thawed state is shown. Plate 2(d) shows the appearance while still in the frozen state of the fish previously dipped in polyphosphate as in (c); light areas correspond with ice.

the myofibrils. Notice how the pale circular flaws in the formvar support can be seen *through* the fibrils, which show very poor contrast although the specimen was prepared exactly as in Plate 1(a).

The solvent action of the polyphosphate solutions was next investigated quantitatively. One-gram samples of fresh post-rigor cod muscle were homogenized exactly as in the standard technique for protein extractability (Ironside & Love, 1958) but using polyphosphate solution instead of sodium chloride. The fish were caught in April 1966, which probably explains the rather low figures for the control. [Ironside & Love (1958) gave a detailed account of the biological factors governing the extractability of cod muscle protein: in April, the fish have not yet fully recovered from spawning.]

TABLE 2. Extractability of the protein nitrogen of cod muscle in polyphosphate solutions

Extractant	Protein N extracted (% of total protein N) (average of two determinations)
Water	20·1
5% NaCl (control)	87·8
12·5% polyphosphate alone	60·6
12·5% polyphosphate + 4% NaCl	69·7

It can be seen from the results in Table 2 that polyphosphate exerts a strong solvent action on the muscle proteins, though not as great as that of 5% NaCl. The solvent action of the polyphosphate–NaCl mixture is slightly greater than that of polyphosphate alone. The partial dissolution of the surface proteins presumably accounts for the rather slimy feel of the fillets after treatment with polyphosphate.

The mechanism whereby fluid exudate is reduced was shown by a histological study of the intact muscle. It was found that when the muscle was dipped in phosphate solution, frozen at −30°C and then thawed, the surface of the fillet became covered by a layer of protein not seen in undipped specimens. This is presumed to be the direct result of leaving the fillets to drain after dipping, covered by a layer of the fluid which has a solvent action on the muscle. Sodium chloride alone, long known to diminish post-thawing fluid exudate, was always thought to form a 'skin' on the surface, but no histological demonstration of it has come to the authors' attention.

As far as could be judged, the action of all the phosphates was similar. Examples are given in Plate 2, which shows the appearance after thawing of cod muscle previously dipped in (a) sodium hexametaphosphate, pH 6·2, (b) tetrasodium pyrophosphate, pH 9·92, and (c) polyphosphate + NaCl, pH 8·05. The layer of protein runs along the margin of the specimen in each case, being somewhat less distinct in (a). The partial dissolution of the muscle cells by the phosphate solutions is not apparent in these preparations, but it is clear, especially in Plate 2(a) and (b), that the layer of

protein effectively seals off the channels at the surface which communicate with the extracellular space. The phosphates do not, as had been at first suspected, cause the cells to swell and the extracellular space to disappear.

Plate 2(d) shows the appearance of the tissue treated with polyphosphate while it was actually in frozen state: the colourless areas in the tissue represent the spaces occupied by ice. The layer of protein cannot be detected, being much broken up by ice in the same way as the rest of the tissue. Experience of the appearance of many sections of frozen cod muscle leads to the conclusion that the actual quantity of ice present in the specimen illustrated in Plate 2(d) is close to that in normal undipped post-rigor muscle, although examination of the homogenates obtained by the cell fragility method (Plate 1b) shows that the polyphosphate must have penetrated into the tissue at least as far as the upper margin of Plate 2(d). The significance of this observation will be discussed in a moment.

As already stated, it has long been known that dipping in NaCl solution would reduce fluid exudate. However, the disadvantage of using a solution of this kind before freezing is that the oxidation of the fats of the fish during cold-storage is accelerated, probably because the salt, through diffusing into the tissues, increases the quantity of liquid still unfrozen at the usual cold-storage temperatures (Banks, 1952). Atmospheric oxygen diffuses more rapidly through a liquid phase, and thus gains access to the muscle fats. Now it was reported by Boyd & Southcott (1965) that treatment of chinook salmon fillets with polyphosphate before freezing did not accelerate oxidative rancidity, although it did not retard it either. It was felt profitable in the present work, therefore, to measure the freezing point depression of the polyphosphate solutions, using an apparatus as described by Ramsey & Brown (1955).

The results were as follows:

Freezing point of 12·5% polyphosphate: −1·24°C

12·5% polyphosphate + 4% NaCl: −3·347°C

Clearly the freezing point depression in fish treated with such solutions would have a negligible effect on fat oxidation in fish stored at, say −20°C, while it is easy to understand the result of using saturated NaCl, which freezes at −21°C, to brine-freeze herrings, which rapidly become rancid at that temperature.

What, then, can we conclude about the action of polyphosphate in frozen fish?

When tissue freezes, a large proportion of the water separates out into ice crystals. The salts occurring naturally in the tissue therefore become greatly concentrated, and it is thought that, by providing favourable conditions for the release of free fatty acid from phospholipid, these concentrated salts are in part responsible for the denaturative changes which occur in the proteins—toughness on eating, increase in fluid loss on thawing, and other alterations in the properties of the proteins. The subject has recently been reviewed in detail (Love, 1966a). The point is that if the proportion of ice to unfrozen water can be reduced in any way, the tissue salts are less concentrated

and less denaturation should occur. This has been demonstrated. If the proportion of ice is reduced by freezing the muscle pre-rigor (Love, 1962b) or by prior treatment with glycerol (Love & Elerian, 1965) or by freezing at a relatively high temperature, so that much of the water is still bound to the protein (Love, 1966b), the rate of denaturation is reduced. Now there is no sign of any reduction in the amount of ice formed in fish dipped in polyphosphate when the pattern illustrated in Plate 2(d) is compared with patterns normally obtained with untreated fish (Love, 1966c), so that any reduction in denaturation would be by some other mechanism.

In fact, from the papers reviewed in the introduction and from Table 1 in the present work, it seems unlikely that polyphosphate inhibits denaturation at all. The reduction in fluid exudation appears to be by mechanically sealing off the surface of the fillet (Plate 2), keeping the ice melt-water which was imperfectly reabsorbed by the denatured protein within the fillet, but still presumably in the 'free' state. The sealing agent is a uniform layer of protein, dissolved out of the surface layers of the muscle by the solvent action of the polyphosphates: this is probably why, if the fillet is dipped after thawing, the fluid loss is only inhibited usefully where the fish is but slightly denatured (Anon., 1963)—extensive denaturation would render most of the protein inextractable, and therefore unable to form the sealing 'skin'. Where the fillet had been dipped before freezing, then it is probable that denaturation would affect the protein of the 'skin' as well, and render it porous.

Nikkilä *et al.* (1964), one of the groups who found that polyphosphate treatment led to increased denaturation, said 'The inventor (of the polyphosphate process) studied only the drip that takes place during the thawing of the fish without taking into account the protein denaturation, which is of primary importance in respect of the quality of the fish.'

Perhaps the process can best be said to suppress one of the symptoms, rather than to cure the disease.

However, this is not to condemn the use of polyphosphates in fish technology out of hand. It obviously has a useful place in the reduction of fluid exudation in, for example, fresh fish filleted soon after death and sold in transparent plastic packs. Nothing would be more likely to discourage purchase than the sight of varying amounts of a turbid liquid, probably brown, sealed up in the pack with the fish, and it seems that a dip in polyphosphate would do much to reduce this. The loss of fluid from frozen fish after thawing is of economic importance, and provided the denaturation has not gone too far it seems clear that there are advantages in this direction also. The deposition of protein on the surface of the fish following a dip improves its gloss and this (cosmetic) effect may be thought worth while in certain instances. A slight improvement in the texture characteristics of treated frozen fish, reported by MacCallum *et al.* (1964a), possibly relates to the partial extraction of the tissue at the surface, which, judging by Plate 1 (b), should become softer in consistency, but this would only be achieved if the

tissue were not badly denatured to start with, and would only affect the outer layers—the migration of phosphates deeply into fish tissue is very slow (Kuusi, Nikkilä & Kytökangas, 1965).

Taking the literature on polyphosphate as a whole it does appear that certain improvements may sometimes result from its use. However it is clearly no substitute for good freezing and cold-storage practice, and will not be of benefit if the fish is denatured.

Acknowledgments

The work described in this paper was carried out as part of the programme of the Ministry of Technology. Some of it was done during the tenure of a vacation studentship by one of us (G.A.), and in the remainder much of the experimental work was carried out by Mr John Smith. The freezing point measurements were kindly done by Mr F. G. T. Holliday of the Zoology Department, Aberdeen University.

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The relationship between the toughness of cod stored at −29°C and its muscle protein solubility and pH

W. P. COWIE AND W. T. LITTLE

Summary. Protein solubility, muscle pH and organoleptic toughness have been measured in cod fillets stored for up to 82 months at −29°C. During this time protein solubility dropped steadily from 72% to 45% indicating that protein denaturation had taken place.

The organoleptic toughness of the fillets showed no correlation with this decrease in protein solubility; indeed, some of the control fillets with maximum protein solubility were tougher than others with the lowest protein solubilities after 82 months storage. Good correlation was found between organoleptic toughness and the pH of the raw muscle.

Protein solubility by itself is therefore not a meaningful test for the eating toughness of frozen cod stored at −29°C. In cod stored at higher temperatures than −29°C there will be a greater rate and degree of protein denaturation, which may then become more important than muscle pH in determining the toughness of cooked cod.

Introduction

Deterioration in fish products as a result of frozen storage is well known; 'dry', 'woolly' and 'tough' are common descriptions of the texture of cooked frozen fish, especially cod.

The exact causes of this textural deterioration in frozen cod flesh are not yet known. It has been suggested that during frozen storage, chemical cross links are formed between the myofibrillar proteins, myosin and actin, and between myosin molecules themselves (Connell, 1962). These reactions may be the reason for the development of toughness in the cooked frozen fish, and also for the observed decrease in the solubility of the myofibrillar proteins of the raw flesh in neutral 5% sodium chloride solution with time of frozen storage.

The term 'cold storage denaturation' is used to describe the changes taking place in the proteins during frozen storage. Dyer & Dingle (1961) have defined denaturation as 'a change in the protein such that it is no longer soluble or extractable by salt

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solutions under conditions in which native protein is soluble or extractable'.

In fresh cod almost all of the muscle proteins are soluble in 5% sodium chloride solution, but the amount of soluble protein becomes reduced after frozen storage. Several workers have investigated the protein changes in frozen cod stored at different temperatures by measuring the change in the solubility of the proteins in 5% sodium chloride solution.

Dyer (1951) showed that in cod fillets stored at −12°C and −23°C, denaturation of the myofibrillar proteins was more rapid at the higher storage temperature. At −23°C the solubility of these proteins did not change appreciably for the first 2 or 3 months, then it gradually declined to low values at about 9 months. The same protein fraction in fillets stored at −12°C became completely insoluble after only 15 weeks. For cod fillets stored at −12°C and −23°C, Dyer reported good correlation between denaturation as measured by protein solubility and toughness as measured organoleptically.

Love (1956) found that increased toughness determined organoleptically in cod stored at −30°C did not always involve decreased protein solubility. Luijpen (1957) also stated that organoleptic toughness could not be related to protein solubility in 5% sodium chloride for cod stored at −30°C or −20°C. During a storage period of a year at these temperatures he found little or no decrease in protein solubility whereas the toughness measured organoleptically increased over this period. Later Dyer & Fraser (1959) showed that there was neither a decrease in protein solubility nor an increase in organoleptic toughness in cod stored at −23°C for 16 months.

Love (1962) related toughness as measured by his cell fragility method to the decrease in protein solubility of cod stored at −14°C, −20°C, −24°C and −29°C, though later he and his colleagues (Love *et al.*, 1965) found that cell fragility can change independently of protein extractability. In a more recent paper (Kelly *et al.*, 1966) the significance of cod muscle pH in determining toughness organoleptically and also by the cell fragility method was realized. Previous workers tended to overlook the importance of fish muscle pH in studies on fish texture, although muscle pH in meat was known to effect its water binding capacity and hence its tenderness (Hamm, 1960; Deatherage, 1963). Little & Smithies (1963) did point out that in unfrozen cod a muscle pH below 6·4 was usually associated with a high liquor loss on cooking and a tough texture, while a high pH (above 6·8) was always associated with a low liquor loss and a soft texture.

Our experience in this laboratory has shown that the ultimate post-mortem pH of cod muscle can vary from 7·0 to 5·9. Therefore any work involving the organoleptic measurement of the texture of cod muscle necessitates a knowledge of the muscle pH. This work was undertaken to establish the true relationship between the toughness of cod stored at −29°C and the extractability of the muscle proteins in 5% sodium chloride solution, taking the muscle pH into account.

Materials and methods

The cod used in the experiment were from two sources. The controls were obtained from Aberdeen Fish Market and had just passed through rigor. They were filleted and the fillets, after being placed in sealed polythene pouches, were air blast frozen at −29°C. The long-stored fillets were obtained from Dr R. M. Love of Torry Research Station and were closely wrapped in aluminium foil which had prevented desiccation during storage at −29°C. They were from cod all about 20 in. long caught by the Station's research trawler at the same fishing grounds about 30 miles south-east of Aberdeen between January 1959 and June 1964. Three fillets for each of nine different storage periods were obtained.

Experimental procedures

Extractable protein estimation

The extractable protein of each fillet was determined in quadruplicate by a modification of the method of Ironside & Love (1958). For each determination the sample of frozen flesh was taken from near the head of the fillets (in the region of myotomes 9–12) and was freed from connective tissue using a scalpel. All work was done in a cold room at 2°C. About 1 g of frozen fish was weighed into the stainless steel cup of a Marsh-Snow homogenizer fitted with a baffle plate to prevent frothing. About 15 ml of ice-cold 5% sodium chloride solution adjusted to pH 7·0–7·5 were added and the mixture was macerated for 1 min at full speed (about 2200 rev/min), the cup being surrounded by ice and water. The homogenate was washed with 5% sodium chloride solution into a 100 ml standard flask and made up to the mark. A portion of the solution was centrifuged at 2300 ***g*** for 30 min at 0°C; 10 ml of the supernatant was pipetted into a micro-Kjeldhal flask and digested for 2 hr with 2·5 ml of 98% H_2SO_4 and about 1 g of a mixture of 1 part of HgO, A.R., and 20 parts of K_2SO_4, A.R. Nitrogen was estimated using the standard micro-Kjeldhal technique (Markham distillation apparatus).

The non-protein nitrogen content of the flesh was estimated in quadruplicate by macerating 1–2 g of flesh (from the same region of the fillet as the soluble protein sample) in 10% trichloracetic acid solution at 2°C for 15 sec with an Ultra Turrax homogenizer. The extract was filtered through a Whatman No. 1 paper and 25 ml of the filtrate were analysed for nitrogen by the micro-Kjeldhal method.

The total nitrogen content of the flesh was estimated in quadruplicate by digesting 1–2 g of flesh with 25 ml of 98% H_2SO_4 and 12·5 g of a mixture containing 1 part of HgO, A.R., and 20 parts of K_2SO_4, A.R., for 0·5–1 hr. Nitrogen was determined by the standard micro-Kjeldhal method.

The soluble protein nitrogen results were expressed as a percentage of total protein nitrogen by using the formula:

$$\% \text{ Extractable protein nitrogen} = \frac{\text{Extractable N} - \text{Non-protein N}}{\text{Total N} - \text{Non-protein N}} \times 100$$

pH determinations

The pH of raw fish was that of a 1:1 water fish homogenate at 20°C. All measurements were carried out with a glass electrode. Since pH varies down the length of fish the samples for pH measurements were taken adjacent to those used for cooking.

Taste panel

The fish samples (almost 200 g each) were cooked from the frozen state by steaming for 30–45 min in a closed, but not sealed, casserole heated on a steam bath. A trained panel of ten people scored the fish for flavour and texture.

Texture was assessed by a system developed in this laboratory in which an attempt has been made to quantify some of the textural characteristics of cooked fish. A two-dimensional score sheet is used in which the word pairs 'tough–soft' and 'wet–dry' have been used as the axes (Fig. 1). Each member of the panel decides which square

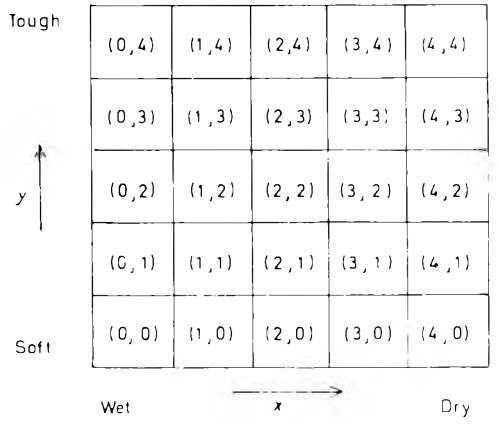

FIG. 1. Texture scoring sheet, showing co-ordinates for each square.

most accurately describes the 'tough–soft', 'wet–dry' characteristics of each fish. By imposing a pair of geometrical axes on the score sheet, each square can be defined by a pair of co-ordinates. Thus the centre square which corresponds to an excellent texture has the co-ordinates (2,2) while the bottom right-hand square which corresponds to a very soft, very dry texture has the co-ordinates (4,0). By using these co-ordinates it is possible to assess the average position of each fish simply by calculating the means ($\bar{x}$ and $\bar{y}$) of the x and y co-ordinates given by the panel (n = number of people on the panel).

Thus $$\bar{y} = \frac{y_1 + y_2 + \ldots\ldots y_n}{n}$$

$$\bar{x} = \frac{x_1 + x_2 + \ldots\ldots x_n}{n}$$

The advantage of this system is that it is easy to see at once properties which characterize the texture of a fish which would be masked by a simple 0–5 overall texture assessment.

Flavour was scored on an arbitrary 0–5 scale from poor to excellent with 'normal' in the middle of the range.

Results

The change in extractability of the cod muscle proteins in 5% sodium chloride with time of frozen storage of the fillets at −29°C is shown in Fig. 2. This shows a steady

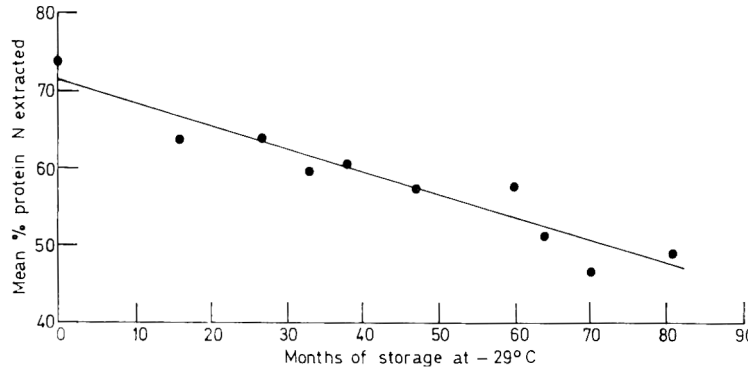

FIG. 2. Relationship between mean % protein N extracted and storage time at −29°C.

decrease in the mean protein extractability over the 82 month storage period from 72% to 45%. The fitted regression line was:

Protein extractability = 72·1834 − (0·3208 × Storage time in months)

During the same period, the toughness ($\bar{y}$) of the cod fillets varied considerably and did not conform to any definite trend (Fig. 3). Indeed fillets stored for 82 months were judged to be considerably less tough than some of the control fillets. When protein solubility was plotted against toughness, as in Fig. 4, there was no correlation between the two.

However, in Fig. 5 toughness has been plotted against raw muscle pH and here it can be seen that a good relationship exists. A regression line has been fitted with the equation:

$$\bar{y} = 20{\cdot}6716 - 2{\cdot}731 \times \text{Muscle pH}$$

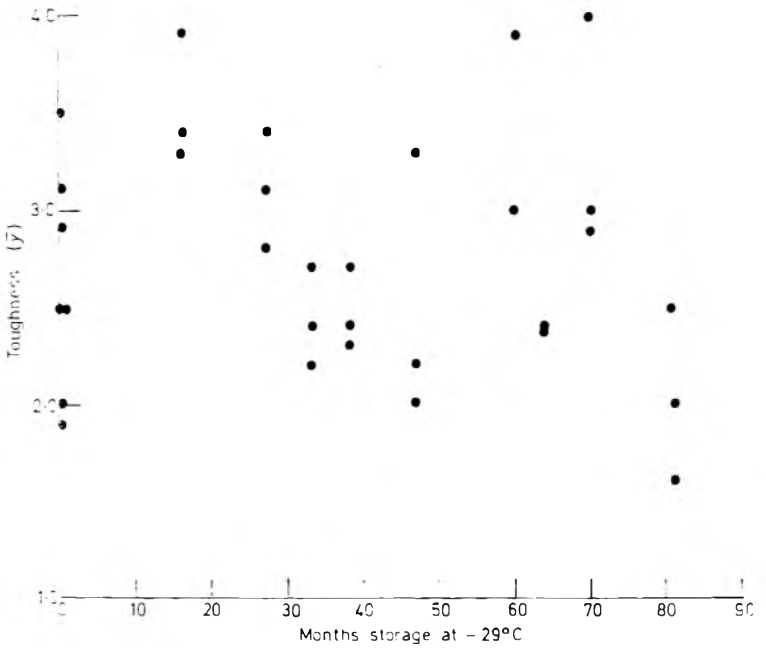

FIG. 3. Relationship between toughness ($\bar{y}$) and time of storage at −29°C.

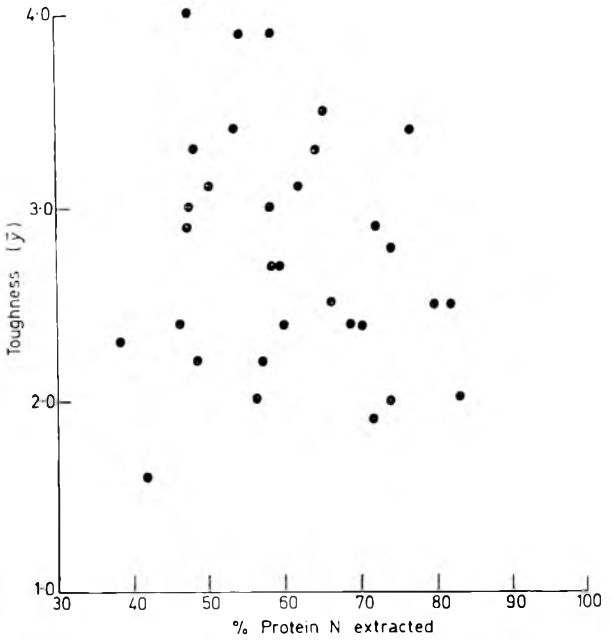

FIG. 4. Relationship between toughness ($\bar{y}$) and protein extractability.

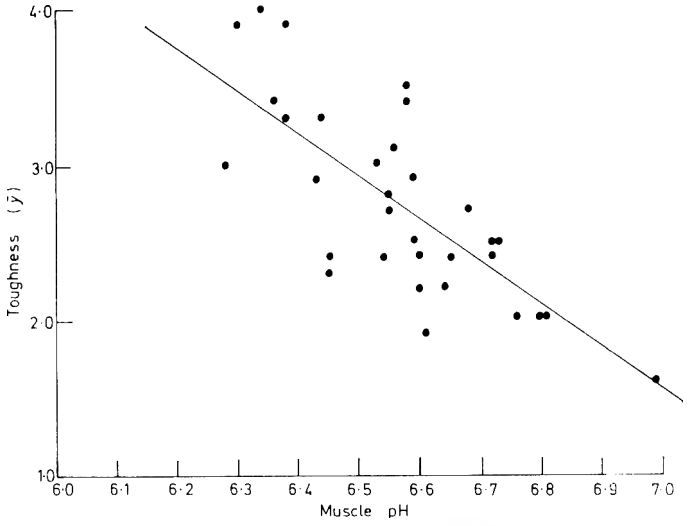

FIG. 5. Relationship between toughness ($\bar{y}$) and muscle pH.

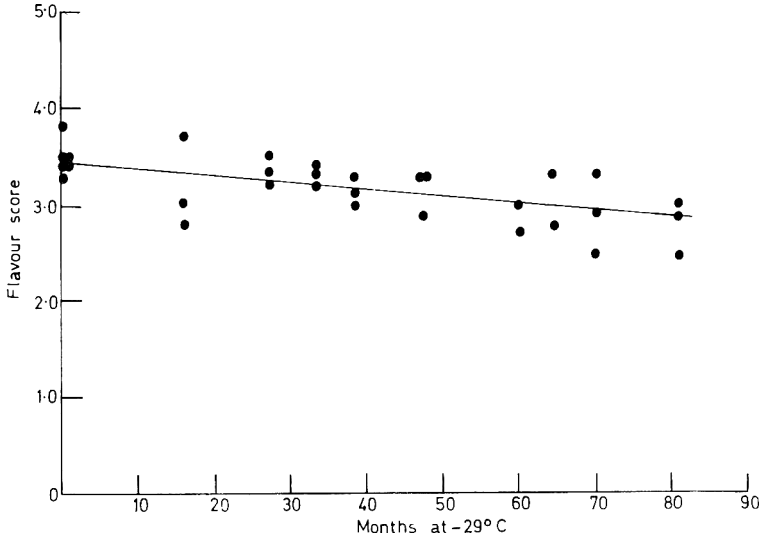

FIG. 6. Relationship between flavour score and time of storage at −29°C.

There was a gradual deterioration in the flavour of the fillets during the 82 month storage as shown in Fig. 6. A regression line was fitted by inspection.

Discussion

The storage of cod fillets for 82 months at −29°C results in a steady decrease in muscle protein solubility from 72% to 45%. This result is in fair agreement with that of Love (1962) and confirms that protein solubility in 5% sodium chloride decreases slowly in cod fillets stored at −29°C.

Contrary to expectations there was no steady increase in organoleptic toughness in the cooked cod concomitant with the steady decrease in protein solubility in the raw cod with time of storage at −29°C. Indeed, cod which had been stored for 82 months were less tough than some of the control fillets which had been frozen but not cold stored. This indicates that in cod stored at −29°C, protein denaturation, as measured by a decrease in solubility, is not the predominant factor in determining the organoleptic toughness of the cooked cod.

We have previously established that the toughness of unfrozen cod is related to its muscle pH. The present results show that the texture of frozen cod stored for up to 82 months at −29°C also depends on pH. For such fish the muscle pH is a much better guide to the toughness of the cooked muscle than the protein solubility figure. The rate and degree of protein denaturation will be higher in cod stored at less cold temperatures than −29°C, and in that case, protein denaturation may be more important than muscle pH in determining the toughness of the cooked muscle. This aspect of the problem is being studied.

The information obtained from this work may help to clear up some of the apparent anomalies in the literature. Love (1956) and Luijpen (1957) both found that in cod stored at −30°C for 1 year, organoleptic toughness increased with time of storage while protein solubility did not alter significantly. These findings were surprising in view of Dyer's (1951) good correlation between increasing toughness and decreasing protein solubility in cod fillets stored at −23°C. The observed toughening in the cod stored at −30°C could be due to the fact that the fillets tested towards the end of the experiment had lower muscle pH than those sampled at the start.

Acknowledgments

We are indebted to Dr R. M. Love for the samples of long-stored cod fillets used in this investigation, to Dr T. R. Kelly for developing the taste panel system and to Mr M. Duckworth for technical assistance.

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Cannery retort operation and procedure to cope with modern can-handling methods*

J. D. FELMINGHAM AND R. J. LEIGH

Summary. It is well known that the heat transfer properties of air or air–steam mixtures are much inferior to those of steam and that, because of this, entrapped pockets of air in a cannery retort may lead to under-processing and spoilage. Consequently there is a need for adequate purging or venting of air from any retort during the processing 'come-up' period.

Following the introduction to a U.K. cannery of a high-speed can handling system, necessitating the use of can divider plates (or layer pads) in the retort crates, this cannery experienced an outbreak of spoilage that was shown, by microbiological and can examination, to be due to under-processing. This led to an investigation of heat distribution in the cannery retorts and of the effect on this of divider plates with varying percentages of void area. This work was extended to other canneries and various types of batch retorts.

Results showed that some venting procedures in long-standing use were not entirely satisfactory and that certain modifications were necessary, even to normally adequate procedures, when certain high-speed can handling techniques were introduced.

Graphs are presented to show quite dramatic differences in heat distribution throughout retorts when venting is inadequate.

Recommendations are made for correct venting procedure and the modification necessary when divider plates are used in conjunction with high-speed can handling systems; also regarding the area that should be left void in these divider plates. Attention is drawn to correct maintenance of retorts and retort instruments, recording equipment, etc.

The effect of entrapped air during retorting on external can rusting is also discussed.

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Introduction

In conventional retorting techniques, for low acid products, cans are processed in saturated steam at pressures up to 21 lb/in^2 (1·48 kg/cm^2) or even higher. Saturated steam is a most effective medium of heat transfer for, as it condenses on the cans, it gives up its latent heat and the condensed water droplets drain away leaving the can exposed again to the full heating effect of the steam. Air, on the other hand, is a very poor thermal conductor and, being a non-condensible gas, may form an insulating barrier around the can. In addition it may mix with the steam to form air–steam mixtures or remain as pockets of stagnant air.

Gillespy (1961) reports that the effect of an air film on a can might be equivalent to increasing the dimensions of the can by $\frac{1}{4}$ in. all round.

The effect of an air–steam mixture is to lower the temperature at constant pressure or to increase the pressure at constant temperature and, as one would not expect an even distribution of air within the steam, temperature gradients will exist between one point and another within the retort. Pockets of entrapped air will give rise to even wider temperature gradients which will only gradually reduce and may in fact still exist at the end of the heating cycle. These temperature gradients will at the best give rise to product inconsistency and external can rusting and at the worst give rise to localized spoilage or even present a potential public health hazard.

It is clear then that air initially present in the retort must be effectively removed, prior to the commencement of process timing, by a procedure known as venting.

Some canners use water as the heating medium thus obviating the necessity for venting, but in the U.K. this practice is not widespread for low acid, pressure processed products.

As the density of air is considerably greater than that of steam it would in theory be logical to inject steam into the top of the retort whilst venting out the air at the bottom. In practice, however, the cans present such an obstruction to the flow that air can only be removed by a vigorous blasting action through the retort and, so long as the air is vented out from the opposite end to the steam entry point, it would appear to be immaterial whether the flow is from top to bottom or vice versa. Bottom steam entry is almost universally adopted.

In the U.K. high-speed can handling systems have been introduced. In some cases, in order to operate the semi-automatic equipment for loading and discharging retort crates, perforated divider plates are inserted between each layer of vertically stacked cans.

Following the introduction of such systems to U.K. canneries, there were some outbreaks of spoilage that were shown, by microbiological and can examination, to be due to under-processing. This led to the investigation described here of the heat distribution in these canneries' retorts, particularly during venting, with and without

divider plates in use and with plates having varying perforations, or amount of void area.

This work was extended to other canneries and involved different types of retorts. Basically it has been an investigation of venting procedures and the effect of divider plates.

Equipment and procedure

Temperature changes at various points within the retort were recorded by the use of needle type thermocouples located in the free-spaces between the cans.

Initially work was carried out using a six-point self-recording potentiometric instrument together with chromel–constantan thermocouples. However, the lack of portability was a distinct disadvantage and subsequent work was therefore carried out using a portable electric precision thermometer together with copper–constantan thermocouples introduced into the retort via a standard fitting supplied by the manufacturer.

It was established that the points of maximum temperature lag were most likely to be located in a region around the centre of each crate; the thermocouples were therefore sited at these points. Readings were taken at $\frac{1}{2}$ or 1 min intervals from the moment the steam was turned on and continued until all readings corresponded to the retort operating temperature.

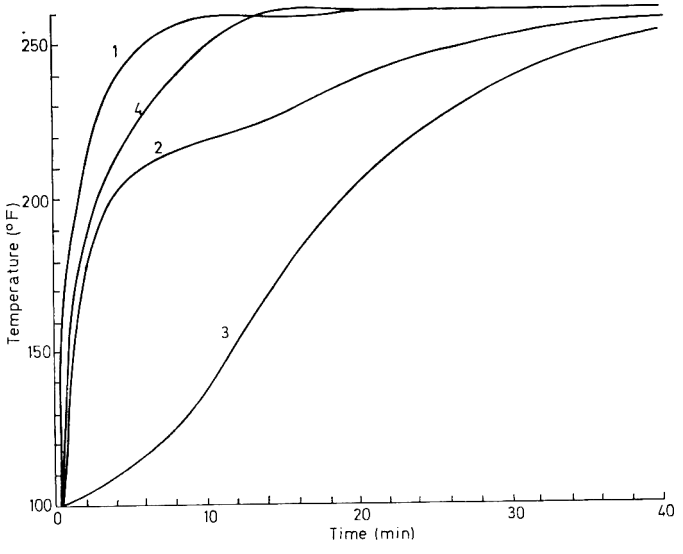

FIG. 1. Venting of a four-crate vertical retort with thermocouples located in the centre of each crate. An unauthorized procedure (poor practice). 1, Top; 2, second from top; 3, third from top; 4, bottom.

Results

(1) *The effect of venting practice on temperature distribution in cannery 'A'*

The investigation in this cannery followed an extensive spoilage outbreak. Bacteriological tests and can examinations had indicated gross under-processing as the likely cause.

The spoiled cans had been processed in four-crate vertical retorts. These retorts were of conventional design but divider plates perforated with $\frac{7}{8}$ in. diameter holes on $1\frac{3}{8}$ in. centres were used between each layer of cans.

Fig. 1 shows temperature distribution during venting by a procedure which, although contrary to operating instructions, had become prevalent in this cannery, as personal observation and discussions with retort operatives established.

Procedure:

0 min: Steam on. Top vent/overflow closed. Bottom drain open. Bleed cock open.

3 min: Bottom drain closed.

8 min: Up to retort operating temperature on mercury-in-glass thermometer.

It is important to note that the top vent/overflow remained closed throughout, thus preventing the through-flow necessary to the venting operation.

Fig. 2 shows a result of the procedure authorized in the same cannery.

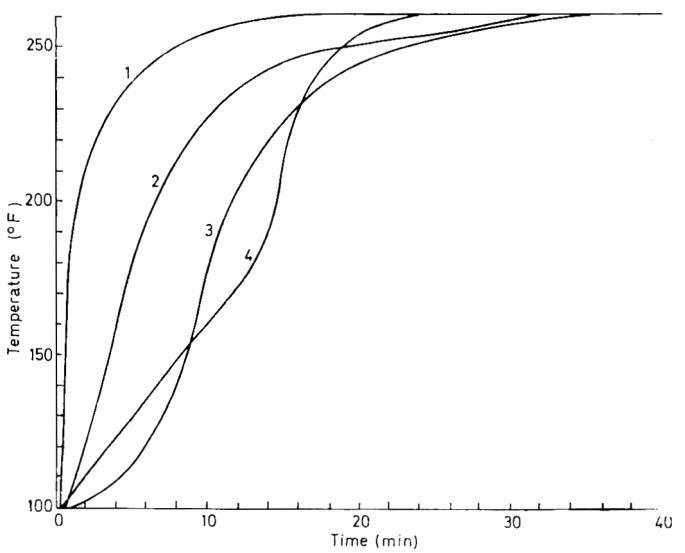

FIG. 2. Venting of a four-crate vertical retort with thermocouples located in the centre of each crate. An authorized procedure but still poor. 1, Top; 2, second from top; 3, third from top; 4, bottom.

Procedure:

0 min: Top vent and bottom drain open. Bleed-cock open. Steam on.

$2\frac{1}{2}$ min: Top vent and bottom drain closed.

$8\frac{1}{2}$ min: Retort operating temperature indicated on mercury-in-glass thermometer.

Although the top vent has now been opened at the commencement of venting, the graph clearly shows that it has been closed too early, with resultant temperature lags.

It will be seen that, although there are minor differences in the recorded come-up times between these two procedures, in both cases there is an extremely serious lag before all the thermocouple readings reach those recorded on the retort control equipment. This lag is particularly marked with the thermocouple placed in the centre of crate 3 which is the third from the top in the retort.

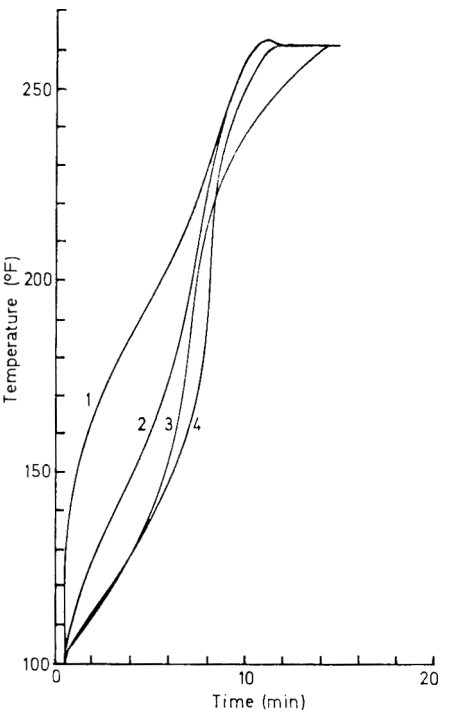

FIG. 3. Venting of a four-crate vertical retort with thermocouples located in the centre of each crate. Recommended procedure. 1, Top; 2, second from top; 3, third from top; 4, bottom.

In Fig. 3 a procedure to comply with normally recommended practice has been put into operation.

Procedure:

0 min: Top and bottom drain open. Bleed cock open. Steam on.
3 min: Bottom drain closed.
8 min: Top drain closed.
10 min: Retort operating temperature indicated on mercury-in-glass thermometer.

Comparing Fig. 3 with Figs. 1 and 2 it is quite apparent that a marked improvement has been obtained, so that the retort control equipment is giving readings at the end of the come-up period that truly represent the condition throughout the retort.

It is equally clear that the previous practice resulted in a substantial proportion of cans not receiving the heat process that control equipment records would have indicated.

(2) *Comparison of* 1½ *in. and* 2 *in. steam entry pipes in cannery 'B'*

Cannery 'B' was equipped with four-crate horizontal retorts of conventional design. In this cannery the established venting procedure was found to be satisfactory but some retorts were fitted with 1½ in. diameter steam supply pipe whereas others, fed by the same header pipe, were fed by 2 in. diameter pipe. Fig. 4 shows how this factor has influenced the speed of venting.

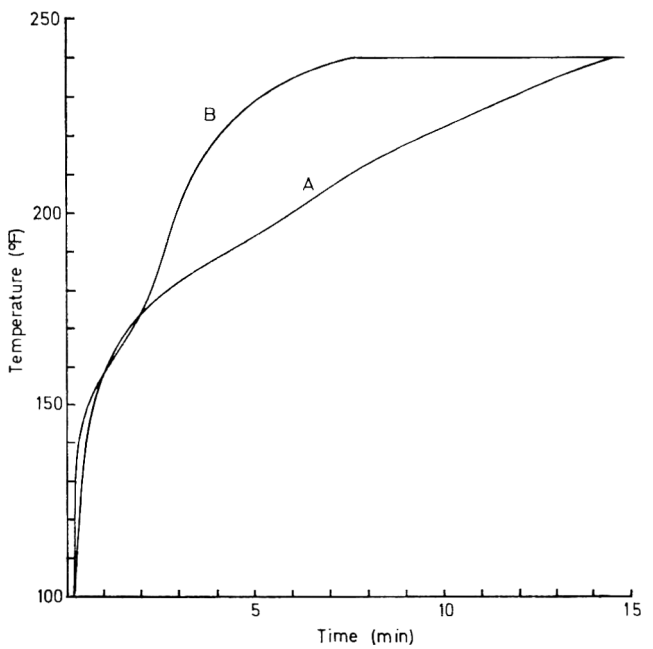

FIG. 4. Effect of steam entry pipe diameter. Thermocouples in centre of slowest heating crate in four-crate horizontal retort. A, Come-up curve for 1½ in. diameter; B, come-up curve for 2 in. diameter.

(3) *The effect on come-up time of poor and obstructed steam supply*

Cannery 'C' was a large cannery using three-crate horizontal retorts. The work described here was carried out in modern three-crate horizontal retorts of round section which had been quite recently installed.

Procedure:

0 min: Top and bottom drains open. Bleed cock open. Steam on.
29 min: Bottom drain closed.
37 min: Top drain closed.
52 min: Retort operating temperature indicated on mercury-in-glass thermometer.

Although the drains have been closed at 212 and 200°F respectively the rules of venting were unquestioningly applied when it was apparent that these inordinately long times were indicative of something being seriously amiss (see Fig. 5).

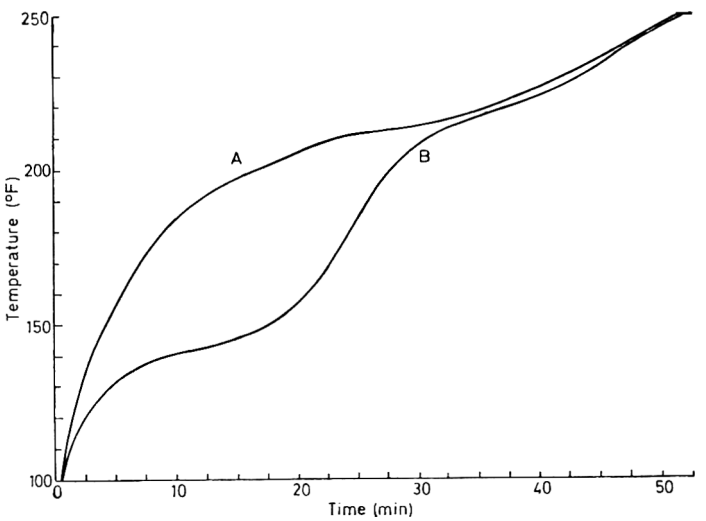

FIG. 5. Excessively long come-up times due to restricted steam entry into a horizontal retort. The thermocouples located: (A) in free space in the top of the retort, and (B) in the centre of the middle crate.

Subsequent investigation revealed almost complete blockage of the holes in the steam spreader and complete blockage of the bleed cock. Since cooling water shared a common entry with the steam, it was hardly surprising to find that 60 min was necessary just to fill the retort with water. In addition the door gasket was leaking seriously, wasting appreciable amounts of the already limited supplies of steam or water entering the retort. Not surprisingly, gross differences in process lethality were found between

cans situated at the top and bottom of the retort. This is discussed in more detail later (see Fig. 9).

The cooling water available at cannery 'C' was of a particularly corrosive nature, which should have lent even greater emphasis to the need for effective preventative maintenance. This cannery has a history of external corrosion troubles.

As might be expected this cannery was also suffering from a serious shortage of retort capacity.

(4) *The effect of divider plates*

Cannery 'D' was operating conventional four-crate vertical retorts. Our investigation followed a spoilage outbreak which occurred soon after the changeover from scramble-packing to a divider plate system. Fig. 6 shows the temperature distribution

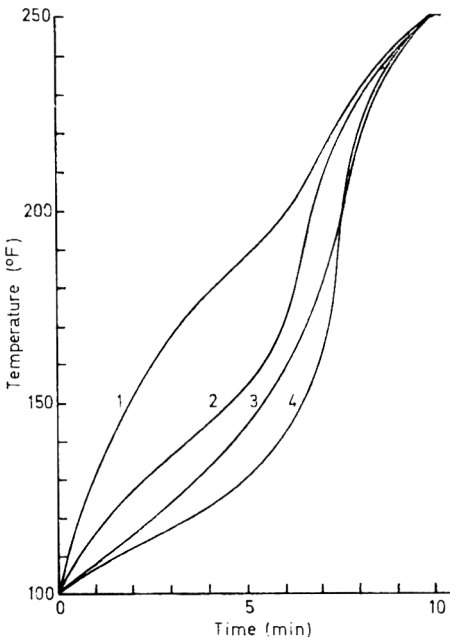

FIG. 6. Temperature distribution during venting with scramble packed crates. 1, Top; 2, second from top; 3, third from top; 4, bottom. Thermocouples located in the centre of the crates.

during venting of the scramble-packed crates and Fig. 7 shows what happened when a venting procedure, on the same time-controlled basis, was applied after the introduction of the divider plate system. The obstruction to air removal caused by these dividers is clearly demonstrated.

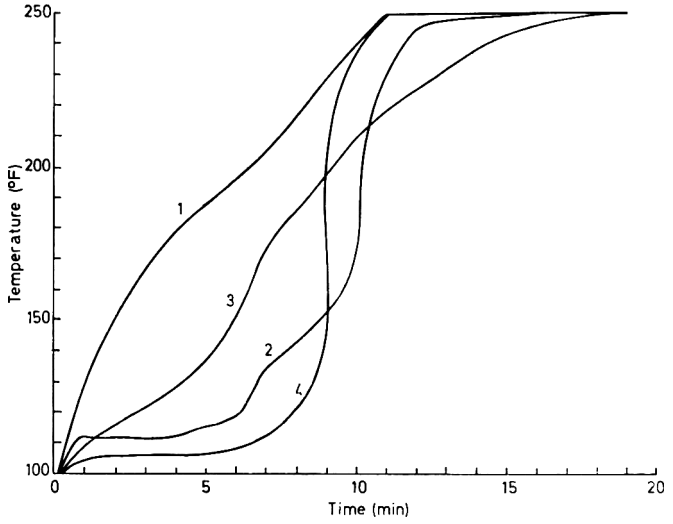

FIG. 7. The same retort as in Fig. 6 with the divider plate system. Thermocouples located in the centre of the crates. 1, Top; 2, second from top; 3, third from top; 4, bottom.

Procedure

0 min: Top and bottom drains open. Bleed-cock open. Steam on.
6 min: Close bottom drain.
8½ min: Close top drain.

(5) *The void area in divider plates*

Fig. 8 shows how the void area of the divider plates affects the venting times. The divider plates as originally supplied were perforated with 1 in. diameter holes on 2 in. centres giving a void area of 19·6%. It soon became apparent that excessive restriction was taking place and further sets of divider plates, perforated with 1 in. diameter holes on $\sqrt{2}$ in. centres, were supplied, doubling the void area to 39·2%. Fig. 8 shows the benefit derived from this modification.

(6) *The effect on process lethality of unduly extended venting times due to inadequate steam supply*

When steam supply is inadequate large temperature gradients will exist during the extended venting time necessary to sweep out the air entrapped between the cans. During this time there will be a significant heat input into those cans exposed to the steam whilst those in the protected inner areas will be exposed to the heating medium hardly at all.

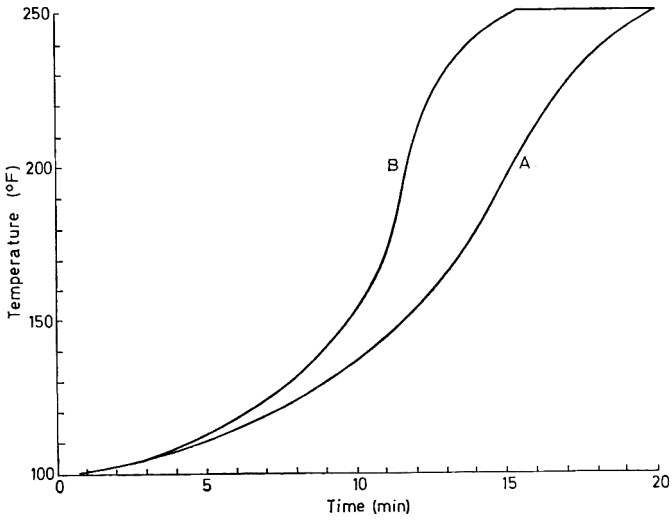

FIG. 8. The effect of void area in divider plates. Thermocouples at the slowest heating point in a four-crate vertical retort. Divider plates with 1 in. diameter holes on 2 in. centres (A) and 1 in. holes on $\sqrt{2}$ in. centres (B).

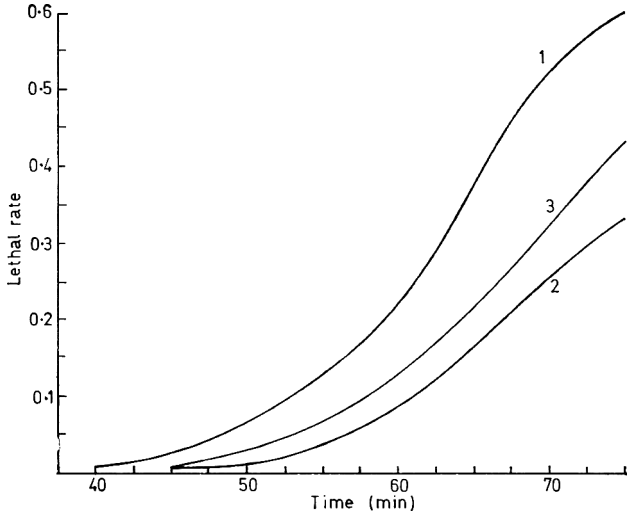

FIG. 9. The effect on process lethality of prolonged venting times. Thermocouples located in cans: (1) near the top of the retort, (2) just above the centre, and (3) near the bottom, during the entire heating cycle.

Fig. 9 shows lethality curves during the heating cycle for three differently situated cans during a 19-min venting period. Can 1 was situated near the top of the retort, can 2 just above the centre and can 3 near the bottom. Divider plates were in use and these were perforated with 1 in. diameter holes on 2 in. centres.

It will be seen that there are marked differences in the curves and, in fact, calculation of the F_0 values reveals that the lethal value in can 2 is less than half that in can 1.

Discussion

Inadequate venting causes temperature lag in varying degree throughout retorts and may result in under-processing spoilage. This much is well established and confirmed by the work described which also indicates that, in three- or four-crate vertical retorts, the centre of the middle crate or of the third crate from the top respectively, are likely to be the points of greatest temperature lag.

What was surprising, however, was the prevalence of poor venting practice. It is really only the introduction of new can-handling techniques that has brought this fact to light. That under-processing spoilage has not been more widespread is undoubtedly due to safety margins on the process times stipulated and it is quite certain that many processes could be reduced by improving venting efficiency. This could result in economies and more uniform product quality.

By inference from this work, and from past experience of one of the authors, it is also apparent that much external can corrosion has been due to poor practice at the beginning and end of the retort process.

Many canners who are aware of the dangers, and who wish to obtain efficient operation, are hampered by various factors. Foremost among these is the frequent inadequacy of steam input into their retorts. The National Canners' Association (1956) recommend a steam pressure in the cookroom main of not less than 90 lb/in^2 at all times during operations. The authors have observed pressures as low as 15 lb/in^2 with a consequent long and laborious come-up and the inevitable temptation for operatives to take dangerous short cuts to reach retort temperatures.

A strict interpretation of the U.K. Factories Act does not permit steam to be fed into retorts at pressures in excess of the safe working pressures, which means in practice that pressures of 15–35 lb/in^2 (approximately 1–2·5 kg/cm^2) are common.

It has been argued that, under these conditions, the dual criteria of time and temperature are not enforceable, because the prescribed temperature may never be reached without partially closing the main vent, a dangerous practice. Hence it may be advocated that venting should be carried out on a time-only basis.

This procedure cannot be condoned because it is so obviously inconsistent in effect, being at the mercy of steam pressure fluctuations and operator errors. It is believed

that the dual time and temperature criteria should be compulsory and if this leads to excessively long venting times the causes should be sought out and remedied.

Apart from the feed pressure, steam supply may be restricted by small bore inlet pipes and it may be misdirected when it is in the retort. In some cases correct spreaders have been found to be inoperative because of blockage due to dirt and corrosion.

Frequently one meets the twin arguments against adequate venting, of excessive steam consumption and 'filling the place with steam'. Although steam usage is highest during the venting period, any increase can usually be offset by an overall reduction to process time with an efficient operation and, in any case, the few pennies involved are as nothing beside the loss of goods and custom from even one spoilage outbreak. The extraction of steam from the atmosphere is clearly a question of layout and ventilation but here again the argument is usually that external can corrosion is increased, whereas far more damage is actually occurring in the inadequately vented retorts.

Automatic venting

Manual venting suffers from the inherent possibility of operator error and varying degrees of automatic control have been devised, ranging from fairly simple, thermostatic devices to complete automatic control of the retorting cycle from start to finish.

It is not within the scope of this paper to discuss the relative merits of these systems, and manufacturers' advice should be sought to ensure that any system installed is able to meet all the requirements of good venting practice. Some of the simpler thermostatic devices are unsuitable for full-sized retorts and instances have been encountered where their installation has led to a poor degree of air removal. Possibly this may have been attributable to the expense of obtaining the correct equipment as specified by the manufacturers. If this is so then it is false economy because either the process time or the spoilage hazard will have been increased.

Although reference is made in the title to the effect of high-speed can handling, only general venting practice has so far been discussed. For this no apology is made because of the fairly wide-spread ignorance of basic principles.

Assuming that good practice is established for conventional retorting, it has been shown that modification is necessary where high-speed can handling, employing divider plates, is introduced. These dividers clearly are impediments to steam circulation that necessitate two main points of attention. First the number and size of perforations, or amount of void area, need to be carefully controlled, and second the actual amount of venting must be slightly increased. Recommendations are given to cover both of these factors.

Recommendations

It must be emphasized that these recommendations are necessarily couched in general

terms and that although, failing any evidence to the contrary, they should be satisfactory for most circumstances, there is no substitute for cannery performance data.

(1) The first recommendation, therefore, is that the conditions pertaining should be established by experiments, such as those described, carried out under the most adverse cannery operating conditions (using the smallest can size, a retort at the end of the steam line, lowest steam pressure, etc.). An operational procedure should then be laid down accordingly.

(2) It is recommended that, unless a fully tested automatic system is employed, venting should be based on the dual criteria of temperature and time and that both conditions must be satisfied.

The following venting schedule, for standard vertical retorts with $1\frac{1}{2}$ in. diameter top vents, is given as a guide to establishing satisfactory procedures:

(i) Open fully all orifices leading to the atmosphere—top vent and bottom drain, petcocks or bleeds in lid and instrument pockets.

(ii) Turn steam on.

(iii) When retort thermometer reaches 212°F close bottom drain, leaving 'crack' to allow condensate to drain away throughout processing.

(iv) After not less than *a further* 2 min *and* when the thermometer reaches 220°F, *or*, if divider plates are in use, not less than 4 min and 220°F, close top vent.

(v) Petcocks or bleeds remain open throughout processing although, if they are wide bore, they may be partially closed to leave the equivalent of $\frac{1}{16}$ in. bleeds.

(3) Steam supply must be adequate to provide efficient venting within the schedules quoted. This means attention to entry pressure, diameter of inlet pipes and vents (which must be at least one pipe size larger than inlets), correct valves on all pipes, efficiency of steam spreaders, number and diameter of holes in these, maintenance of all equipment, particularly with respect to prompt removal of corrosion debris that could cause blockage. Leaking valves are a danger since the authors have encountered retorts putting air back in nearly as fast as the venting procedure has removed it.

(4) Divider plates should be perforated to a minimum of 1 in. diameter holes on $1\frac{3}{4}$ in. centres or equivalent void area, and venting increased accordingly.

(5) All vents, whether individual or manifold, must discharge to atmosphere and not into a drain or under water as these conditions could allow a back pressure to build up.

(6) All retorts should be fitted with a mercury-in-glass thermometer as a primary standard. This should be constantly checked for accuracy by comparing with other instruments and against a standard instrument at least once a year. As well as this thermometer, retorts should normally be fitted with a pressure gauge and temperature

recording equipment, both of which should correspond to the mercury-in-glass thermometer.

(7) Safety valves will be fitted by retort manufacturers. They should be tested periodically to ensure that they will blow off at the set pressure.

(8) Cooling water should enter rapidly to ensure uniform cooling and this will necessitate flooding and the use of top sprays.

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Note on the difference between smooth and wrinkled peas after processing

S. B. THUNG AND L. GERSONS

Summary. Due to the fact that smooth-seeded and wrinkle-seeded pea varieties are both on the market, canned as well as quick-frozen, it is of practical importance to determine the difference between these two types. A simple microscopic examination has been worked out, in which the distinction between smooth and wrinkled peas can be easily established. After treatment with ethanol the shape of the starch granules is fixed, which differs in smooth and wrinkled varieties. The method cannot be used for intermediate varieties, raised by crossing between the two types.

Introduction

In most countries peas are one of the main products of the processing industry. For many years the most important method of preservation of peas has been by canning, and more recently by packing in glass, but increasing amounts are now being quick-frozen.

Peas may be sub-divided into two types, namely those with smooth and those with wrinkled seeds. When dried, the former are round and, as a rule, light green in colour and when cooked have a pronounced pea flavour. The latter type, when dried, are wrinkled and have often a bright or deep green colour, a fairly hard skin, and very tender cotyledons. When cooked they have a sweet taste and a less pronounced pea flavour. In Europe most canned fresh peas belong to the smooth-seeded type, whereas the quick-freezing industry uses mainly the wrinkle-seeded type. In some countries, however, wrinkle-seeded peas are the main type used for canning. Smooth-seeded varieties are not commonly used for quick-freezing.

Curiously, the consumers' preference for either or the two types of peas varies between countries. In Holland, although the output of wrinkle-seeded peas has increased to the point where it forms 20% of the total production, virtually all wrinkle-seeded peas which are quick-frozen are for export, for the Dutch, like the Belgians and the French, prefer smooth-seeded peas. In Germany, Sweden, Britain and the U.S.A., on the other

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hand, the greatest demand is for wrinkle-seeded peas both for canning and for freezing.

In spite of the pronounced preference on the consumers' side for one particular type of pea, distinguishing between the two types in an objective manner used to be merely a matter of academic interest, as the two types were very seldom found as a canned product in the same country. However, the vegetable processing industries of the E.E.C. countries conferred with each other and agreed to put the two types on the market, though under different label declarations. As a result smooth-seeded peas are now sold under the name of 'continental peas', wrinkle-seeded peas being labelled 'garden peas'. The quick-freezing industries in these countries intend to solve the problem in the same way.

Since both continental and garden peas may now be marketed in the same country, it is of great importance that a simple method be available by which the difference between the two types may be established. So far organoleptic methods have proved unsatisfactory. When peas have undergone heat-treatment (blanching, sterilization) there is no longer any difference between smooth and wrinkled skins. The skins of both types become smooth. As some wrinkle-seeded varieties can have a light green colour, this factor cannot be a distinguishing characteristic. Although, as a rule, the skins of wrinkle-seeded varieties are firmer than those of smooth-seeded ones and their taste is sweeter, these characteristics are not sufficiently reliable to be used in identifying the two types.

Several attempts were made in the past to find a solution for the problem mentioned above. When smooth and wrinkle-seeded peas are fresh or have been dried, they can easily be told apart by microscopic examination, as they are characterized by the shape of their starch granules. The starch granules of smooth-seeded peas are oval and simple, whereas those of wrinkle-seeded peas are round and have radical crevices, giving them the appearance of composite granules. However, when peas are subjected to heat treatment, as happens when they are sterilized or even sufficiently blanched, complete gelatinization of the starch takes place and the differences mentioned above disappear. The starch of both types shows up as an amorphous mass under a microscope.

Schneider (1951) managed to show differences between sterilized peas belonging to the two types by freezing them one by one in liquid air and then pulverizing them in a mortar. In this way he isolated individual starch granules which could then be identified. Still, this method is not a very satisfactory one for practical purposes. It was felt, moreover, that a simpler method, not involving the use of liquid air, was to be preferred.

Experimental

The method described below gave satisfactory results. A small quantity of cotyledon substance is pulverized with a spatula in 96% ethanol on a microscope slide, a drop of

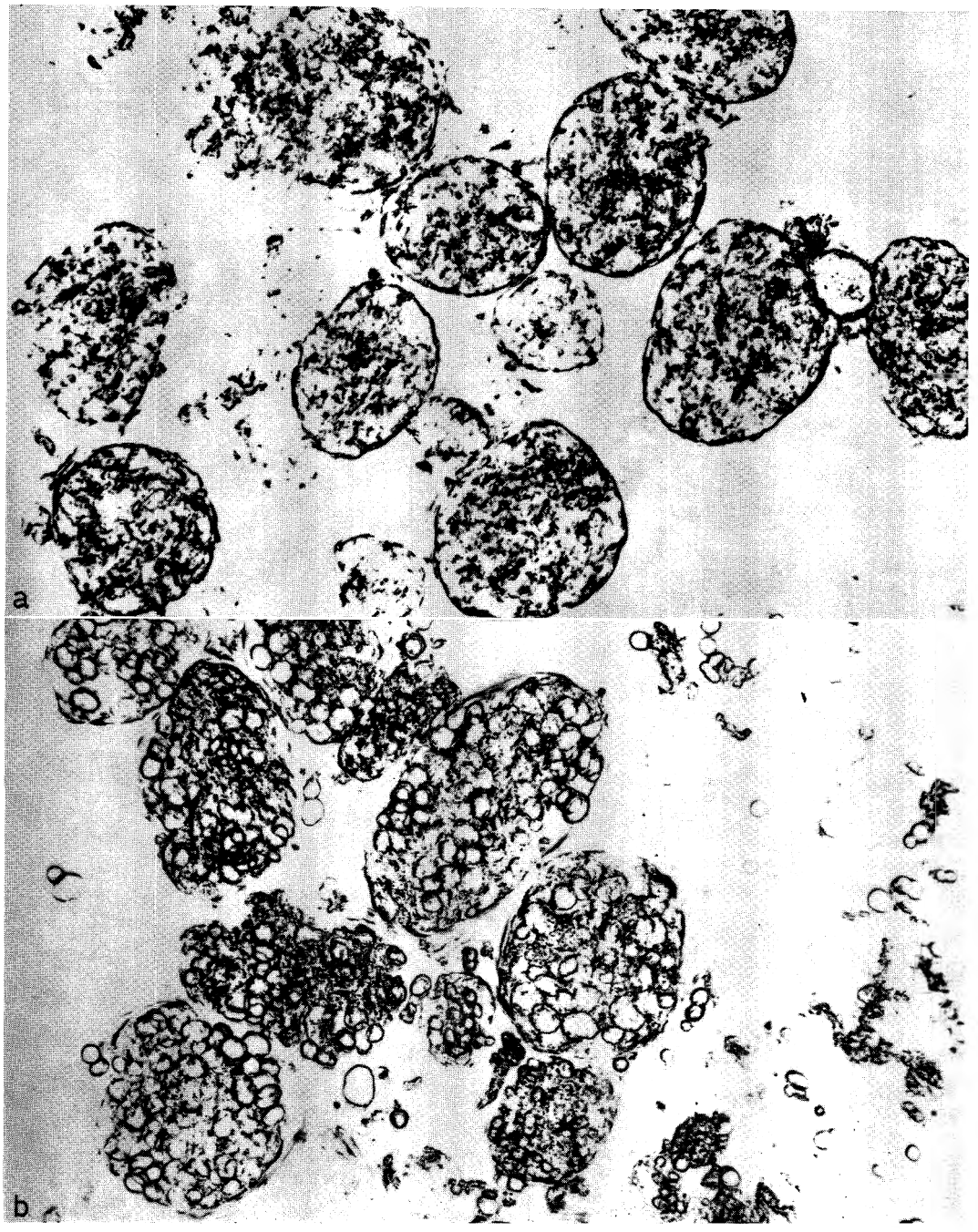

PLATE I. (a) Cells of cotyledons with starch grains of smooth-seeded variety. (b) Cells of cotyledons with starch grains of wrinkle-seeded variety.

(*Facing p.* 360)

glycerol is added and the whole is covered with a cover glass. Examination of the cells under a microscope shows them to be filled with swollen starch granules. When the granules belong to a smooth-seeded pea they will show up as an amorphous mass (Plate 1a), but when they belong to a wrinkle-seeded pea they can be seen as more or less sharply defined spherical granules (Plate 1b), which is their most striking distinguishing characteristic. So when sharply defined particles show up microscopically, the cells belong to a wrinkle-seeded pea, whereas when no such particles are to be distinguished, the pea concerned must be of the smooth-seeded type.

The difference described above can be seen under direct light, but can be established much more clearly when phase contrast or dark-field illumination is used.

The method was applied in the investigation of a large number of samples of canned and quick-frozen smooth and wrinkle-seeded peas from this country and abroad. The type of pea was correctly established in 100% of the tests.

The difference between the two types of peas described above can also be observed in quick-frozen peas that have been blanched to such an extent that their starch granules are completely gelatinized. When blanching has not gone quite so far, the starch granules of wrinkle-seeded peas look very much like they do when the peas are fresh.

The different shapes of the starch granules can only be observed in true smooth-seeded and wrinkle-seeded peas. Varieties obtained by crossing the two types, such as Cennia and others, which when dried have the outward appearance of wrinkle-seeded peas, show the starch granule form of smooth-seeded peas when examined microscopically.

Reference

SCHNEIDER, A. (1951) *Züchter*, **21**, 275.

J. Fd Technol. (1966) **1**, 363–365.

Note on a modified device for headspace evaluation of cans

R. HOENIG, D. REZNIK AND H. C. MANNHEIM

A modification of the 'Beckman Headspace Sampler' (designed by Beckman Instruments Inc., Fullerton, California) has been developed in our laboratories to determine accurately the volume of the headspace, its oxygen content and the vacuum in cans. The modified headspace sampler is used in our laboratories for corrosion and deaeration research.

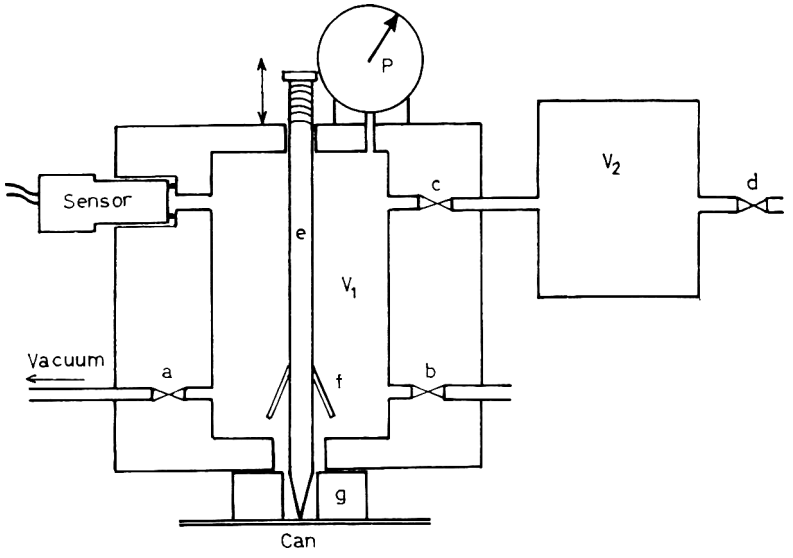

FIG. 1. Schematic description of the modified headspace-sampler.

High vacuum has to be established in the headspace sampler before puncturing the can for headspace determination. The volume of the cell in the original sampler is about 3 ml, consequently it is usually filled with liquid from the can after puncturing. The oxygen sensor is wetted and measurements are impossible. In addition, in cases where the headspace is small, the volume of the cell, which is usually neglected, leads to erroneous results.

In view of these difficulties a modified cell (Figs. 1 and 2) was constructed in our

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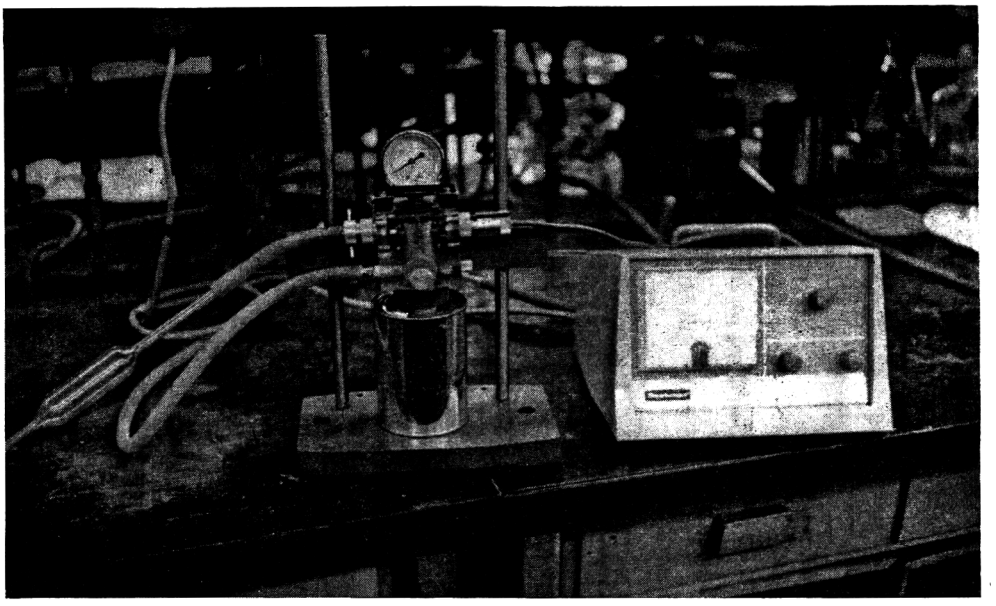

FIG. 2. The modified headspace analyser.

laboratories. The volume of this cell (V_1) is about 30 ml. A cone (f) is fastened to the puncturing bar (e) to avoid splashing of the liquid on the sensor. Liquid might, therefore, enter the cell, but it does not reach the sensor. An additional cell of 55 ml (V_2) is connected, so as to enable calculation of the headspace parameters.

Mode of operation

In order to evaluate the vacuum (or pressure) in the can (P_c), the oxygen partial pressure (q_c) and the volume of the headspace (V_c) the following operations take place.

The sensor is connected to the cell and the apparatus is fastened to the end of the can. Valves (b) and (c) are closed and the cell is connected to the vacuum pump. When vacuum is achieved, valve (a) is closed and the pressure in the cell (P_1) is read by the manometer (P). The partial pressure of oxygen in the cell (q_1) is measured by the oxygen analyser. The can is punctured now and after waiting a few minutes, allowing the gases of the headspace and the cell to equilibrate, the new total pressure (P') and partial pressure of oxygen (q') are determined. The next stage is the connection of the volume (V_2) (initial total pressure P_2 and oxygen partial pressure q_2) by opening valve (c), and determination of the total pressure established (P'') and the partial pressure of oxygen (q'').

Calculation of headspace parameters

The equation on which the calculations are based is PV = Const. at constant temperature.

After puncturing the can:

$$P_c V_c + P_1 V_1 = P' (V_c + V_1) \tag{1}$$

After addition of volume (V_2):

$$P' (V_c + V_1) + P_2 V_2 = P'' (V_c + V_1 + V_2) \tag{2}$$

Similar equations may be written for the oxygen partial pressure.

The final equations for calculation of:

Volume of the headspace

$$V_c = \frac{P_2 V_2 + P' V_1 - P'' (V_1 + V_2)}{P'' - P'} = \frac{q_2 V_2 + q' V_1 - q'' (V_1 + V_2)}{q'' - q'} \tag{3}$$

Pressure in the can

$$P_c = \frac{P' (V_1 + V_c) - P_1 V_1}{V_c} \tag{4}$$

and oxygen partial pressure in the can

$$q_c = \frac{q' (V_1 + V_c) - q_1 V_1}{V_c} \tag{5}$$

The ratio $q_c/P_c \times 100$ expresses the percentage of oxygen concentration in the headspace. Measurements and results from three cans containing orange juice after processing and cooling are given in Table 1.

TABLE 1. Data for headspace parameters of three cans containing orange juice

Total pressures (mmHg)					Vc	Oxygen partial pressure (mmHg)					Vc	O_2
P_1	P_2	P'	P''	P_c	(ml)	q_1	q_2	q'	q''	q_c	(ml)	(%)
50	760	305	555	382	100	7	156	52	110	66	98	17·3
40	760	90	460	190	20	7·5	156	20	90	37	22	19·5
50	760	80	570	109	31·5	7·5	156	10·7	117	14	29·5	12·9

The results of V_c may be checked by weighing the can. Results show good consistency between values measured by these two methods.

The oxygen analyser might be connected to a recorder and a continuous reading of oxygen content in the headspace be obtained. This arrangement was used in our laboratories for investigations on oxygen disappearance in canned foodstuff.

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Bread as a source of protein. By T. Moran and J. Pace.

Isolation of structural elements of food products by preparative ultracentrifugation: ice cream and ice-cream mix. By J. J. Wren and B. K. Bullimore.

The effect of tylosin lactate on the shelf life of semipreserved herring fillets ('titbits'). By Inger Erichsen.

Shortening as a factor in meat ageing. By C. L. Davey, Helen Kuttel and K. V. Gilbert.

Volatile carbonyls in cooked chicken. By C. H. Lea and A. Hobson-Frohock.

New synthetic antioxidants based on caffeic acid. By B. H. Thewlis.

Measurement of the flow properties of powders. By G. W. White, A. V. Bell and G. K. Berry.

Low temperature and the storage of carrots. By W. Hugh Smith.

Structural changes in meat during ageing. By C. L. Davey and K. V. Gilbert.

Studies on mechanical factors affecting dough development. By P. W. Heaps, T. Webb, P. W. Russell Eggitt and J. B. M. Coppock.

INSTITUTE OF FOOD SCIENCE AND TECHNOLOGY

SYMPOSIUM: QUALITY CONTROL IN THE FOOD INDUSTRY

5th and 6th April 1967
Hoare Memorial Hall, Church House, Westminster, London, S.W.1

DAY I. 5th April. THEME: "WHAT ARE THE QUALITIES WE ARE CONCERNED WITH?"
1st Session. *Chairman* Lord Kings Norton
10–12.30 (1) Welcome to Members of the Symposium
(2) Introduction—Dr S. M. Herschdoerfer
(3) What does the Consumer want?

2nd Session. *Chairman* Lord Sainsbury
2–4.30 (4) Chemical and Physical Qualities—Mr. D. Pearson (National College of Food Technology)
(5) Microbiological Qualities
(6) Organoleptic Qualities—Miss M. P. Dixon (Arthur D. Little)

DAY II. 6th APRIL. THEME: "HOW ARE THE DESIRED QUALITIES ACHIEVED?"
3rd Session *Chairman* Lord Trenchard
10–12.30 (1) The Control of Raw Materials—Mr A. Davies (H. J. Heinz)
(2) Plant and Process Control—Mr A. Taylor (Colworth House)
(3) Packaging Control—Mr F. A. Paine (William Thyne & Co.)

4th Session *Chairman* Sir Samuel Salmon
2–4.30 (4) Distribution Control
(5) The Administration of Quality Systems—Mr W. H. Holland (Lyons)
(6) Summing up and Closing Remarks—President of the Institute

INSTITUTE OF FOOD SCIENCE AND TECHNOLOGY

TENTATIVE PROGRAMME, 1967

Date	*Time and Place*	*Subject*	*Speakers*
14 February	The University, Whiteknights Park, Reading (Joint with Students Society) 5.15 p.m.	Fats in Relation to Food Technology	Mr K. Berger
23 February	The University, Leeds (North of England Branch) (Joint with Students Society) Evening	Film and Talk: The Science and Technology of Spices	Mr H. B. Heath and Mr T. S. E. Powell
1 March	School of Agriculture, University of Nottingham, Sutton Bonnington (Midlands Branch)	Symposium: Current Trends in Food Legislation	
2 March	Imperial College, London, S.W.7 (Joint with Institute of Packaging) 6.30 p.m.	Vacuum and Gas Packaging of Foods	Mr F. Fidler Mr J. J. Cavett
15 March	Dublin and Carlow (Joint with Carlow Scientific Council)	Visits, Dinner and Meeting: What we will be Eating in A.D. 2000	Dr J. G. Davis
16 March	Belfast	Microbiological Standards in the Food Industry. The Next 21 Years in Frozen Food Industry	Dr J. G. Davis Mr H. Symons
5–6 April	Church House, Westminster, S.W.1	Symposium: Quality Control in the Food Industry	See previous page
12 April	Gas Showrooms, Manchester (North of England Branch)	Cider/Cheese Tasting Meeting	Mr Buckle Mr B. D. Peacock
3 May	School of Agriculture, Food Science Department, University of Nottingham, Sutton Bonington (Midlands Branch)	Open Day	
10 May	Rowntree & Co., York (North of England Branch)	Visit to Factory and Meeting	
18 May	Bridewell Hall, 6 Eccleston St, London, S.W.1 (Joint with Royal Society of Health) 6.30 p.m.	Man and his Food	Sir William Slater
21 June	Midlands Branch	Visit to Factory and Lecture by Member of Staff	

Arrangements are in hand for a meeting in January to discuss the formation of a North of Ireland Branch; in June and July visits to factories or research stations; in September a Ladies' Evening to one of Safeway Food Stores; in late autumn (North of England Branch) Symposium on 'Flour and Bakery Products'.

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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.

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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	centimetre(s)	cm
microgram(s)		litre(s)	l
(10^{-6} g)	µg	millilitre(s)	ml
nanogram(s)		pound(s)	lb
(10^{-9} 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.

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