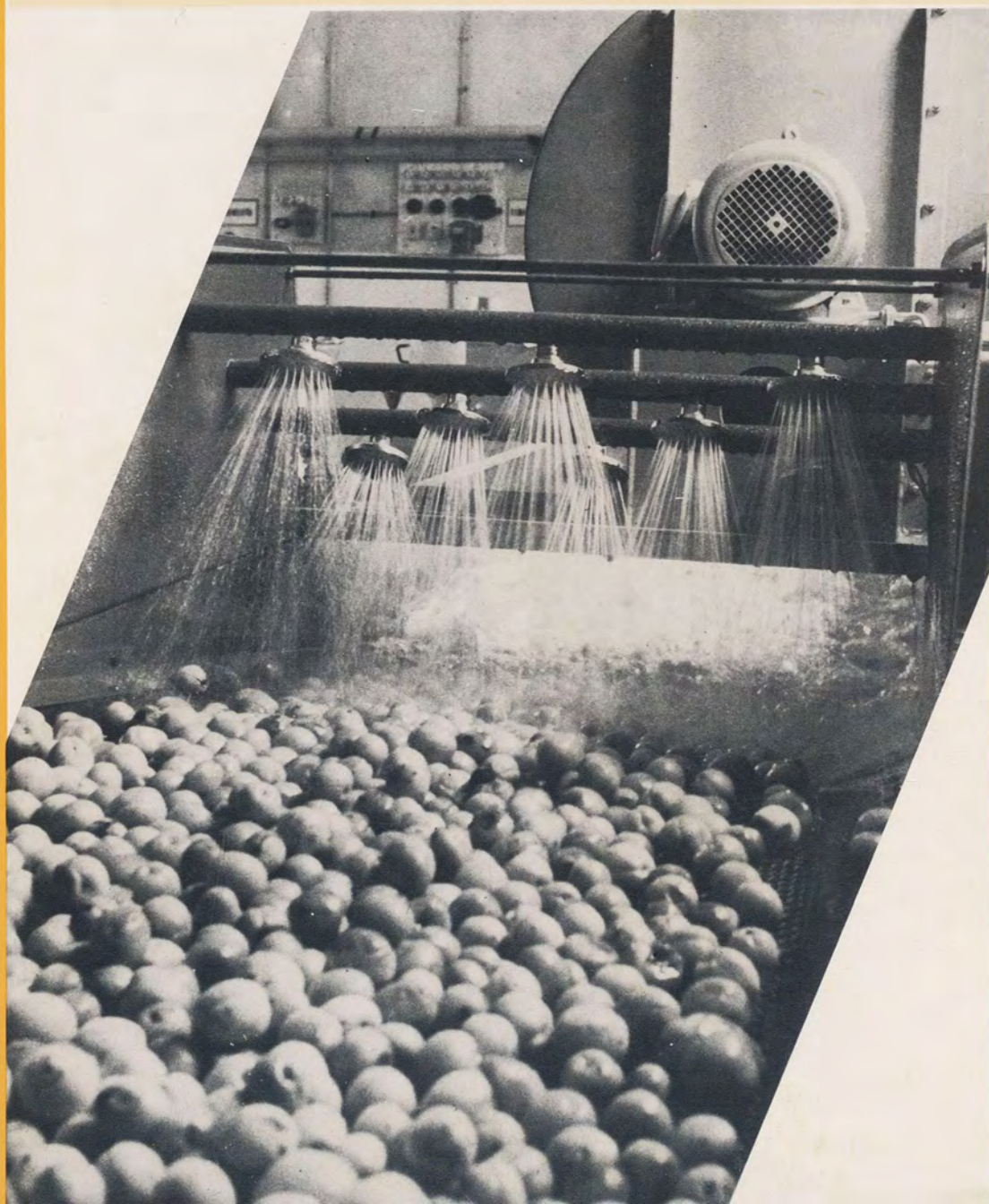


Quality control in fruit and vegetable processing



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by
P.W. Board

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Chapter 1

INTRODUCTION

Fruit and vegetable processing industries produce very large quantities of products which are intended for consumption, often on a daily basis, by the population at large. Such industries therefore have a special responsibility to ensure that their products are both wholesome and safe, as well as successful in the marketplace.

This manual is intended for use by processors of fruit and vegetable products as a guide to the establishment and operation of soundly based and effective quality control systems. The manual deals with quality control in the following types of fruit and vegetable processing:

- . canning;
- . dehydration;
- . freezing;
- . pickling, syrumping, crystallizing and chemical preservation.

In preparing the manual it was accepted that only limited resources can be made available in any commercial enterprise for quality control. It was also recognized that at least a minimum level of quality control is essential because the products of the fruit and vegetable processing industry are intended for human consumption. The amount of a company's resources that should be given to quality control operations will vary according to the nature of the product and process, the possible hazards associated with defective products being produced and consumed, and other factors. It was also recognized that the way quality control systems are structured and manned varies according to the nature of the product and process and the size of the manufacturing operation. In some factories employing only a few people, quality control may be only one of several responsibilities of a particular member of staff but even in that situation it is essential that an appropriate level of quality control be maintained. In large processing organizations employing hundreds of people, quality control may be the sole responsibility of a defined team. In view of the diverse nature of individual factories in the fruit and vegetable processing industry, it was decided that the manual would:

- . discuss the principles of quality control systems appropriate to fruit and vegetable processing operations (chapter 2).
- . describe how quality control procedures should be applied in fruit and vegetable processing operations (chapter 3).
- . give details of methods of examination and calibration that are appropriate for use in quality control in the fruit and vegetable processing industry (chapter 4).

Chapter 2

ESTABLISHING QUALITY CONTROL SYSTEMS

2.1 PRODUCTION PLANNING

The production of acceptable, wholesome and safe fruit and vegetable products depends on three interrelated factors:

- . specifications for raw materials, the product and the packaging system, must be based on the requirements of the intended market and the requirements of regulatory authorities;
- . a production procedure must be designed to give a product which complies with the specification and must be established and properly applied;
- . A quality control system must be established for monitoring the raw materials, the production procedure and the final product to ensure, as far as possible with available resources, that the product is within the specifications.

The preparation of product specifications should involve representatives of marketing, production and quality control staff and occasionally the research and development staff. The marketing staff should be able to give advice on what qualities the product should have in order to meet the requirements of intended consumers. At the same time the production and the quality control staff with the research and development staff should be able to give advice on how a product having those qualities can be produced safely and repeatedly. The production and the quality control staff should also interact in the detailed planning of the production procedure and the related quality control systems. In this way both groups understand what each is trying to do and how their respective task can be carried out most effectively. Clearly, it is important that there be joint planning of the process before production starts and that the production and quality control staff review their procedures as often as needed after production starts to ensure the product complies with the agreed specifications.

In small factories where one person may be responsible for marketing, production and quality control, that person should assess the whole production operation as described above; i.e. firstly develop specifications for the raw materials and product, then decide how the product will be monitored.

2.2. INTERACTION OF QUALITY CONTROL AND PRODUCTION STAFF DURING PRODUCTION

The close collaboration between quality control and production staff, which is so important during the planning of the manufacturing process, should be continued when the process is put into operation. This requirement is probably more pertinent in the fruit and vegetable processing industry than

in most other industries because few attributes of these products are amenable to objective measurement and fewer still of these measurements can be automated. Much reliance must therefore be placed on subjective assessments in efforts to ensure that the manufacturing process is operating properly and the product is satisfactory. It follows that the effectiveness of quality control increases as the number of people observing the product and process increases. Clearly there are advantages involving everyone who is in contact with the product and process in some aspect of quality control. For example, people operating can closers should inspect the loose ends to confirm that they are correctly formed, the compound is properly distributed and that the ends are the type required for the product being packed. The closer operators should also check the canner's double seam as often as possible for defects such as droops, spurs or cutovers (see "Double seams" p.33).

The involvement of production personnel in some aspects of quality control is contrary to some management concepts in which different functions within the organization are compartmentalized. These forms of organizational structure clearly separate responsibilities for production and quality control. However, for the fruit and vegetable processing industries this separation is a hindrance to ensuring that the manufacturing process is operating satisfactorily and that the final product complies with its specification. The preferred organizational structure therefore requires some overlap of the responsibilities of the production and quality control staff. The most important advantages of this arrangement are that the number of dedicated observers on the production line is increased and deviations from the norm in the process or product should be detected and reported with minimum delay.

Even though the recommended organizational structure for quality control and production in fruit and vegetable processing factories requires close collaboration between the two groups, it is also essential that they both report directly to and have equal status with management. This requirement is especially important when a conflict of opinion occurs. For instance, quality control personnel may consider that a product does not comply with the specification while production personnel wish to continue processing; this type of problem should be referred to management for a decision.

2.3 ALLOCATION OF RESOURCES TO QUALITY CONTROL

Management is responsible for deciding how much of the organization's available resources are given to quality control. This decision would be influenced by the requirements of statutory authorities, e.g. the authorities may require a canner to measure the concentration of chlorine in retort cooling water so he would have to provide appropriate staff and equipment. The decision would also have to be based on a judgement of the protection afforded by establishing a particular level of quality control compared to the possible risks to consumers, to the processor's reputation and perhaps even to the industry's reputation if fault products reached the market. The risks vary according to the nature of the product, the requirements of the market and other factors and they are impossible to quantify. It must also be accepted, in deciding how much of the company's resources should be given to quality control, that even the most elaborate quality control system will not give absolute assurance that only acceptable products reach the market. Management must also be aware that the absence of consumer complaints does not mean that a quality control system is unnecessary; it may mean that the

system is indeed effective; it may mean that by good manufacturing practice and good luck no defective products reached the market or it may mean that consumers simply failed to complain.

As the resources available for quality control are usually limited they should be directed to monitoring those points which are judged to be most effective in ensuring that the final product complies with its specifications. These points fall into two broad areas:

- . the manufacturing process which starts with the procurement of raw materials and ends with the despatch of the finished product to the market;
- . the environment in which the manufacturing process is carried out which includes general housekeeping, cleaning, sanitation and calibration of control instruments.

2.4 MONITORING THE MANUFACTURING PROCESS

The number and position of the points in the manufacturing process (including the procurement of raw materials) that should be monitored will also vary according to the assessment of potential hazards if at that point the process deviates from the norm. The points to be monitored will depend on the nature of the product and process as well as the resources available. The frequency and places of monitoring should be selected jointly by the quality control and production staff. The procedure for establishing the quality control regime is sometimes based on the hazard analysis critical control point (HACCP) concept. The traditional emphasis of the HACCP concept has tended to be the control of microbiological hazards but the concept is applicable to monitoring other attributes of the product that may determine its acceptability, e.g., net weight, drained weight or composition of the product. The selection of critical control points should be reviewed at regular intervals and changed if experience shows that the system could be made more effective; monitoring of some points may be discontinued or the frequency of monitoring may be changed. In many instances the critical points will already be staffed and under the supervision of production staff. The HACCP concept should therefore be used as a guide for obtaining maximum benefit from the available resources for quality control.

2.5 MONITORING THE MANUFACTURING ENVIRONMENT

Quality control staff should also be responsible for monitoring the environment of the manufacturing process because factors not directly involved with the process may affect its efficiency and the quality and safety of the final product. These responsibilities include:

- . monitoring action taken to prevent infestation of the processing plant by insects, birds and animals;
- . assessing the effectiveness of cleaning and sanitizing programmes.
- . determining that personnel are clean, in good health and dressed in appropriate garments;
- . ensuring that storage areas are clean and tidy and have appropriate temperatures and humidities;

- . inspecting field bins for cleanliness and state of repair;
- . determining that waste disposal systems are operating satisfactorily;
- . calibrating control and indicating instruments on processing equipment such as retorts;
- . responding to consumer complaints.

2.6 END-PRODUCT TESTING

The amount of quality control effort applied to end-product evaluation, once a reliable and effective manufacturing process has been established, should be small compared to the resources given to monitoring the process itself. Tests on the end-product give information which is largely of historic value, whereas monitoring of the process should give an early warning of a fault so that corrective action may be taken before unnecessarily large amounts of materials and time are wasted. Another advantage of in-process inspection, especially where production personnel are accepting some responsibility for quality control, is that all the product is being watched by the operatives. In contrast, end-product inspection requires sampling of the production and this is a major impediment to detecting faulty material.

Any practical sampling procedure is next to useless when the tolerance for defects in foods, and especially hazardous defects, is extremely low. It is often assumed that sampling procedures that are based on statistical theory give a true assessment of the quality of a batch and that such procedures always detect defective units within the batch. It must be emphasized that sampling according to a statistical plan does not offer a miraculous solution to the problem of locating defective units; perhaps the most important advantage to statistical sampling is that under defined conditions it gives a measure of the efficiency of the sampling regime. For example, if 0.5% of the randomly distributed units in a large batch were defective and ten samples were taken at random there is a 95% chance of missing a faulty unit; 20 samples would give a 90% chance of accepting the batch and even 200 samples would not contain a defective unit on an average of about one in three occasions. Such figures may be calculated using the equation:

$$p = [(100 - x)/100]^n$$

where p is the probability of failing to find at least one defective unit by taking a random sample of n units when the fraction (%) of randomly distributed defective units is x. Clearly, practical levels of sampling will be reliable only when a large fraction of the batch is defective. Sampling may be even less efficient than these figures indicate because in many instances the defective units may not be randomly distributed.

2.7 INCIDENCE OF DEFECTIVE PRODUCT

Regardless of the effectiveness of quality control and production procedures, small amounts of defective products usually occur in other-wise satisfactory material. These faulty products are often discarded without investigation by the manufacturer because it is not realized that changes in the incidence of defective products and knowledge of the cause of the defects may warn the manufacturer that the process is about to fail. For instance, an increase in the number of cans showing post-processing contamination may indicate that

the quality of the cans has deteriorated, that the canner's seam has departed from specification, or that the cans were contaminated by the cooling water or during the handling operations after cooling. The weakness in the cannery operation should be identified and corrected before the number of spoiled cans exceeds the usual minimum number which occur even with good manufacturing practice.

2.8 TREATMENT OF QUALITY CONTROL DATA

Quality control data that relate to the long-term acceptability and safety of the product should be recorded in detail and the records should allow ready identification of the appropriate batch of the product. Such records should be retained for a period in excess of the usual commercial life of the product; with processed fruits and vegetables; three years is probably adequate. The main use of these records is to assist in responding to complaints about the product when it reaches the market or to help in investigating the cause of defects if they are detected in warehouse stocks.

There are advantages in graphing some quality control data in order to discern short term trends in the process or product. Can seam dimensions, incidence of leaky cans, or net weights are often plotted on control charts or x charts; i.e. the measured values are plotted on the vertical axis. The desired value may be indicated on the graph by a horizontal line and operating limits above and below that value are similarly indicated (figure 1).

Another simple method of treating these types of data, but one which usually gives earlier warnings of deviations from a desired value, is to plot cumulative sum (CUSUM) charts. The difference between the measured value and the required value is calculated and these differences are plotted as a cumulative total against time or the number of the sample. Figure 2 was obtained by plotting the data in Figure 2 as a CUSUM chart and the resultant line indicates that after the first five observations there is a general upward trend.

Both control charts and CUSUM charts may be used simply as pictures showing trends in quality control parameters. Under these circumstances the decisions about action to be taken in response to deviations from desired values are largely subjective but nonetheless useful. Better based decisions may be made if statistical methods are applied in developing the charts and in assessing trends; these methods are described in some standard texts on statistics.

2.9 STAFF/MANAGEMENT RELATIONS

The system of quality control recommended for the processed fruit and vegetable industry requires collaboration between marketing, production and quality control staff during planning of the process, and between production and quality control personnel during production. This in turn requires some overlap or breakdown of the boundaries that separate these functions in conventionally structured manufacturing organizations and that requires the support and encouragement of management. Each department must also have direct and independent access to management.

The successful operation of quality control systems for processed fruits and vegetables requires management to encourage personnel at all levels to report deviations from the established process or defects in the product. Any disincentive to report real or apparent problems may result in defective products reaching the market which may have consequences ranging from loss of sales to the death of consumers.

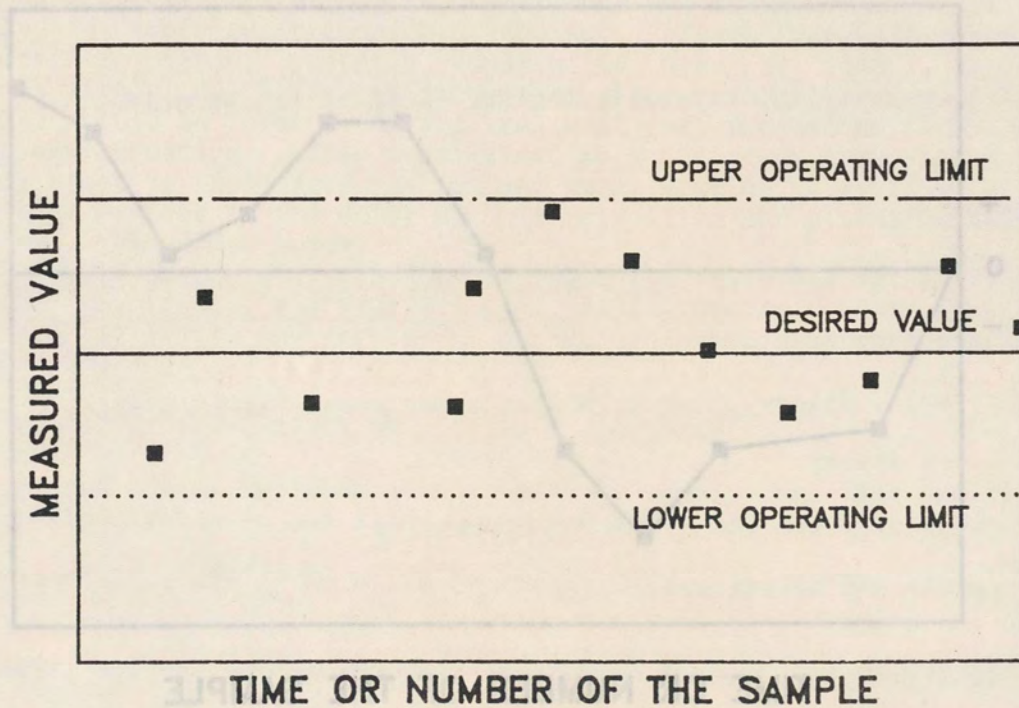


Figure 1. A control or x chart showing typical data points on either side of the desired value and within the upper and lower operating limits

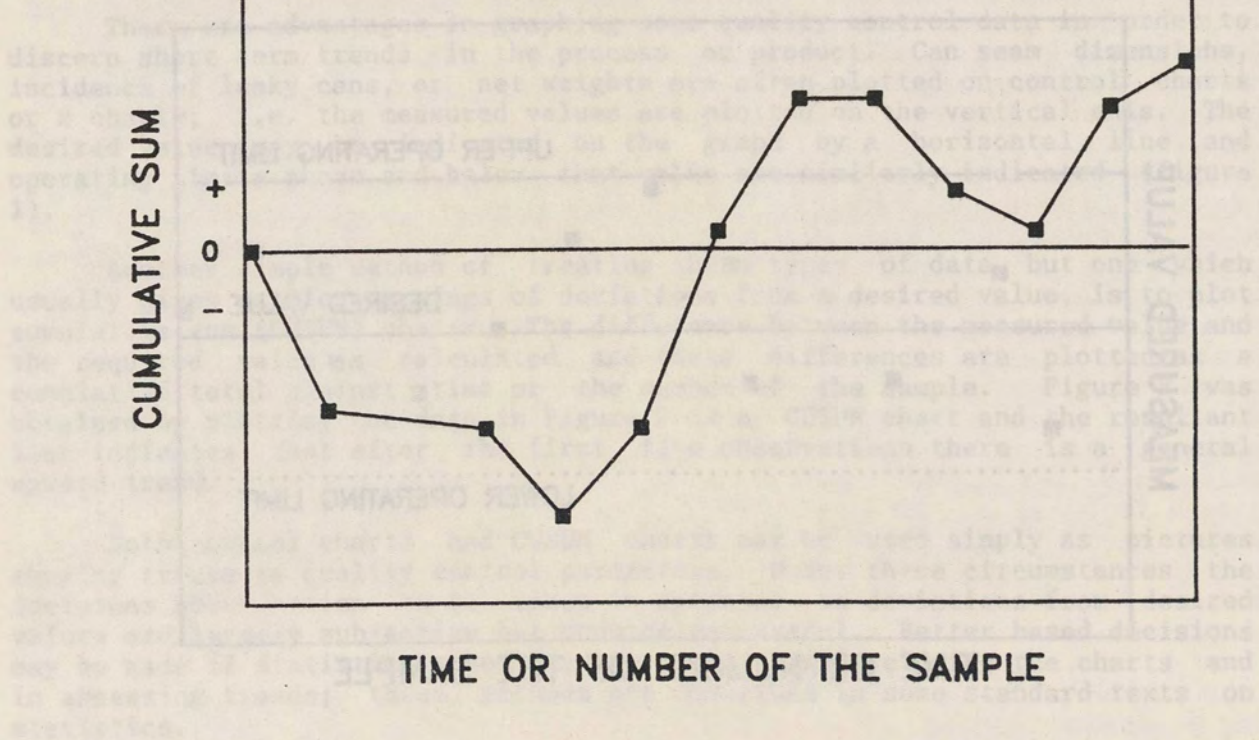


Figure 1. This chart indicates a probable upward trend which is not obvious in Figure 1.

The successful operation of quality control systems for processed fruits and vegetables requires management to encourage personnel at all levels to report deviations from the established process or defects in the product. Any disincentive to report real or apparent problems may result in defective products reaching the market which may have consequences ranging from loss of sales to the death of consumers.

Manufacturing processes fall into two groups: 1. Primary processing - This group includes the operations which convert raw materials into finished products. It includes the operations of cutting, peeling, washing, and blanching. 2. Secondary processing - This group includes the operations which convert primary products into finished products. It includes the operations of canning, freezing, and packaging.

The raw materials for the fruit and vegetable processing industry include: 1. Fruits - Apples, pears, peaches, cherries, etc. 2. Vegetables - Tomatoes, cucumbers, green beans, etc. 3. Spices - Salt, sugar, etc. 4. Other ingredients - Water, oil, etc.

These raw materials are processed in the following manner: 1. Primary processing - The raw materials are cut, peeled, washed, and blanched. 2. Secondary processing - The primary products are canned, frozen, and packaged.

Fruit and vegetable processing involves the following steps: 1. Raw materials - Apples, pears, peaches, cherries, etc. 2. Primary processing - Cutting, peeling, washing, and blanching. 3. Secondary processing - Canning, freezing, and packaging.

Quality control is essential in the fruit and vegetable processing industry. It is essential to ensure that the products are safe and of high quality. The following are some of the key areas of quality control: 1. Raw materials - Ensuring that the raw materials are of high quality and free from defects. 2. Primary processing - Ensuring that the primary processing steps are performed correctly. 3. Secondary processing - Ensuring that the secondary processing steps are performed correctly. 4. Final product - Ensuring that the final product is of high quality and free from defects.

Chapter 3

QUALITY CONTROL SYSTEMS FOR FRUIT AND VEGETABLE PROCESSING

The processing of fruits and vegetables involves several steps or unit operations which start with the procurement of the raw material. Following preparative treatments such as cleaning, peeling, blanching and mixing; the product is processed and packaged to give the required finished product. The manufacturing processes fall into four groups:

- . canning;
- . dehydration;
- . freezing;
- . pickling, syruping, crystallizing and chemical preservation.

3.1 RAW MATERIALS

The raw materials for the fruit and vegetable processing industry include:

- . fruits and vegetables;
- . sugar, salt, spices, food acids and other minor ingredients;
- . water and steam;
- . containers, labels and packaging materials;
- . detergents and sanitizers.

Fruit and Vegetables

Quality control staff should become familiar with the practices involved in growing, harvesting and transporting the fruits and vegetables to the processing factory.

Field Practices: Quality control staff should determine what agricultural chemicals are used by growers and how and when they are applied to the crop. Only produce that has been grown under approved conditions (and that complies with the processor's specifications for the raw material) should be accepted for processing because it is seldom feasible to analyse raw fruits and vegetables for residuals of agricultural chemicals before the consignment is processed.

The growing areas should also be inspected to ensure that the raw fruits and vegetables are not contaminated with other potentially hazardous materials such as toxic waste water or gaseous emissions from neighbouring industries.

Maturity of fruits and vegetables. The quality of many processed fruits and vegetables is markedly influenced by the physiological maturity of the raw materials at the time of harvest. The maturity of fruits and vegetables is often determined by visual inspection and from the tactile properties of the products but objective methods are also used. For example, the maturity of sweet corn is related to its moisture content, refractive index and to the Succulometer reading; the maturity of green peas is indicated by the content of alcohol insoluble solids and by the readings of such instruments as the F.M.C. Tenderometer, Maturimeter or Ottawa Texture Measuring System. The Effe-gi and Magness-Taylor pressure testers are used to measure the maturity of some pome and stone fruits and sugar/acid ratios indicate the maturity of citrus fruits (for analytical methods see "Chemical analyses" p.20 and Kramer and Twigg, 1962).

Transport of fruits and vegetables. The quality of the final product is often influenced by the way the raw fruits and vegetables are harvested and handled during transport from the growing area to the factory. Quality control staff should inspect these operations to ensure that the raw materials are handled carefully to minimize mechanical damage. These products should be transported in clean, properly constructed bins or other appropriate containers without delay to the processing plant. Loads of raw fruits and vegetables should be covered during transport for protection from the sun, rain and contamination. Some vegetables, such as green peas and asparagus which are prone to rapid self-heating and deterioration in quality, should be hydro-cooled or iced if the delay between harvesting and processing is likely to be longer than a few hours.

Storage of raw fruits and vegetables. Quality control staff should also inspect the raw fruits and vegetables during storage at the processing plant to ensure they are:

- . protected from attack by rodents and insects;
- . stored under suitable conditions of temperature and humidity for periods which do not allow excessive deterioration (see Annex "A");
- . handled carefully to minimize mechanical damage.

Sugar, salt, spices, food acids and other minor ingredients

These materials may also have a marked effect on the quality and safety of the finished product so quality control staff should require these ingredients to be obtained from reputable suppliers and the materials should be of at least "food grade" quality. If possible quality control staff should visit suppliers to satisfy themselves that these items are produced, packed and stored under conditions of good manufacturing practice. Again it is seldom possible for quality control staff to analyse such materials for purity or for the presence of any of a large number of potentially dangerous contaminants.

Inspection on receipt. Quality control staff should inspect all consignments of ingredients on receipt to confirm that they comply with the company's purchasing specifications. If resources are limited the quality control staff may restrict the inspection to the essential minimum of:

- . confirming that the materials delivered are the ones ordered (e.g. salt should be confirmed to be salt and not another white crystalline

substance);

- . determining that the consignment was not damaged or contaminated to the extent that the materials are not suitable for their intended used.

Acceptance testing may require simple chemical tests, e.g. to determine the concentration of acetic acid in vinegar (see "Acidity", p.20), or the ingredient may have to be used in the production of a small batch of the product to test that it is satisfactory, e.g. to assess the potency of spices.

Microbiological tests of ingredients for the manufacture of processed fruit and vegetable products are seldom warranted. However, it is prudent to limit the number of thermophilic sporing bacteria that may be incorporated in low-acid canned products such as soups through the use of contaminated ingredients such as thickening agents or dry spices, especially if these products are intended for distribution to hot climates. Mouldy materials such as dried peas and beans must not be accepted as ingredients because of the risk of aflatoxins. It is seldom feasible to test mouldy material for such toxic substances; although relatively simple methods of detection are available (see "Aflatoxins", p.21).

Water and steam

Quality and distribution of water. Water of various qualities may be used at different points in most food processing operations and it is common for water to be reused. Water with obvious turbidity is often satisfactory for soaking root vegetables or for fluming fruit which is to be chemically peeled, but only water which is suitable for human consumption should be used for the final washing of the product or as an ingredient, e.g. in brines or syrups. Only properly chlorinated clean water should be used for cooling canned products after the heat sterilization process.

Quality control staff should inspect water supply and distribution systems to ensure that only water of an appropriate quality is being used, and they should regularly check the concentration of free residual chlorine in the chlorinated water supply at the points of use (see "Chlorine in water", p.24). Water lines should be purged before processing operations start to ensure that only water of the required quality is being delivered.

Steam quality. Steam also comes into contact with fruits and vegetables in some processing operations. These include some lye-peeling systems, steam-heated exhaust boxes, steam-flow closers, steam blanchers, and water blanchers which are heated by direct injection of steam. Quality control staff should determine that either the steam that comes into contact with the product is not contaminated by boiler additives or that the additives are not harmful, or both. Quality control staff should also ensure that condensate and the products of corrosion that may accumulate in the pipework of the steam distribution system during shut-downs are removed by purging the system before food processing operations start. It is also important to determine that steam traps and strainers are maintained in good condition to help ensure that clean steam is delivered to processing points.

Containers, labels and packaging materials

Quality control staff should inspect each consignment of containers, labels and packaging materials to ensure that they are delivered in good conditions and that the items comply with the purchasing specification. In addition, samples of primary containers (cans, drums, glass jars, flexible pouches and bags and semi-rigid aluminium tray packs) should be examined to determine that the properties of these items that are critical to the safety and storage stability of the product are satisfactory.

Cans. Samples of cans should be examined to determine that the following features are within specifications:

- . the double seam (see "Double seams", p.33);
- . the side seam and flange of the open end;
- . the type and coating mass of internal lacquers, and their coverage and degree of adhesion (see "Lacquer adhesion", p.51);
- . the tin coating mass ("Tin coating mass", p.58);
- . the structure of the loose ends and the placement and amount of compound.

Some cans should also be filled with water and closed so that the canner's double seam may be assessed (see "Double seams", p.33). If the seams are of doubtful quality the cans should also be leak tested (see "Leak tests", p.52).

Glass containers. Samples of glass containers should be examined for defects in construction and for variations in dimensions that may affect their ability to be properly closed and to withstand impacts and other abuses which are encountered during filling, closing, processing, distribution and storage. Quality control staff should give special attention to the sealing surface of glass containers; this surface should be horizontal and smooth and it should make a cleanly-defined and continuous contact with the gasket when the closure is applied under usual processing conditions.

Many types of closures are applied to glass containers so quality control staff should obtain information on the structure and application of the closures from the container manufacturer to plan realistic inspection procedures. The critical factors influencing the quality of the seal on glass containers include the dimensions of the closure and the type, quantity and distribution of the gasket material. The storage performance of glass containers closed with metal closures often depends on the resistance of the closure to corrosion by the product, especially an acidic product containing salt and sulphur dioxide. Metal closures are usually protected by an internal lacquer which must have excellent barrier properties. Sample closures should therefore be examined to determine that the lacquer is essentially continuous, applied at the specified coating mass (see "Lacquer coating mass", p.51) and adheres strongly to the surface of the metal (see "Lacquer adhesion", p.51).

Plastic pouches and semi-rigid aluminium tray packs. Semi-rigid aluminium tray packs have some technical features in common with plastic pouches in that they are closed by heat sealing and they have some flexibility. Quality

control staff should confirm the identity and thickness of plastic packaging materials since these properties mainly determine the barrier properties of the package (British Cellophane, 1970). If the shelf life of the product is critically dependent on the barrier properties of the package, the oxygen permeability and/or the water vapour permeability and the integrity of sample packages should be determined before they are used in production (see "Leak tests", p.52; "Oxygen permeability", p.53; "Pinhole test for packaging films", p.55; "Water vapour transmission rate", p.59).

Labels, cartons and other ultimate containers. Quality control staff should inspect and measure these items to determine that they conform to the purchasing specifications. The information displayed on labels and on ultimate containers should be checked for accuracy, and the registration and quality of the printing and art work should be assessed by quality control staff.

Detergents, sanitizers and similar materials

Quality control staff should inspect these items on receipt using the same approach as that described for food ingredients (see "Sugar, salt, spices, food acids and other minor ingredients", p.11).

3.2 PREPARATIVE TREATMENTS

Production staff have primary responsibility for carrying out such treatments as sorting, grading, washing, blanching and mixing. However, because quality control of these operations is mainly based on visual inspection production staff should be encouraged to assist quality control staff to ensure, for instance, that:

- . sorting and trimming is properly done;
- . foreign material is removed;
- . the product is thoroughly washed;
- . the product is sub-divided as required for the style of the pack;
- . the product moves continuously and with minimum delay through the various operations.

Quality control staff should monitor the concentration of chemicals in chemical peeling baths; usually the active material is caustic soda (sodium hydroxide) so acid titrations should be done often enough to ensure that the required concentration can be maintained (see "Caustic soda (lye)", p.23).

Quality control staff should also check the composition of brines and syrups used as packing media and in the manufacture of pickled, syruped and crystallized products. The concentration of salt in simple brines may be estimated by hydrometry (see "Salt", p.27), and indicated by a simple test of electrical conductivity, or even by tasting. In brines containing other substances chemical methods are used to determine the concentration of salt (see "Salt", p.27).

The concentration of sugar in simple syrups is most readily determined

by refractometry but hydrometry may also be used (see "Soluble solids, sugar", p.28). Again tasting gives an estimate of the strength of a syrup.

Brines and syrups are sometimes acidified and in some instances the level of acidification is critically important in determining the safety of the final product. Quality control staff should check the acidity of each batch of acidified brine or syrup by alkaline titration (see "Acidity", p.20); pH measurements or tasting are not appropriate in this situation.

3.3 FILLING

Quality control staff should monitor the filling operation to ensure that each container receives at least the nominated amount of the product and that the container is not excessively filled. Excessive fills are commercially wasteful and in some instances they may be hazardous. For example, the double seams of canned foods may be damaged if the can is overfilled because the product expands more than the metal of the can during the heat sterilization process. The fill may be checked by weighing or by measuring the volume of the product. The headspace, i.e. the distance between the top of the open can and the surface of the product, may also be measured (see "Headspace of canned foods", p.50). It should be remembered that the mass and volume of a product will vary with its density, and density will vary according to the composition of the product and its temperature.

Quality control staff, with support from production staff, should inspect the filling operation to ensure that the sealing surfaces of the containers are not contaminated with spilt products which may cause the seal to be substandard. Fibrous vegetable tissue may cause leaky seals if the tissue is incorporated in double seams of cans or between the gasket and finish of glass containers. The heat-seal surfaces of flexible pouches and semi-rigid aluminium tray packs should also be free of the product and moisture if sound heat seals are to be produced.

In some processes, e.g. the hot-fill, close, hold and cool procedure which is sometimes used in canning acid foods, the temperature of filling is a critical factor in ensuring the product is shelf-stable. This temperature should be monitored continually using a temperature recorder, or it should be under frequent observation of quality control staff with the support of production staff. The temperature of filling may also be an important factor in producing the required vacuum in canned products and again this temperature should be monitored as described above.

3.4 CLOSING AND SEALING OPERATIONS

Many processed fruit and vegetable products must be hermetically sealed in their primary package if they are to be safe and shelf-stable. Quality control staff, preferably with assistance from production staff, should inspect filled containers, loose ends and caps as they arrive at the closing machines or heat-sealers, and some of the closed containers as they leave the closing operation.

Operators of can closers should inspect the finished double seams for defects such as droops, spurs, cut-overs and false seams. At regular intervals, depending on the rate of production, detailed examinations should be made of the closures on sample containers taken from each head of the closing machines. Samples should also be examined after the machine is

adjusted and after accidents that may have altered the settings of the closing machines and the quality of the seals (see "Double seams", p.33).

The assessment of heat seals should be mainly based on visual examinations during production but the seals should be pressure tested when the sealing machines are being adjusted before processing starts or after accidents that may alter their performance ("Leak tests", p.52).

Quality control staff should also check that the correct date, product name and manufacturer's codes are being applied in legible form to the primary containers, and that the appropriate codes are changed frequently enough to allow production batches to be identified and isolated if there is evidence that they are defective.

3.5 PRESERVATION TREATMENTS

In addition to packaging processed fruits and vegetables in properly sealed containers, the products must be given other treatments to ensure that they are stable during storage. Some preservation treatments such as dehydration and crystallizing are usually done before packaging while other treatments are usually applied after packaging, e.g. the heat sterilization process in canning. The preservation treatments involve different physical and chemical processes and they require different actions by quality control staff to ensure that safe, stable products are produced.

Canned Foods

The safety and storage stability of canned foods, including heat-processed foods in drums, glass containers, flexible and semi-rigid containers, depend on the product being heated at a specified temperature for a specified time. The most commonly used heating media are hot water, sometimes under a superimposed pressure, and air-free saturated steam.

Quality control staff should ensure that the primary control instruments for heat-sterilization processes, the thermometer and clock, are accurate and maintained in good condition. Mercury-in-glass thermometers, or temperature-measuring instruments of at least the same accuracy and reliability, should be calibrated at least twice a year or more frequently if their settings appear to have been disturbed (see "Calibration of thermometers for processing equipment", p.30).

Most heat-sterilizing equipment should also be fitted with chart recorders which give a permanent graphical record of the temperature of the heating medium and, with batch processes, a record of the duration of heating. This record should be used by quality control staff at the end of each shift to check that the specified processes were applied and to confirm the processing details recorded in the log book maintained by the operators of the heat-processing systems.

At regular intervals, perhaps weekly, quality control staff should inspect the heat-sterilizing equipment to ensure that it is operating in the required way. Special attention should be given to the valves on the compressed air and water lines that are connected to steam-heated sterilizing equipment. The traps on steam-heated retorts should also be inspected to ensure that condensate is quickly removed from retorts. The steam, water and air distribution pipes should be inspected for blockages and rust deposits

and the systems used to circulate water in water-heated equipment should be checked to determine that the equipment is operating satisfactorily (Codex 2-1969; Codex 23-1979).

Dehydrated Foods

The safety and storage stability of dehydrated foods, including some syruped and crystallized foods, depend on the moisture content of the product being reduced to a value at which potential spoilage organisms cannot grow (Codex 3-1969; Codex 5-1971). Although equilibrium relative humidity ("Equilibrium relative humidity", p.46.) is the best index of the amount of water available for microbial growth, quality control of dehydration processes is usually based on the measurement of moisture content ("Moisture", p.24). The relationship between moisture content and equilibrium relative humidity varies according to the composition of the food so the moisture content required to give a shelf-stable product should be determined for each food.

Sulphur dioxide is added to some foods before, during or after the drying process, sometimes as an anti-microbial agent, but more often to block non-enzymic browning of the product during storage. Quality control staff should therefore monitor the sulphuring processes and the concentration of sulphur dioxide in the finished product to ensure that enough is present to meet the technological needs but the amount does not exceed the limits set by regulatory authorities ("Sulphur dioxide", p.30).

Frozen foods

The critical factor in ensuring that frozen foods are safe and store satisfactorily is the temperature of storage which should be maintained at an essentially constant value at or below - 18°C (Codex 8-1976).

Quality control staff should measure the temperature of the product (see "Temperature of frozen foods", p.57) as it leaves the production line to ensure that freezing is complete and to determine whether the product should be close- or open-stacked in the cold storage room. The temperature of the cold storage room should be monitored at least twice daily or preferably by using a chart recorder. Quality control staff should also inspect the cold storage space to determine that it is clean and that stock is properly handled and rotated.

Pickled and chemically preserved foods

Spoilage of these foods is prevented by establishing a defined chemical environment throughout the product and by processing the raw materials so that the product is contaminated only by low numbers of micro-organisms. The materials used to make these products shelf-stable include some of those described in section 4.2: acetic and other food acids (p.20); benzoic acid (p.22); salt (p.27); sugar (p.28); sorbic acid and sorbates (p.29); and sulphur dioxide (p.30). In many cases the pH of the food must also be controlled to obtain shelf-stable products (see "pH", p.25).

Quality control staff should ensure that the manufacturing process is carried out so that the level of microbial contamination is minimized. Quality control staff should also monitor the pH and the concentration of the critical preserving agents in each product batch often enough to ensure that

the end product will comply with its specifications. Usually samples for analysis should be taken from different parts of pickling or syruping tanks to determine that the critical materials are properly distributed and present at concentrations within the limits set for the process.

3.6 LABELLING, PACKAGING AND WAREHOUSING

Quality control of these operations involves inspection of the labelling and packing process to determine that the correct materials are being used, that the products are being properly packed and the ultimate containers are securely stacked ready for distribution.

Quality control staff should inspect warehouse stocks at approximately weekly intervals for evidence of spoilage and other forms of deterioration, and to ensure the warehouse is clean, orderly and free of foreign materials and infestation.

A short time before stored stocks are to be shipped quality control staff should select one ultimate container from five widely distributed points in each batch in the consignment. Each primary container in these samples should be inspected for evidence of spoilage and other forms of deterioration. One primary container from each ultimate container should then be taken for more detailed examinations. The product from each primary container should be tasted to determine that it has acceptable organoleptic properties.

Cans should be examined for vacuum, excessive internal corrosion, staining and loss of adhesion by the lacquer (see "lacquer adhesion", p.51, "vacuum", p.58). If the microbiological stability of the product depends on its pH, e.g., in acidified low-acid foods, the pH should be measured (see "pH", p.25).

If the intended market requires particular characteristics in the product, e.g., more than a minimum drained weight or syrup strength, appropriate measurements should be made (see "Soluble solids (sugar)", p.28; "Drained mass", p.32).

Dried foods should be inspected for integrity of the package, insect infestation and microbial spoilage, especially by moulds. The moisture content and, when appropriate, the concentration of sulphur dioxide or other materials specified by the purchasing and regulatory authorities should be determined (see "Moisture", p.24; "Sulphur dioxide", p.30).

With frozen foods the final examination usually involves inspection of the primary package and organoleptic testing of the product. The temperature of the product should also be measured to confirm that it is at or below the recommended value (see "Temperature of frozen foods", p.57).

The concentration of salt, acid and preservatives and the pH should be determined as appropriate in pickled and preserved products before they are dispatched (see "Acidity", p.20; "Benzoic acid", p.22; "pH", p.25; "Salt", p.27; "Soluble solids, sugar", p.28; "Sorbic acid and sorbates", p.29; "Sulphur dioxide", p.30). Attention should be given to the condition of metal containers and to the metal closures on glass jars especially as these products are often particularly corrosive.

3.7 CLEANING, SANITATION AND WASTE DISPOSAL

Quality control staff should monitor all operations that contribute to the manufacturing process being carried out under satisfactory hygienic conditions. The food processing equipment that handles the product during and after the last steps of the preparative operations should be kept at least as clean as the kitchens of a well-run hotel. Quality control staff should inspect the processing lines before manufacturing starts to determine that they are clean and free of waste food, foreign materials and insect infestation. The lines should also be inspected after they are cleaned at the end of the shift. During production quality control staff should inspect the line for evidence of accumulated dirt and waste material, and for any product which has been delayed or by-passed on the line. Special attention should be given to canned foods immediately after the heat sterilization process; the outside surface of the containers must be kept as free of microbial contamination as possible reduce the risk of organisms gaining entry to the product through the wet closures which at that stage may not have formed an hermetic seal. Wet containers should not be manually handled or contaminated by contact with wet and dirty mechanical equipment. Quality control staff should require can handling equipment to be dry, or if it must be wet it should be regularly sanitized. There is little value in taking microbiological swabs of fruit and vegetable processing equipment to assess the effectiveness of cleaning operations; it is usually sufficient to inspect the equipment, to search for off-odours, to feel metal surfaces for sliminess and to wipe the surfaces with a clean tissue to detect residual dirt (Codex 1 - 1979).

Quality control staff should also inspect all other areas of the processing plant and the immediate outside areas for evidence of waste, insects, rodents and other animals, birds and other materials that may present a risk to the production of safe, wholesome processed fruit and vegetable products.

Chapter 4

METHODS OF ANALYSIS AND PHYSICAL TESTING

4.1 SCOPE

This chapter gives details of selected methods of analysis and physical tests that are suitable for use in the fruit and vegetable processing industries. The methods are taken from, or are based on, those described by different authorities and authors. They were selected because they are effective, sufficiently accurate for quality control purposes and in general they required equipment that should be readily available to the fruit and vegetable processing industry.

It is possible that alternative methods may have to be used to comply with the requirements of regulatory authorities or the purchaser of the product. It is also possible that analyses and tests in addition to those described may be required.

The methods described are presented in alphabetical order in this chapter in Sections 4.2 and 4.3. The methods are intended for use by appropriately trained technical personnel so only in instances involving special hazards are warnings about safety included.

4.2 CHEMICAL ANALYSES

Acidity

The concentration of food acids such as acetic, citric, lactic and malic acids is estimated by titrating a sample of the food with sodium hydroxide to an end-point at pH 8.1.

Procedure: For liquid samples take 10 ml and dilute to about 100 ml with distilled water. For solid samples take 10 g and blend with about 50 ml of distilled water to a uniform dispersion, transfer to a conical flask with water.

Add 0.3 ml of indicator (1% phenolphthalein in 95% ethanol) and titrate with 0.1 N sodium hydroxide to a permanent pink colour.

If the colour of the sample obscures the colour change of the indicator the end-point of the titration (pH 8.1) should be determined using a pH meter (see "pH", p.25). An alternative procedure is to use an external indicator (1% phenolphthalein in powdered potassium sulphate on a spot tile).

Results: Titratable acidity is usually reported as percentage mass/volume for liquid products and percentage mass/mass for solid and semi-solid products. The results must also name the acid selected for calculating the

titratable acidity, this acid usually being the predominant acid in the product, e.g., citric acid in citrus products, acetic acid in pickles and malic acid in pome fruits.

For 10 ml or 10 g samples of product, the percentage titratable acidity is calculated using the equation.

Titratable acidity (%) = number of ml of 0.1 N sodium hydroxide multiplied by a conversion factor.

where the conversion factor is:

Acetic acid	0.060
Citric acid (anhydrous)	0.064
Citric acid (hydrous)	0.070
Lactic acid	0.090
Malic acid	0.067
Tartaric acid	0.075

Aflatoxins rapid screening method

Aflatoxins are extracted from the sample by blending at high speed in aqueous acetone, purifying with ammonium sulphate, re-extracting into benzene, evaporating to dryness, and redissolving in chloroform-acetone. The final solution is subjected to descending minicolumn chromatography through successive layers of alumina, silica gel and Florisil, and the aflatoxin band is visualized by fluorescence.

Procedure: Preparation of column. Plug one end of a glass tube (3 mm internal dia x 23 cm long) with 5 mm of glass wool. Add in the following order: 5-7 mm fine granular sand, 5-7 mm Florisil (200-300 mesh, dried 2 h at 110°C), 19-20 mm silica gel (dried 2 h at 110°C). Tap the tube after each addition to form firm layers.

Preparation of sample. Blend 50 g portion of dry sample with 10 g Celite filter aid and 150 ml acetone water (85 + 15) for 3 min at high speed. Filter by gravity through 18.5 cm open-texture filter paper and collect 60-70 ml filtrate.

Transfer 50 ml filtrate to 250 ml beaker, add 20 ml 40% aqueous ammonium sulphate and 130 ml distilled water, stir, and let stand 2-3 min. Add about 10 g Celite, stir, and filter through 18.5 cm paper. Transfer 100 ml filtrate to 125 ml separatory funnel with Teflon stopcock. From pipette, add 3.0 ml benzene; stopper funnel and shake vigorously 60 sec. After phase separation, drain and discard lower (aqueous) layer. Add 50 ml distilled water to benzene in separatory funnel and wash by swirling (very gently, to avoid emulsion formation); again drain and discard aqueous layer. Transfer benzene extract to vial and evaporate just to dryness immersion in hot water under a stream of dry nitrogen. Redissolve residue in 3.0 ml chloroform-acetone (9 + 1). Prepare blank in identical way from materials which is known to be aflatoxin-free.

Chromatographic analysis. Using syringe, transfer 1.0 ml aliquots of blank and samples on to series of columns which are supported in vertical position, and let each drain completely. To each column, add 1.0 ml chloroform-acetone (9 + 1) and again let drain. Observe fluorescence under long-wave ultraviolet illumination. Presence of aflatoxins in sample results in blue

fluorescent band at top of Florisil layer, clearly distinguishable by comparison with blank.

Warning. Aflatoxins are very toxic so rubber gloves should be worn and manipulations should be carried out in a fume cupboard. Grinding of dry samples may result in airborne dust so protective masks should be used. Spills of toxin and contaminated glassware and equipment should be treated with 5% sodium hypochlorite solution and then thoroughly washed with water.

Alcohol insoluble solids

The content of alcohol insoluble solids is determined by weighing the residue obtained by blending a sample of the drained product with alcohol, filtering, washing the residue with more alcohol and drying.

Procedure. Canned vegetables are spread on an 8-mesh screen and rinsed with a volume of water equal to twice the volume of the original can. Frozen vegetables are thawed by immersion in about twice their volume of water at approximately 40°C. Both types of product are then drained and any foreign material is removed by hand. The drained vegetables (400 g) are blended with 200ml water to a fine slurry. A 30 g sample of the slurry is refluxed with 200 ml 80% ethanol for 30 min and the mixture is filtered under vacuum using a pre-dried and weighed Whatman no. 1 filter paper in a 120 mm diameter Buchner filter. The residue on the paper is washed three times then dried for 2 hrs. in an air oven at 100°C and the mass of the residue is determined by subtracting the weight of the paper from the total mass.

Results The percentage of alcohol insoluble solids is obtained by multiplying the mass of dry residue by five.

Benzoic acid

Benzoic acid is estimated by titration with sodium hydroxide after the acid is separated from the sample by extraction or steam distillation. Other acids that may be extracted or distilled with the benzoic acid are destroyed with permanganate.

Extraction. Transfer a sample of 100 g to a 500 ml volumetric flask with water. Solid samples should be macerated before the sample is taken.

Add 10 ml 10% sodium hydroxide and 120 g sodium chloride and adjust the volume to about 400 ml. Shake the flask frequently for an hour and make up to 500 ml, mix and filter. Pipette 100 ml filtrate into a separator, neutralize with 3 N hydrochloric acid to litmus paper and add another 4 ml of 3 N hydrochloric acid.

Extract the benzoic acid with 50 ml chloroform by carefully shaking the separator to avoid formation of an emulsion. Allow the separator to stand for 30 min and run off the chloroform layer and filter it. Pipette 25 ml filtrate into a flask and evaporate the chloroform.

If other acids are present the chloroform should be extracted with sodium hydroxide solution. The other acids are then destroyed by adding 5% permanganate to the alkaline solution at 45°C until a pink colour persists. The solution is decolorized with a solution of sulphur dioxide, dilute sulphuric acid is added to dissolve precipitated manganese dioxide and to

make the solution acidic. The solution is saturated with sodium chloride and the benzoic acid is extracted four times with 15 ml volumes of diethyl ether. The combined ether extracts are washed twice with a small volume of water and filtered into a small flask, the filter being washed with more ether. The ether is evaporated and the residue of benzoic acid is dissolved in 2 ml acetone and 2 ml water is added. The benzoic acid is titrated with 0.05 N sodium hydroxide using phenol red solution (dissolve 50mg phenol red in 2.85 ml 0.05 N sodium hydroxide and 5 ml 90% ethanol by warming and make up to 250 ml with 20% ethanol) as the indicator.

Results. The amount of benzoic acid in the original sample is ten times that in the final titration and that amount is calculated from:

1 ml 0.05 sodium hydroxide = 0.0061 g benzoic acid.

Steam distillation. Transfer a weighed sample of 30-100 g to a 500 ml steam distillation flask with 200 ml water. Saturate with sodium chloride and make distinctly acid with phosphoric acid. Rapidly steam distil about 500 ml into a beaker containing 10 ml 1 N sodium hydroxide. Wash the condenser with 25 ml 0.1 N sodium hydroxide adding the washings to the beaker. Evaporate to about 25 ml on a water bath. Cool to about 45°C and add 5% potassium permanganate solution until a pink colour persists to destroy other volatile acids. The solution is decolorized with a solution of sulphur dioxide and dilute sulphuric acid is added to dissolve precipitated manganese dioxide and to make the solution acidic. The solution is then saturated with sodium chloride and the benzoic acid is extracted four times with 15 ml volumes of diethyl ether. The combined ether extracts are washed twice with a small volume of water and filtered into a small flask, the filter being washed with more ether. The ether is evaporated and the residue of benzoic acid is dissolved in 2 ml acetone and 2 ml of water is added. The benzoic acid is titrated with 0.05 N sodium hydroxide using phenol red solution (dissolve 50 mg phenol red in 2.85 ml 0.05 N sodium hydroxide and 5 ml 90% ethanol by warming and make up to 250 ml with 20% ethanol) as the indicator.

Results. The amount of benzoic acid in the original sample is ten times that in the final titration and that amount is calculated from:

1 ml 0.05 N sodium hydroxide = 0.0061 g benzoic acid.

Caustic soda (lye)

The total alkalinity of caustic soda solutions is estimated by titration with hydrochloric acid. Carbonate in caustic soda solutions is precipitated by adding barium chloride when the concentration of sodium hydroxide in the solution is to be determined.

Procedure. Weigh about 10 g solution into a volumetric flask and make up to 100 ml with freshly boiled and cooled water. Titrate a suitable aliquot with 0.5 N hydrochloric acid using methyl orange (0.5% solution in water) as the indicator. This titration gives the total alkalinity.

Transfer an equal aliquot to a volumetric flask, add enough 10% barium chloride solution to precipitate all carbonate, make up to volume with freshly cooled and boiled water, stopper, shake and allow to stand. When the solution clears pipette half of the volume and titrate with 0.5 N hydrochloric acid using phenolphthalein (1% in 95% ethanol) as indicator.

Results. Twice the number of ml of 0.5 N hydrochloric acid required for the second titration is equivalent to the sodium hydroxide in the original aliquot. The difference between that figure and the number of ml of 0.5 N hydrochloric acid required for the total alkalinity is equal to the number of ml of acid equivalent to the sodium carbonate in the aliquot. One ml of 0.5 N hydrochloric acid is equivalent to 0.02 g of sodium hydroxide or 0.026 g of sodium carbonate.

Chlorine in water

Soon after chlorine is added to water some of the chlorine may exist as **free residual chlorine**, some may be loosely bound to other materials as **combined residual chlorine** and some may react with other substances and become inactive as a germicide. Some regulations require that a minimum concentration of **free residual chlorine** be maintained in water used in food processing operations and other regulations specify minimum concentrations of **total residual chlorine** which comprises the free and combined residuals.

Free residual chlorine and **total residual chlorine** may be determined by measuring the intensity of the colour formed when a solution of orthotoluidine is added to samples of water.

Procedure. Dissolve 1.35 g orthotoluidine hydrochloride in 500 ml distilled water. With constant stirring add 500 ml dilute hydrochloric acid which is made by mixing 350 ml distilled water and 150 ml concentrated hydrochloric acid (specify gravity 1.18). The orthotoluidine solution may be stored in amber bottles in the dark for up to six months.

For the determination, 0.5 ml orthotoluidine solution is mixed with 9.5 ml of the water in a tube and the resultant amber-brown colour is measured after 5 sec and after 5 min to estimate **free residual chlorine** and **total residual chlorine** respectively. The colour may be compared to that formed in solutions of known concentrations of chlorine in distilled water. A similar tube containing more of the same sample of water is used as a blank to correct for colour or turbidity.

Colour comparators that are fitted with discs calibrated for concentration of chlorine are commercially available and widely used. These comparators are often sold with the test reagent in the form of tablets, one of which is crushed in the tube before the sample of water is added. Other test systems are also available commercially, but some of these only measure **total residual chlorine**.

Moisture

Moisture content is commonly determined by weighing the sample before and after the moisture is removed by drying in an air oven or in a vacuum oven.

Procedure. A sample of about 20 g is weighed to the nearest mg into a flat-bottomed cylindrical aluminium dish which has a closely fitting lid. The dish should be about 9 cm diameter and about 2 cm deep. The preparation of the sample and weighing into the dish should be done quickly to minimize loss of moisture. A known amount of spreading agent such as clean dry sand or dry filter paper may be added to foods which are subject to case-hardening, creeping or spattering.

The sample is then dried in the open dish in an air oven at 100-102°C preferably with forced convection. The sample may also be dried at 70°C in a vacuum oven at a gauge pressure of about minus 90 kPa (about 27 in Hg). Air should be admitted to the vacuum oven through a drying train at a rate of at least two bubbles per second to reduce the water vapour pressure in the vacuum chamber. The drying time should be sufficient to give a constant mass; for most fruit and vegetable products overnight drying (about 16 h) is adequate. After drying, the lid is placed on the dish which is allowed to cool to room temperature in a desiccator before reweighing to the nearest mg.

Results. The moisture content is usually expressed as percentage mass of water: original mass, but with dried foods it is sometimes expressed as percentage mass of water: dry mass.

Peroxidase

The absence of peroxidase activity is an accepted indication that fruits and vegetables have been properly blanched in preparation for drying or freezing. The test for peroxidase activity is based on the ability of the enzyme to oxidize guaiacol to a brown material in the presence of peroxide.

Procedure. About 10 g tissue is macerated with added distilled water and clean sand if required and the mixture is filtered. Add 2 ml filtrate to 20 ml distilled water in a test tube. Add 1 ml 0.5% guaiacol in 50% ethanol without mixing. Then add 1 ml of freshly prepared hydrogen peroxide solution (2.8 ml 30% hydrogen peroxide per litre, store in a dark bottle in a refrigerator for up to a week) again without shaking. Mix contents of the tube and observe colour development after 5 min. Carry out a blank test on tissue that has been boiled for 5 min and cooled.

Results. If colour does not develop in 5 min peroxidase has been inactivated. Some products such as corn may give a faint colour even when the enzyme has been inactivated so the blank is especially important in these instances.

pH

The best method for measuring pH involves the use of a pH meter. This instrument consists of a potentiometer, a glass electrode which responds to changes in hydrogen ion concentration, and a reference electrode. Many commercially available pH meters give readings accurate to at least 0.1 pH unit when properly operated and maintained. Modern pH meters have direct reading pH scales and some have digital readout systems. These instruments are available for operation on mains power or with batteries. If the mains voltage is likely to be unstable the pH meter should be fitted with a voltage regulator. The batteries in battery operated pH meters should be checked at regular intervals.

The electrodes of pH meters should be soaked in buffer solution, distilled or deionized water or other liquid specified by the manufacturer for several hours before use. The electrodes should be stored with their tips immersed in distilled water or in the buffer solution used for standardization.

Calomel electrodes are often used as the reference electrode and they should be kept filled with saturated potassium chloride solution or a

solution specified by the manufacturer.

A lag in pH meter response may indicate ageing effects or fouling of the electrodes, and cleaning and rejuvenation of the electrodes may be necessary. This may be accomplished by placing the electrodes in 0.1 N sodium hydroxide solution for 1 min. and then transferring them to 0.1 N hydrochloric acid solution for 1 min. The cycle should be repeated twice, ending with the electrodes in the acid solution. The electrodes should then be thoroughly rinsed with water and blotted with soft tissue before proceeding with the standardization.

To obtain accurate results, the same temperature should be used for the electrodes, the standard buffer solutions and the samples and during the standardization of the meter and the pH determinations. Tests should be made at a temperature between 20°C to 30°C; when tests have to be made outside this temperature range appropriate correction factors should be established and applied. While thermal compensators are available, they should not be relied upon to give accurate results.

Procedure. The instructions of the manufacturer of the pH meter must be followed; usually these instructions will include the following steps:

- . switch the instrument on and allow the electronic components to warm up and stabilize;
- . standardize the instrument and electrodes with commercially-prepared standard pH 4.0 buffer or with freshly prepared 0.05 molar potassium acid phthalate buffer solution. This buffer is prepared by dissolving 10.12 g potassium acid phthalate (dried 2 h at 110°C) in 1 L distilled water. (This buffer has a pH of 4.0 at temperatures ranging from 0°C to at least 40°C). Note the temperature of the buffer solution and set the temperature compensator control at the observed temperature;
- . rinse the electrodes with water and blot, but do not wipe, with soft tissue;
- . immerse the tips in the buffer solution and take the pH reading, allowing about 1 min for the meter to stabilize. Adjust the standardization control so that the meter reading corresponds to the pH of the known buffer (e.g. 4.0) for the temperature observed. Rinse the electrodes with water and blot with soft tissue. Repeat procedure with fresh portions of buffer solution until the instrument remains in balance on two successive trials. To check the operation of the pH meter, measure the pH of another standard buffer such as one having a pH of 7.0 or check it with freshly prepared 0.025 molar phosphate solution. This buffer is prepared by dissolving 3.387 g potassium dihydrogen phosphate (dried 2 h at 110°C-130°C) in 1 L distilled water. This buffer has a pH of 6.9 at temperatures ranging from 10°C to 30°C and a pH of 6.8 at temperatures ranging from 35°C to 40°C;
- . check the indicating electrodes for proper operation by first using an acid buffer then a base buffer; First standardize the electrodes using a pH 4.0 buffer at or near 25°C. Standardization control should be adjusted so that the meter reads exactly 4.0. Electrodes should be rinsed with water, then blotted and immersed in a pH 9.18 borax buffer. This buffer is prepared by dissolving 3.80 g sodium

borate decahydrate in 1 L freshly distilled water. The buffer should be protected from carbon dioxide and used within 10 min of exposure to the atmosphere. The pH reading should be within ± 0.3 units of the 9.18 value;

- . test the pH meter for proper operation by shorting the glass and reference electrode inputs, thereby reducing the voltage to zero. In some meters this is done by switching the instrument to standby, and in other instruments by using a shorting strap. With the instrument shorted out, standardization control should be turned from one extreme to another. This operation should produce a deflection greater than ± 1.5 pH unit from centre scale;
- . adjust the temperature of the sample to room temperature, and set the temperature compensator control to the observed temperature;
- . rinse and blot the electrodes. Immerse the electrodes in the sample and take the pH reading, allowing 1 min for the meter to stabilize. Rinse and blot the electrodes and repeat on a fresh portion of sample. Oil and grease from the samples may coat the electrodes, therefore it is advisable to clean and standardize the instrument frequently;
- . determine two pH values on the well-mixed sample. These readings should be in agreement with one another to indicate that the sample is homogeneous. Report values to the nearest 0.1 pH unit.

Salt

The concentration of salt in large volumes of simple solutions may be determined by hydrometry but in samples containing other soluble materials or where the sample is available in small volumes only, salt should be determined by titration.

Hydrometry. Hydrometers are hollow glass "spindles" terminating at the lower end in a weighted bulb and having the upper end in the form of a slender stem within which a graduated scale is sealed. When floated in a liquid, a hydrometer sinks to a depth determined by the specific gravity of the liquid. Thus a reading on the hydrometer scale with the surface of the liquid as a reference point gives a measure of the specific gravity of the liquid which, in simple salt solutions, is related to the concentration of salt. Salometer (or salinometer) hydrometers have a scale reading from 0° to 100° , on which 0°Sal is the reading in pure water at 15° and 100°Sal is the reading in a saturated salt solution (26.5%) at the same temperature. Salometer readings are thus approximately equal to percentage sodium chloride by weight multiplied by 4; or 4°Sal is equivalent to 1.06% salt. The reading of the hydrometer must be corrected if it is used at temperatures more than a few degrees from the calibration temperature.

Titration. Weigh about 5 g material to the nearest mg and transfer with 80% ethanol to a 100 ml volumetric flask. Add enough 80% ethanol to give a volume of about 50 ml. Shake well and add 1 ml nitric acid (1 + 4). Add excess of 0.1 silver nitrate solution from a pipette and make up to 100 ml with 80% ethanol. Transfer to a centrifuge and spin down solids. Pipette 50 ml of supernate into a flask, add 2 ml saturated ferric ammonium sulphate and 2 ml nitric acid (1 + 4) and titrate to a permanent light brown colour with 0.1 ammonium thiocyanate.

Results. The percentage of salt in the original sample is given by:

$$\% \text{ salt} = \left[\frac{\text{ml 0.1 N silver nitrate}}{2} - \text{ml 0.1 N ammonium thiocyanate} \right] \times 1.17$$

x W where W is the mass of the sample in g.

Soluble solids (sugar)

The concentration of soluble solids in large volumes of simple syrups may be determined by hydrometry or refractometry. Refractometry is an appropriate method if only small samples are available.

Hydrometry. Hydrometers are hollow glass "spindles" terminating at the lower end in a weighted bulb and having the upper end in the form of a slender stem within which a graduated scale is sealed. When floated in a liquid, a hydrometer sinks to a depth determined by the specific gravity of the liquid. Thus a reading on the hydrometer scale with the surface of the liquid as the reference point gives a measure of the specific gravity of the liquid which, in simple sugar solutions, is related to the concentration of sugar.

Brix (or Balling) hydrometers are calibrated to read directly the percentage by weight of sucrose in pure solutions of sucrose in water, i.e. the number of g of sucrose in 100 g of solution. The density of sugar solutions, and hence the Brix reading from a hydrometer, varies with temperature so hydrometers are available for use at different temperatures.

Refractometry. The refractive index of a solution is related to the soluble solids content. In liquid foods in which the solutes are mainly sugars, refractive index may be used as a measure of soluble solids content, the reading being converted to a percentage of soluble solids, expressed as sucrose, by reference to standard tables.

The refractometric method for estimating solids content has two important advantages over the specific gravity method: it is applicable to less fluid products such as jams, sauces, and pulps that cannot be tested by hydrometry, and it requires only a very small sample. Although refractometers differ in design, all use the critical angle of total reflection to measure refractive index. The observer sees an optical field partly obscured by a shadow with a sharp boundary, the position of which is determined by the refractive index of the sample.

The Abbé refractometer is the type most commonly used. It gives a direct reading of refractive index for the sodium lines (n_d), with an approximate range of 1.3 to 1.7 and precision of ± 0.0002 , corresponding approximately to $\pm 0.1\%$ in soluble solids content expressed as sucrose.

The refractometer consists of a prism system with a water-jacket, a thermometer, a telescope with a compensator to eliminate dispersion and a mirror. The instrument is set up in front of a source of white light, such as a brightly lit window or a suitable bench light, and the mirror is adjusted to give maximum illumination in the optical field. The prisms are opened, and with a glass rod a few drops of the test sample are placed on the lower prism. Only sufficient liquid is required to fill the space (0.10-0.15 mm) between the clamped prisms. The prisms are clamped, the eyepiece is focused sharply on the cross lines, and the shadow on the field prism brought into view by turning the measurement knob. If the shadow shows a coloured

border it is achromatized by turning the graduated ring of the compensator until the edge of the shadow is just turning blue. At this point definition is sharpest, and the boundary is brought into exact coincidence with the junction of the cross lines. The refractive index is then read to four decimal places or the soluble solids content is read directly if the instrument has a Brix scale. The temperature is also read on the thermometer in the instrument as refractive index varies with temperature. Refractometers are usually calibrated for a temperature of 20°C and tables are available setting out temperature corrections for sugar solutions. Temperature control, by circulating water from a constant temperature bath through the refractometer, is especially important when examining samples of hot material. Small hand held refractometers are also used but they often cover only a limited range of concentrations of soluble solids and may not be as accurate as the usual types of Abbé refractometers.

Sorbic acid and sorbates

Sorbic acid and sorbates are estimated calorimetrically after reaction with thiobarbituric acid.

Standard curve of absorbance/concentration. Pipette 5, 10 and 15 ml sorbic acid solution (weigh 134 mg potassium sorbate, equivalent to 100 mg sorbic acid and make up to 1 L with water) into separate 500 ml volumetric flasks and make up to volume with water. Pipette 2 ml samples of each solution and 2 ml water for the blank into separate 15 ml test tubes. Add 1.0 ml 0.3 N sulphuric acid and 1.0 ml potassium dichromate solution (dissolve 147 mg potassium dichromate in water and make up to 100 ml with water) and heat in boiling water for exactly 5 min. Immerse the tubes in ice and add 2 ml thiobarbituric acid solution (dissolve 250 mg thiobarbituric acid in 5 ml 0.5 N sodium hydroxide in 50 ml volumetric flask by swirling under hot water. Add about 20 ml water, neutralize with 3 ml 1 N hydrochloric acid and dilute to volume with water. Make up fresh daily). Heat in boiling water for 10 min. Cool and determine absorbance at 532 nm using matched 10 mm cells and a blank prepared as above starting with 2 ml water. The 2 ml blank and the samples contain 0, 2, 4 and 6 µg sorbic acid.

Procedure. Weigh 1.5-2.0 g sample into a steam distillation flask containing boiling chips. Add 10 ml 2 N sulphuric acid and 10 g hydrated magnesium sulphate. Steam distil to collect 100-125 ml distillate in a 250 ml volumetric flask in about 45 min while maintaining about 20-30 ml in the distillation flask. Rinse the condenser with water and add the rinse water to the volumetric flask. Make up to volume with water.

Pipette 2 ml solution into a 15 ml test tube and proceed as above starting with the addition of 1.0 ml 0.3 N sulphuric acid.

Results. Determine the amount of sorbic acid in the solution from the absorbance curve and calculate the concentration of sorbic acid in the original sample of product using the equation:

$$\text{mg sorbic acid per g product} = \frac{\mu\text{g sorbic acid from curve}}{8 \times \text{mass sample (g)}}$$

The concentration of potassium sorbate is 1.34 multiplied by the figure for sorbic acid.

Sulphur dioxide

Sulphur dioxide is determined by distilling the sample with acid and oxidizing it to sulphuric acid with an excess of hydrogen peroxide; the amount of sulphuric acid is then determined by titration with sodium hydroxide.

Procedure. A sample containing preferably 3-15 mg of sulphur dioxide is placed in the round-bottomed flask in the apparatus shown in Figure 3. Solid samples should be ground or minced.

Connect the apparatus so that all joints are greased and tight. Place 15 ml and 5 ml 3% hydrogen peroxide in the conical flask and the U-tube, respectively, and add water to each if necessary to ensure that bubbles will form in both places.

Add 350 ml water and 20 ml concentrated hydrochloric acid to the sample in the round-bottomed flask. Adjust the flow of nitrogen gas into the round-bottomed flask to give 6-12 bubbles per min in the U-tube. Heat the round-bottomed flask quickly to boil its contents and then reduce the heat to give steady boiling for 30 min; the flow of nitrogen is maintained and the condenser is cooled with tap water throughout this time. Disconnect the conical flask and U-tube and transfer the solution in the latter to the former, washing it with a little water. Add 3 drops bromophenol blue indicator (0.4 g bromophenol blue in 6 ml 0.1 N sodium hydroxide made up to 100 ml) and titrate with 0.05 N sodium hydroxide that was standardized using the same indicator. Titrate the free acid in a further 20 ml of hydrogen peroxide solution as a blank.

Results. The concentration of sulphur dioxide in the sample in mg/kg (or ppm) is given by:

$$\text{Sulphur dioxide (mg/kg)} = \frac{[\text{Sample titration (ml)} - \text{Blank titration (ml)}] \times 1600}{\text{Mass of sample (g)}}$$

4.3 PHYSICAL TESTS

Calibration of thermometers for processing equipment

Some food processing operations, such as the heat sterilization of low-acid canned foods, require temperatures to be measured to an accuracy of at least $\pm 0.5^\circ\text{C}$. Mercury-in-glass thermometers or instruments having at least the same sensitivity and reliability should be used for these measurements. These instruments should be inspected at regular intervals to determine that they are in good working condition and their calibration should be checked at least annually and more often if there is any suspicion that the instrument is not working satisfactorily.

Procedure. Mercury-in-glass thermometers should be inspected for breaks in the mercury column, for leakage of steam through the packing near the base of the thermometer stem and to determine that the stem and scales are firmly fixed. The thermometers should be calibrated against a certified thermometer at a temperature close to the temperature it is intended to measure. With

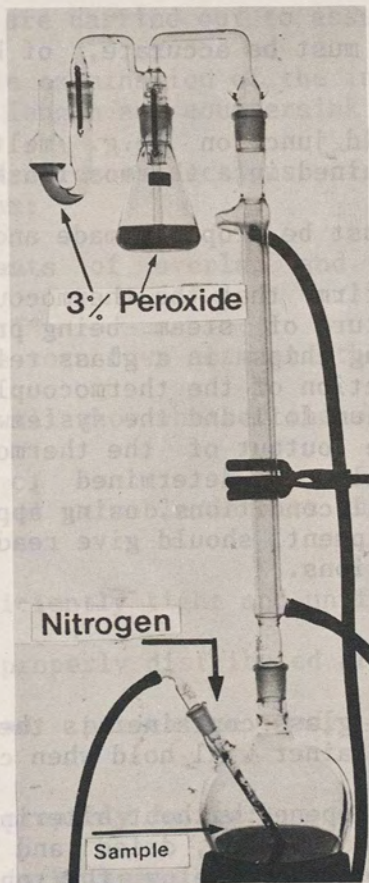


Figure 3. Apparatus used for estimating sulphur dioxide in foods.

retort thermometers it is convenient to mount the certified thermometer close to the thermometers to be calibrated in a lagged manifold which is connected at one end to a retort and which has a bleed cock at the other end to ensure a steady flow of steam past the bulbs of the thermometers. The steam pressure in the retort is brought to an appropriately steady value and the temperature scales in the thermometers under test are adjusted to the value shown by the certified thermometer. The retort thermometers must be carefully handled when they are being removed from the test manifold and reinstalled in the original retort. Thermocouples may also be used to calibrate temperature-measuring equipment under the following conditions.

- . the thermocouple wires must be of high quality, properly insulated and unstrained;
- . The potentiometer must be accurate, of high quality and capable of reading to $\pm 5\mu\text{V}$;
- . the reference cold junction (e.g. melting ice) must be properly prepared and contained in a thermos flask;
- . all connections must be properly made and appropriately protected.

It is prudent to confirm that the thermocouple system is satisfactory by measuring the temperature of steam being produced by gently boiling distilled water with boiling chips in a glass reflux apparatus which is not water cooled. The hot junction of the thermocouple should be surrounded by a radiation shield of aluminium foil and the system should be allowed to reach a steady state before the output of the thermocouple is measured. The barometric pressure must also be determined to find the boiling point of water under the experimental conditions, using appropriate tables. Commonly available thermocouple equipment should give readings accurate to $\pm 0.25^\circ\text{C}$ under good operating conditions.

Capacity of containers

The capacity of a metal or glass container is the volume of distilled water at 20°C that the sealed container will hold when completely filled.

Procedure. Closed cans are opened without altering the height of the double seam and the can is emptied, washed, dried and weighed. The can is then filled to 4.8 mm vertical distance below the top of the can with water at 20°C and again weighed. The difference in mass between the full and empty can in g is equal to the capacity of the can in ml.

The same procedure is used with glass containers but the water is filled to the level of the top of the container.

Drained Mass

The drained mass of a product is determined by weighing the solids retained on a sieve after the liquid component has drained away.

Procedure. Distribute the contents of the container over the meshes of a circular sieve which has been previously weighed or for which a tare has been established. Without shifting the material, incline the sieve to about 20 degrees to facilitate drainage of the liquid. After draining for 2 min determine the mass of material on the sieve.

The sieve should have a square mesh with 11.2 mm openings. For containers having less than 1.5 kg total contents the sieve should be 20 cm dia and for retail containers over 1.5 kg capacity the sieve should be 30 cm dia.

Double Seams

Four types of examination are carried out to assess the quality of a seam:

- . visual and tactile examination of the intact seam and measurement of seam width, seam length and countersink depth;
- . visual inspection of cross-sections of the seam taken at specified points on the seam;
- . optical measurements of overlap and body hook butting in the cross-sections;
- . inspection of the torn-down seam and the body of the can.

These examinations will show whether the seam meets the acceptability criteria which are that:

- . the seam is free of visible defects which are likely to impair the hermetic seal;
- . the seam is sufficiently tight and uniform around the can;
- . the compound is properly distributed around the seam;
- . the overlap and percentage body hook butting are within specifications;
- . the juncture is properly formed;
- . the countersink is at least equal to the seam length.

Definitions. In assessing double seams the following terms are used:

Body hook. Portion of the double seam formed from the turned-back flange of the can body (figure 4).

Body hook butting. Distance occupied by the internal body hook length expressed as a percentage of the internal seam length (figure 5).

Chuck wall. Part of the can end extending from the top of the double seam to the bottom of the countersink (figures 4, 6).

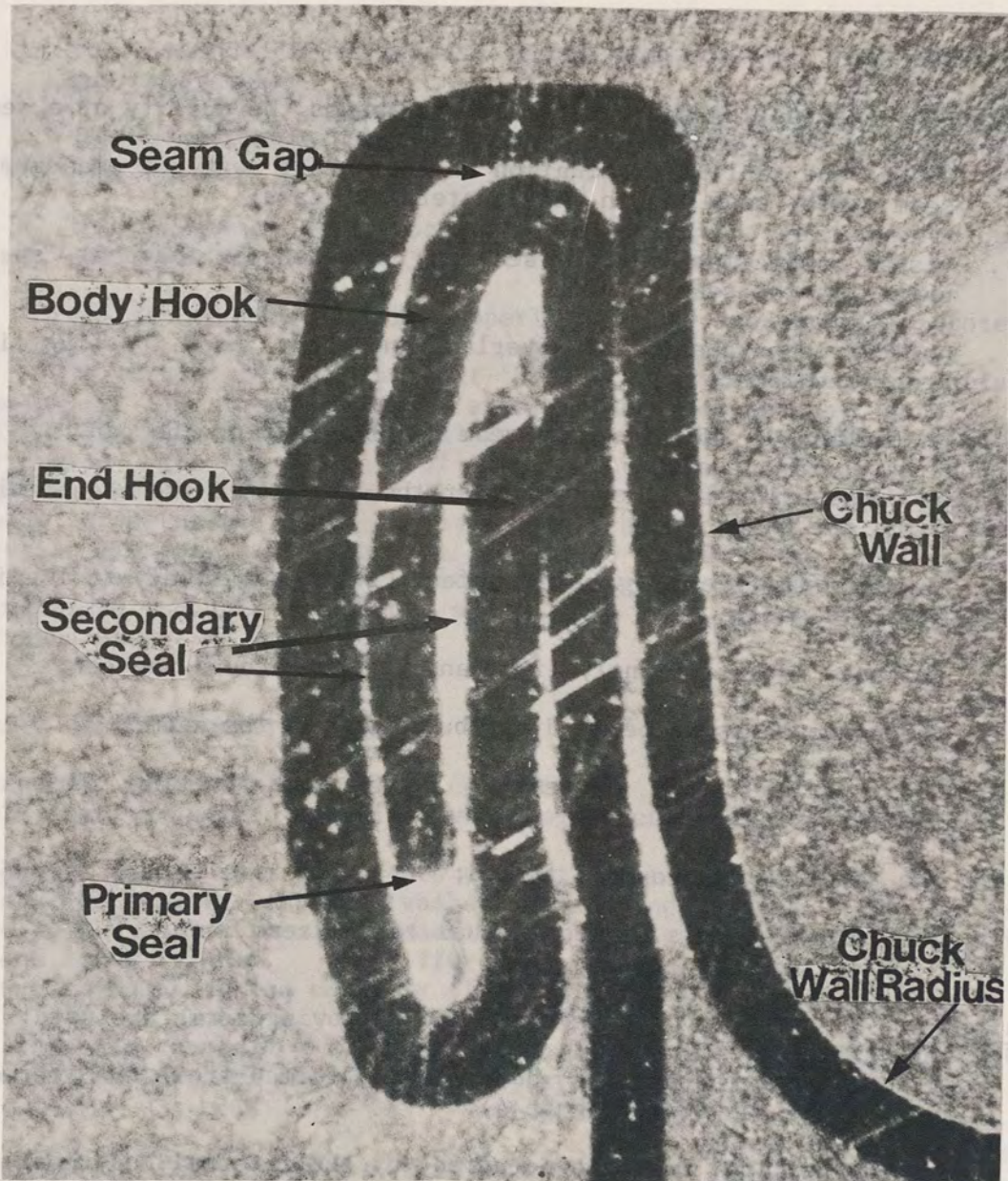


Figure 4. Cross-section of a double seam from a tinplate can; the important components of the seam are identified.

Compartment. Term used to describe the overall interrelationship of those attributes of the assembled seam which determine whether the seal is hermetic.

Compound. Sealing material, either latex or synthetic rubber, placed in the surface of the seam to fill the space between the metal sheets and to form a hermetic seal (the compound is usually applied by a special machine which suits the product).

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elsewhere in the document. It is noted that the compound may be incorporated in the seam by a special machine.

Knock-off. The material which is removed from the body of the can during the process of forming the double seam.

Lap. The distance between the two sheets of metal in the seam.

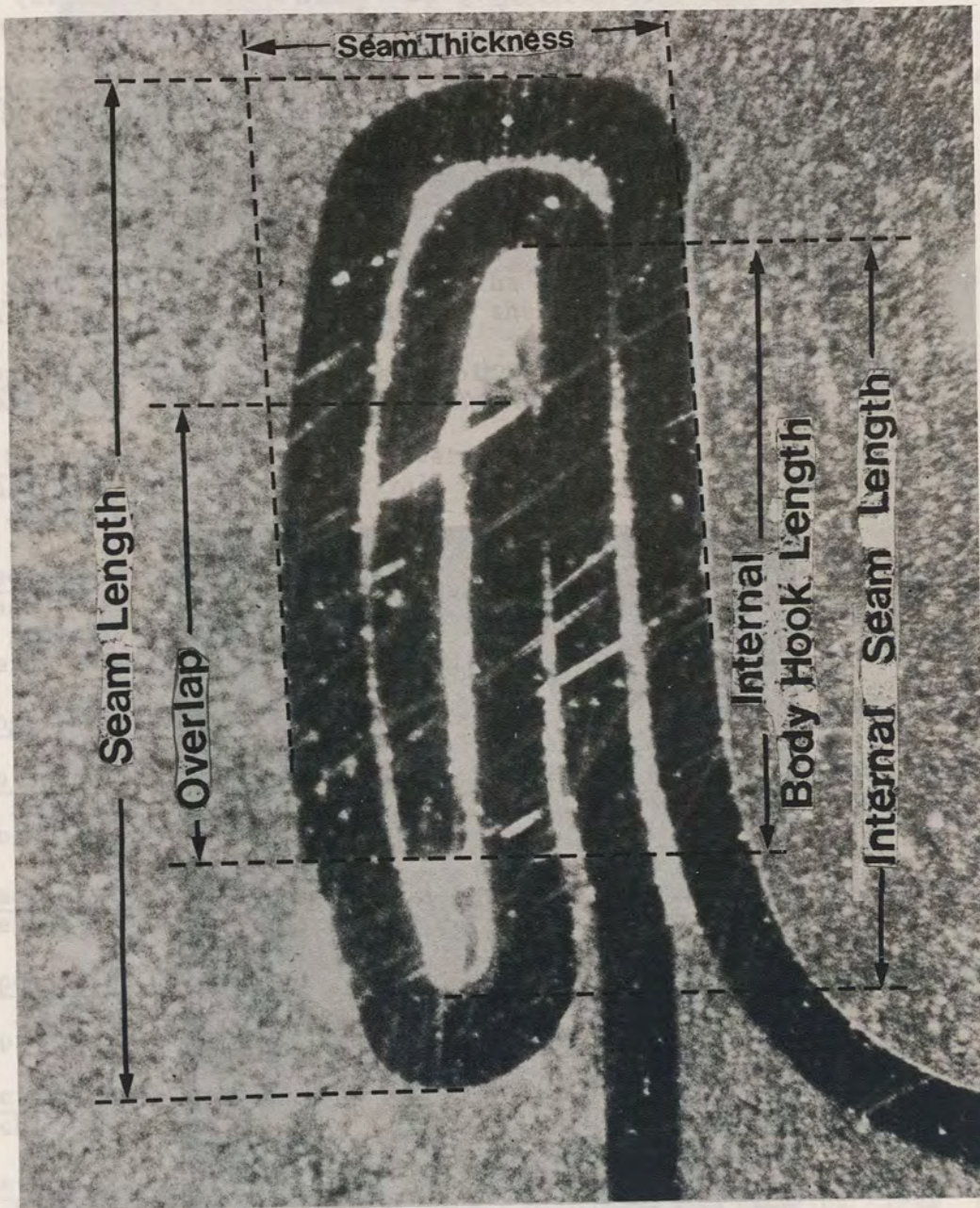


Figure 5. Cross-section of a double seam from a tinplate can. The attributes that contribute to the quality of the seam are marked.

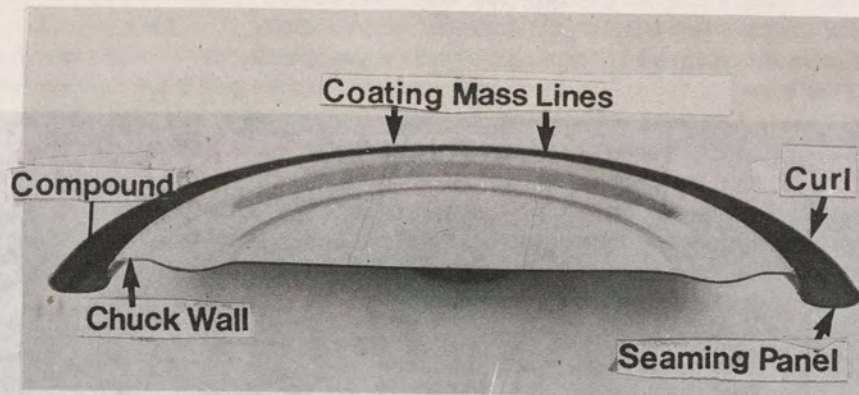


Figure 6. A can end sectioned across the centre to show its component parts.

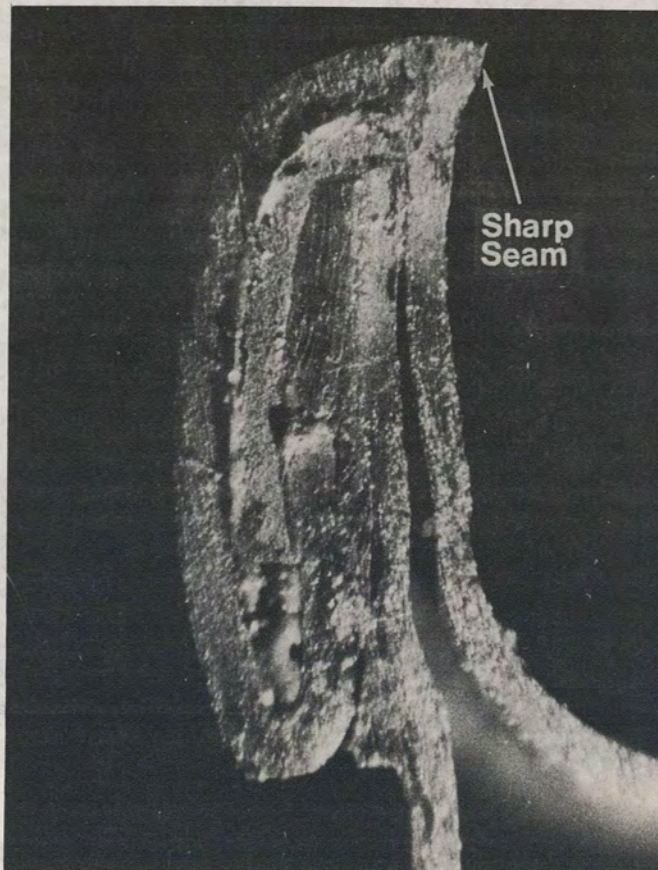


Figure 7. Cross-section of a cut-over or sharp double seam from a tinplate can.

Compactness. Term used to describe the overall interrelationship of those attributes of the assembled seam which determine whether the seal is hermetic.

Compound. Sealing material, either latex or synthetic rubber, placed in the curl and on the seaming panel of the end. It serves to fill spaces within the double seam, and to assist in the formation of an hermetic seal (figure 6). (Note: The composition of the compound must suit the product and thermal process).

Countersink depth. Distance from the top of the double seam to the base of the chuck wall radius (figure 4).

Cut-over. Projection at the inside top edge of the seam which may be associated with fracturing of the end place (figure 7). A cut-over is usually more pronounced at the juncture (see "Sharp seam").

Cut seam. Double seam in which the outer layer of plate is split usually near the end hook radius at the juncture.

Droop. Smooth projection of the double seam below the bottom of a normal seam. It is frequently found at the juncture (figure 8).

End hook (also known as the cover hook). Portion of the end which is turned back between the body wall and the body hook during formation of the double seam (figure 4.)

False seam. Seam or portion of a seam where the end hook has failed to engage (i.e. lock under) the body hook. If the folded body hook does not project below the seam, the false seam may only be detected on careful examination or sectioning (figure 10).

Flange. Curled out open end of the can body (figure 9).

Free space. Difference between the measured thickness of the seam and the sum of the thickness of the five layers of plate present in the double seam.

Hermetic seal. Seal which prevents micro-organisms and other materials entering a can.

Jumped seam. Double seam which is not rolled tightly enough adjacent to the juncture. It is caused by the second operation seaming roll jumping as it passes the juncture.

Juncture (also known as the crossover). Region of the double seam which coincides with the side seam of the can body.

Juncture rating. Measure of the amount of end hook at the juncture which is available for overlap, this amount often being less than elsewhere on the seam because of the extra layers of plate that may be incorporated in the seam at the juncture (figure 11).

Knocked down flange. Form of false seam in which a mislocked portion of the body hook is readily visible.

Lap. Two thicknesses of the body plate bound together at the extremities of the side seam (figure 9).

Compactness. Term used to describe the overall interrelationship of those attributes of the assembled seam which determine whether the seal is hermetic.

Compound. Sealing material, either latex or synthetic rubber, placed in the curved space between the two plates of the seam. The compound is placed within the seam and is not visible until the seal is cut.

Counter. The part of the seam which is opposite to the main body of the seam.

Cut-over. The part of the seam which is cut over the main body of the seam.

Cut seam. The seam which is cut over the main body of the seam.

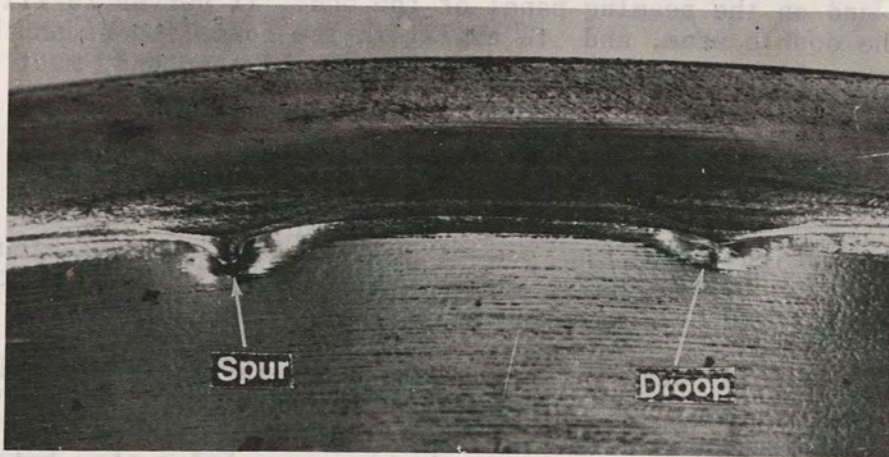


Figure 8. Part of the outside surface of the double seam of a tinplate can showing a spur and droop.

False seam. A seam which is not properly formed and does not engage the body of the can. It is often detected on the outside of the can by the presence of a small gap or by the fact that the seam does not project back between the body wall and the body hook during formation of the double seam (Figure 4).

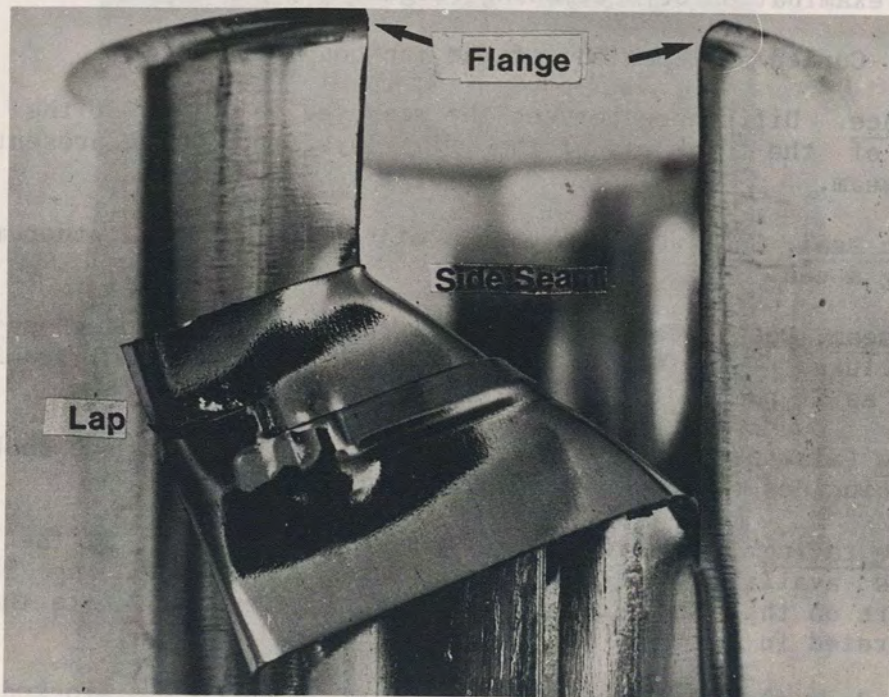


Figure 9. The side seam region of an unusual tinplate can bent downwards to show the flange and lap.



Figure 10. Cross-section of a false double seam from a tinplate can.

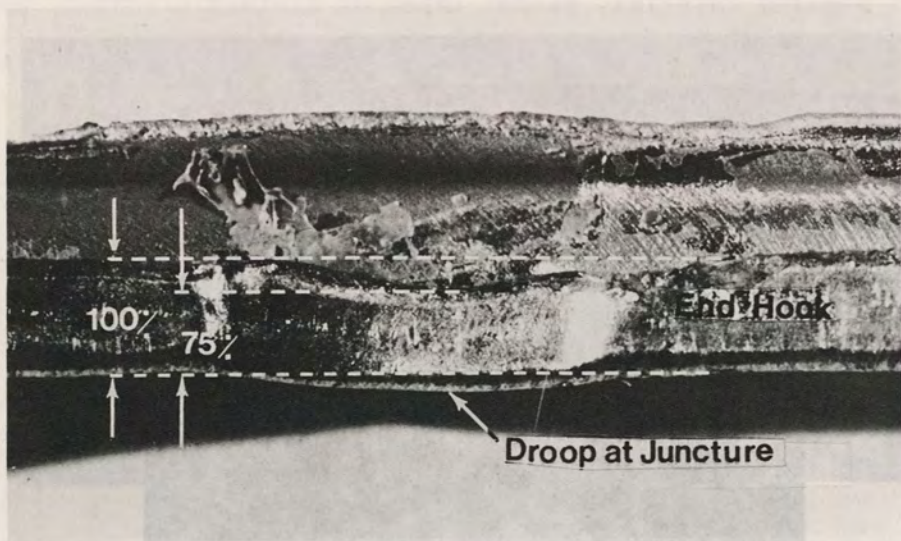


Figure 11. The inside surface of part of the end hook of a double seam showing how the juncture rating is measured.

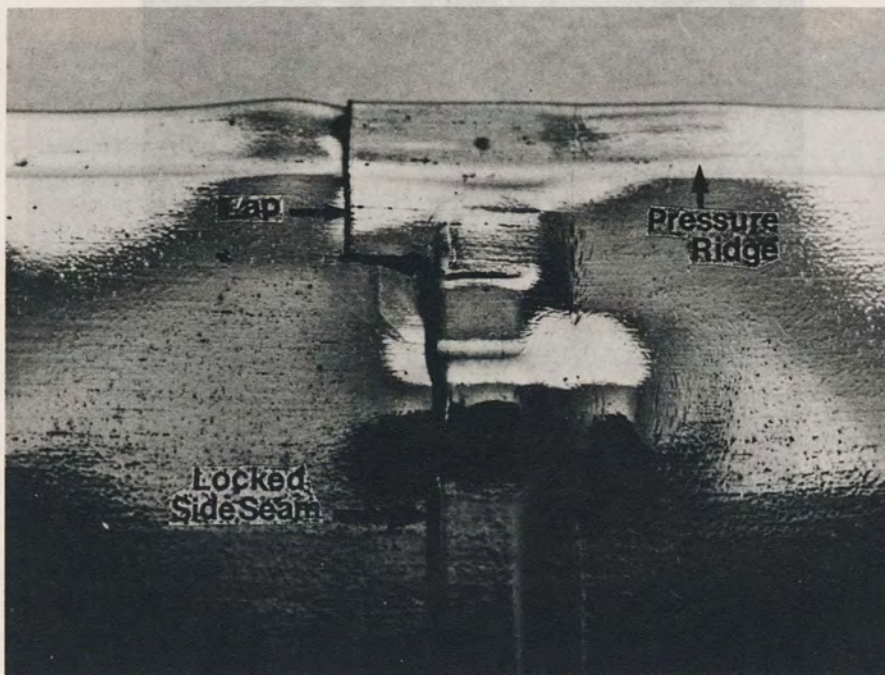


Figure 12. The pressure ridge is exposed on the inside surface of a tinplate can when the double seam is torn down.

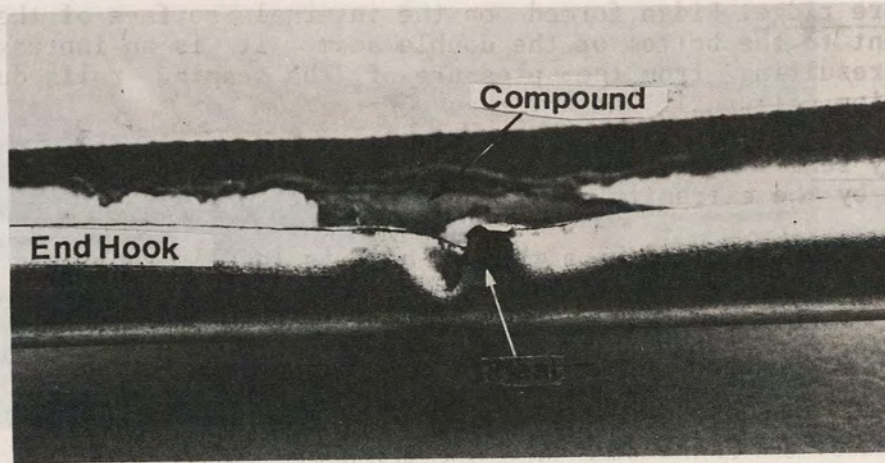


Figure 13. A pleat in the end hook of a double seam from a tinplate.

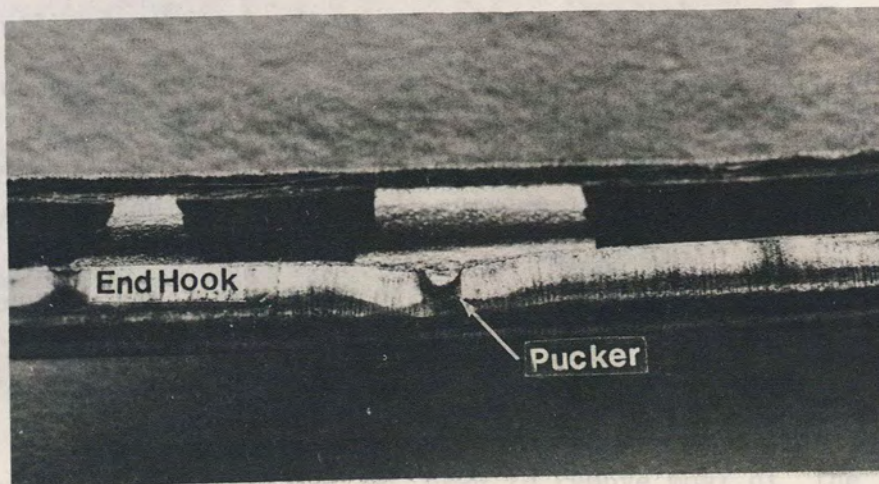


Figure 14. A pucker on the end hook of a double seam from a tinplate can.

Overlap. Length by which the body hook overlaps the end hook (figure 5).

Pleat. Fold in the metal of the end hook. It extends from the cut edge downwards towards the end hook radius and sometimes below this radius in a spur or droop (figure 13).

Pressure ridge. Ridge formed on the internal surface of the can body adjacent to the bottom of the double seam. It is an impression of the chuck resulting from the pressure of the seaming rolls during seam formation (figure 12).

Primary seal. Area where the compound is compressed into the end hook radius by the extremity of the body hook (figure 4).

Pucker. Condition in which the end hook is locally shortened at the cut edge without the hook being pleated (figure 14).

Seam gap. Gap between the body hook and the seaming panel (figure 4).

Seam length. Outside dimension of the seam measured parallel to the axis of the can (figure 5).

Seam thickness. Outside dimension of the seam measured at right angles to the chuck wall (figure 5).

Secondary seals. Areas where the compound is compressed on both sides of the body hook at the overlap (figure 4).

Sharp seam. Small cut-over without fracture (figure 7 and see "Cut-over").

Side seam. Seam formed along the length of the can body (figure 12).

Skidder. Condition caused by slippage between the seaming chuck and end. It is characterized by a loose unflattened area of the seam where the second operation has been incomplete.

Spur. Sharp projection of metal below the double seam (figure 8).

Tightness. The compressive tightness of a curved double seam is assessed by the extent of residual wrinkle in the end hook (figure 15). The tightness rating of a double seam is determined by the unwrinkled length of end hook expressed as a percentage of its total length. It is assessed at the point showing the lowest tightness rating on the end hook.

Wrinkles. During double seaming the diameter of the can end is reduced and the plate in the end hook develops waves or wrinkles which extend from the cut edge of the end hook towards the end hook radius (figure 15).



End Hook



Figure 15. Wrinkles on the end hook of a double seam from a tinplate can.

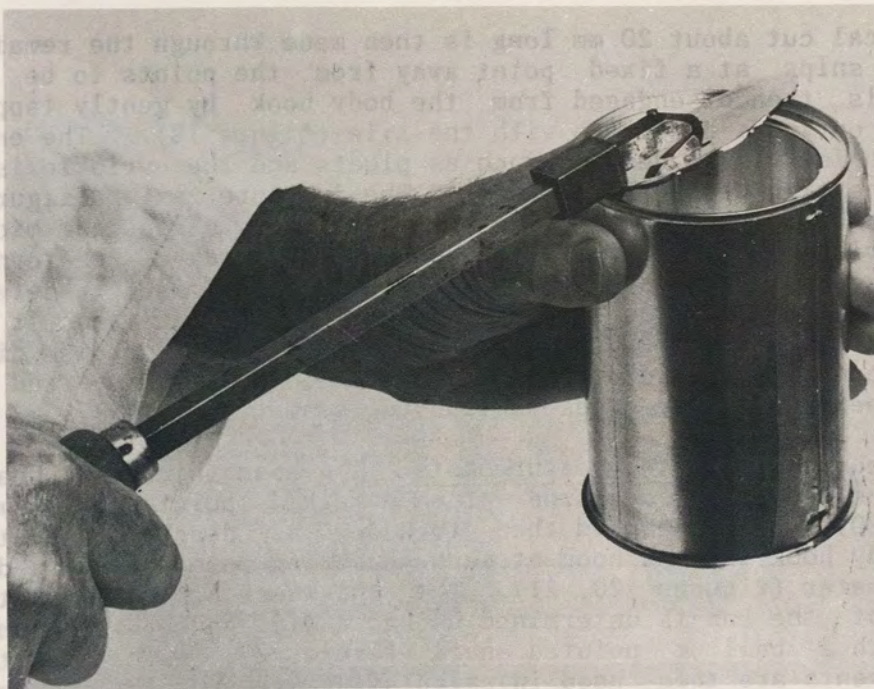


Figure 16. A special cutter is used to remove most of the can end before the double seam is torn down.

Procedure based on optical measurements. Mark the points on the seam where the measurements of seam thickness, seam length, countersink depth, overlap, internal seam length and internal body hook length are to be made. On cylindrical cans the measurement points are usually about 15 mm on each side of the juncture and opposite the side seam.

After measuring the countersink depth with a micrometer or depth gauge the seam is sectioned at the marked points with a high quality metal saw having at least one tooth per mm. The sections should be carefully cut so as to minimize distortion of the components of the seam. The cut surface may be cleaned by rubbing with a coarse eraser, by applying solvent with a stiff short-bristled brush or by immersion in 5% nitric acid for about 20 sec followed by rinsing in water and drying. Additional cleaning or polishing of the cut surface should not be required unless the blade of the seam saw has lost its edge. Extreme care is required if additional polishing of the cross-section is done because the edges of the seam components may become rounded and may not give a true image of the seam for measurement using a microscope or seam projector.

As it may be difficult to tear down a seam after sections have been cut from it another can from the same batch is often taken for that part of the seam examination. The tools required for tearing down a seam are a special cutter, a pair of pliers or nippers, a pair of 200 mm snips and a flat file about 300 mm long. Most of the end plate is removed without disturbing the double seam by using the special cutter (figure 16). The tinplate remaining in the countersink is then removed by tearing the plate along the top of the seam in an upwards and outwards direction with the pliers or nippers (figure 17).

A vertical cut about 20 mm long is then made through the remains of the seam with the snips at a fixed point away from the points to be measured. The end hook is then disengaged from the body hook by gently tapping in a downwards and outwards direction with the file (figure 18). The end hook is then inspected for visual defects such as pleats and the wrinkle is assessed to estimate the tightness of the seam. The juncture rating (figure 11) is also estimated by inspection or by measurements using a can seam microscope. The pressure ridge (figure 12) on the inside of the body of the torn-down can is also assessed and the body hook and end hook are examined to determine the distribution of the compound. In assessing the compound it must be remembered that it will be grossly disturbed when the seam is dismantled. However, the compound should be clearly visible in the primary and secondary seals, in properly prepared cross-sections of seams.

Procedure based on micrometer measurements. The seam thickness, seam length and countersink depth are measured at each marked point with a micrometer (figures 19, 20). The seam is then torn down as described above and the lengths of body hook and end hook at each measuring point are also determined using a micrometer (figures 20, 21). The thickness of the tinplate in the end and body of the can is determined using a dial gauge or a conventional micrometer with a ball or pointed anvil (figure 22). The results of all these measurements are then used in calculating the overlap and percentage body hook butting for each measuring point separately, using the following equations:

$$1. \text{Overlap (mm)} = EH + BH + 1.1 t_e - SL$$



Figure 17. The end plate is pulled away from the top of the double seam in an upwards and outwards direction.



Figure 18. The end hook is disengaged from the body hook by tapping with a file in a downwards and outwards direction.

$$2. \text{ Percentage body hook butting} = \frac{BH - 1.1 t_b}{SL - 1.1 (2t_e + t_b)} \times 100$$

The tightness of the seam may also be measured in terms of free space:

$$3. \text{ Free space (mm)} = ST - (2t_b + 3 t_e)$$

(EH: end hook length; BH: body hook length; t_e is end plate thickness; t_b is body plate thickness; SL is seam length; ST: Seam thickness, and all measurements are in mm.).

Specifications for body hook butting, overlap, tightness, juncture rating and countersink should be supplied by the can manufacturer. However, if that information is not available seams having the following characteristics should be satisfactory:

Body hook butting. 70 per cent minimum for all cans.

<u>Overlap.</u>	Can diameter (mm)	Minimum overlap (mm)
	52	0.75
	58-74	0.90
	83-103	1.00
	126-153	1.15

<u>Tightness:</u>	Can diameter (mm)	Minimum tightness (percent)
	52-65	60
	74-126	70
	153	80

Juncture rating. 50 per cent minimum for all cans.

Countersink. At least equal to the seam length measured at the same point.

Seams that do not conform entirely to the above specifications may still have satisfactory compactness and provide an hermetic seal. Such seams must be referred to an acknowledged expert in double seam technology for further assessment of compactness and integrity.

Equilibrium relative humidity

The equilibrium relative humidity of a food may be measured by determining the change of mass of samples of the food held in atmospheres of known humidity and temperature.

Procedure. Three screw-top jars are used as constant-humidity chambers (figure 23). The humidity in each jar is controlled by a saturated salt solution which fills the jars to a depth of about 20 mm. The humidities produced by saturated solutions of several different salts are given in the following table.



Figure 19. Seam thickness is measured with the micrometer at right angles to the chuck wall of the can.

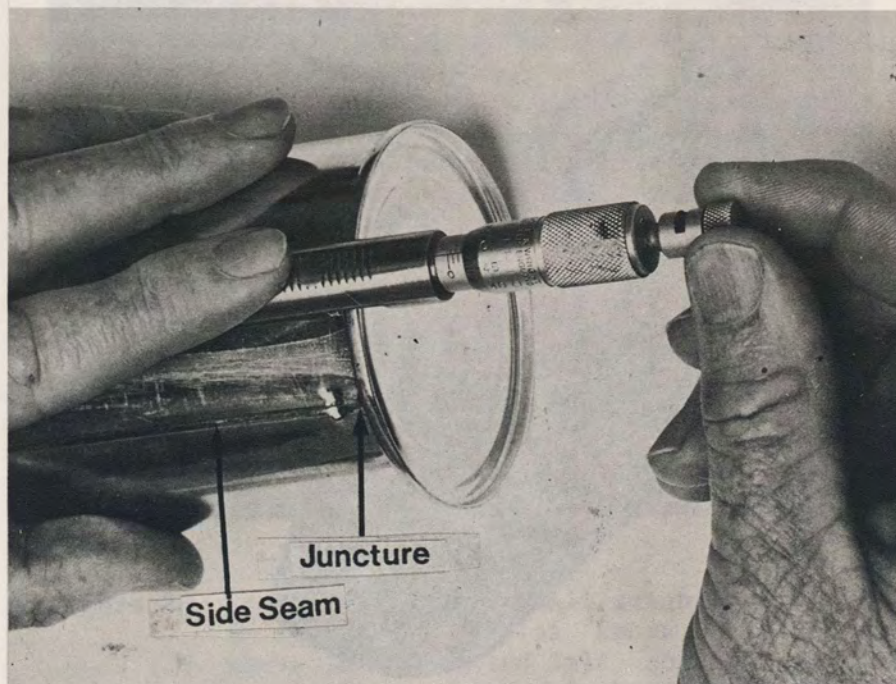


Figure 20. Seam length and body hook length are measured with the micrometer held against the body of the can.

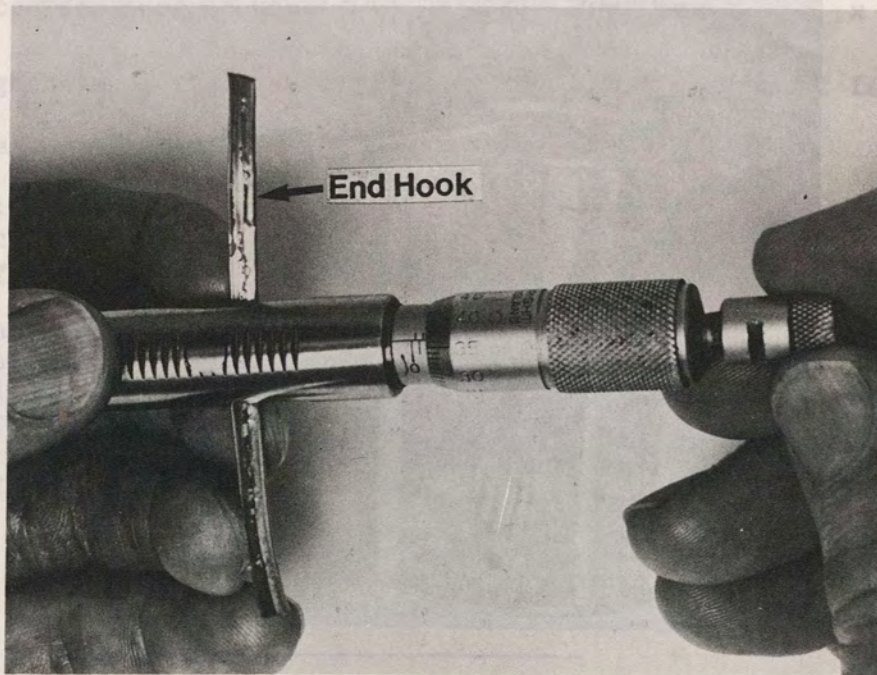


Figure 21. The length of the end hook is measured with a micrometer after the seam is torn down.

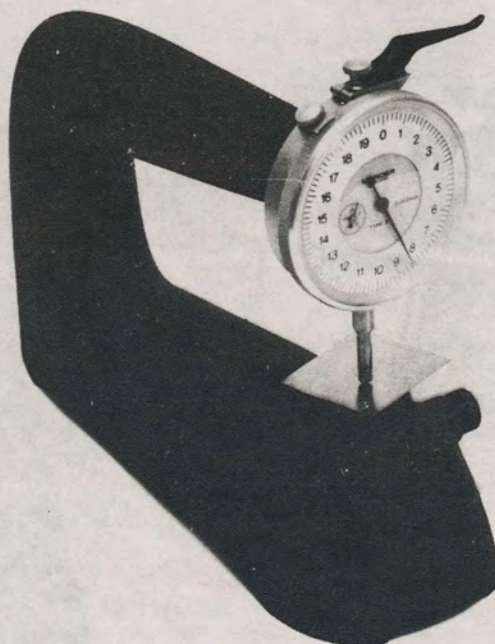


Figure 22. The thickness of tinplate is measured with a dial gauge fitted with a ball probe.

Humidity over saturated solutions

(in percentage)

Relative humidity

The relative humidity is determined by placing a test sample in a desiccator jar over a saturated salt solution. The test sample is placed in a small weighing dish which is held above the saturated salt solution by a wire mesh. The desiccator jar is sealed and held at a well-controlled constant temperature, e.g. 30°C, and the relative humidity is determined by weighing the test sample before and after exposure to the atmosphere of known relative humidity.

Procedure: Weigh a test sample to 0.1 mg. Place the test sample in a small weighing dish. Place the weighing dish in a desiccator jar over a saturated salt solution. Seal the desiccator jar and hold at a constant temperature. Weigh the test sample after exposure to the atmosphere of known relative humidity. Calculate the relative humidity from the change in mass of the test sample.

For the purpose of this test, a desiccator jar is used. The desiccator jar is a glass jar with a lid that has a gasket. The lid is held in place by a clamp. The desiccator jar is used to hold the test sample and the saturated salt solution.

The test sample is placed in a small weighing dish. The weighing dish is placed in the desiccator jar over the saturated salt solution. The desiccator jar is sealed and held at a constant temperature. The relative humidity is determined by weighing the test sample before and after exposure to the atmosphere of known relative humidity.

The test sample is placed in a small weighing dish. The weighing dish is placed in the desiccator jar over the saturated salt solution. The desiccator jar is sealed and held at a constant temperature. The relative humidity is determined by weighing the test sample before and after exposure to the atmosphere of known relative humidity.

Figure 23. The sample in the small weighing dish is exposed to an atmosphere of known relative humidity above the saturated salt solution in the large jar. The subsequent changes in the mass of samples held at different relative humidities are used to estimate the equilibrium relative humidity of the product.

Humidity over saturated solutions

(in percentage)

Temperature (°C)	Magnesium nitrate	Potassium iodide	Sodium chloride	Ammonium sulphate	Potassium chloride	Potassium nitrate
15	55.87	70.98	75.61	81.70	85.92	95.41
20	54.38	69.90	75.47	81.34	85.11	94.62
25	52.89	68.86	75.29	80.99	84.34	93.58
30	51.40	67.89	75.09	80.63	83.62	92.31
35	49.91	66.96	74.87	80.27	82.95	90.79

Approximately equal amounts (2-5 g) of the test product are weighed accurately into three standard-sized dishes, one of which is placed on a support above the saturated salt solution in each jar. The jars are tightly sealed and held at a well-controlled constant temperature, e.g. 30°C, and the dishes are reweighed at intervals.

It is not necessary to wait for the samples to reach equilibrium at the three humidities. A good estimate of the equilibrium value is obtained by plotting the total change of mass of each sample after two days against the relative humidity of the storage atmosphere.

The salt solutions should be selected so that both positive and negative changes in mass are obtained, i.e., at least one salt solution gives a humidity that is greater than the equilibrium relative humidity of the sample and another gives a humidity that is less than the equilibrium relative humidity of the sample. A graph is then drawn of change of mass v humidity and the humidity corresponding to zero change of mass on the graph is taken as the equilibrium relative humidity of the sample. These measurements should be made under conditions of constant temperature; temperature fluctuations great enough to cause condensation on the walls of the jars must be avoided.

Headspace of canned foods

Gross headspace is measured as the distance from the top of the double seam of the can to the headspace surface of the product. Net headspace is the distance from the end of the can to the headspace surface of the product. For most canned foods the net headspace is 4.8 mm less than the gross headspace.

Procedure. Open the can without damaging the double seam and stand the can on a level surface. An engineer's depth gauge having an extended cross-bar is placed across the double seam and the scale is lowered until it just contacts the surface of the product. It is necessary to submerge solid portions of the can contents and to flatten the surface of solid products before the measurements are made. A thin, perforated stainless steel disc, with a diameter slightly less than that of the can is sometimes used for this purpose, a correction being applied to the readings for the displacement

caused by the disc.

Lacquer Adhesion

The adhesion of lacquers to metal cans is tested by determining whether the lacquer can be removed from the metal with commonly available cellulose adhesive tape. The flexibility of the lacquer is also determined by a bend test.

Procedure. Two series of parallel scratches at approximately 2 mm spacing are made at right angles to each other in the lacquer film using a sharp needle or blade. The scratches must penetrate the lacquer film and debris produced during scratching is removed with a soft brush. A length of cellulose adhesive tape is firmly applied to the test area and one end beyond the scratches is left unattached. The sample is then held firmly on a flat surface and the adhesive tape is removed with a sharp upwards movement. The sample is considered to have passed the test if none or only a small amount of lacquer adheres to the tape. As some lacquer is likely to adhere to the tape even when there is satisfactory adhesion it is important to carry out parallel tests on samples which are known to have good adhesion to standardize the test.

For the bend test a coupon of lacquered tinplate or aluminium (50 x 100 mm) is bent through 180° across the smaller dimension round a rod of 3 mm diameter. The lacquered surface shall be towards the outside and it is examined with the unaided eye for cracking or loss of adhesion of the lacquer. The sample is considered to have passed the test if there is no evidence of cracking or loss of adhesion.

Lacquer coating mass

The lacquer coating mass is determined by weighing after separating the lacquer from the metal.

Procedure. A coupon of convenient size (say 50 mm square) is cut from the lacquered metal and weighed. The lacquer is removed from the metal with a suitable solvent system, for example, a mixture of methyl ethyl ketone and chloroform. The coupon is dried and again weighed, the weight loss being equal to the mass of the lacquer.

Some lacquers cannot be readily removed with solvents but they may lift from the metal when the coupon is made the cathode in an electrolytic cell. The cell should contain a solution of 2% sodium carbonate and an anode of stainless steel sheet at least the same area as the test coupon; the cell is connected to a source of about 6 V (DC). Hydrogen produced on the coupon usually lifts the lacquer film from the metal in a minute or so. The coupon is then rinsed, dried and weighed to determine the mass of the lacquer as before. Lacquer films may also be separated from tinplate by standing a cut edge of the coupon in mercury. The mercury amalgamates with the tin and the lacquer may be lifted from the coupon, washed to remove adhering droplets of mercury, dried and weighed to give the lacquer coating mass directly.

The operations involving solvents and mercury should be done in a fume cupboard or in a suitably ventilated space.

Leak Tests

Metal cans are tested for leaks by filling the clean, dry cans with compressed air while they are submerged in water; a steady stream of bubbles issuing from the can indicates the point of leakage.

Procedure. Open one end of the closed can using a suitable cutter so that the end seam is not damaged. Remove the contents, if any, of the can and thoroughly clean the can with a non-ionic detergent (e.g. Teepol) and then rinse. Boil the can in clean water for 1 h, rinse and drain. Dry the can in an air oven for 1 h at 100°C or overnight at 60°C.

Connect the dry can to a controlled supply of clean compressed air and then immerse in clean water. Increase the pressure in the can steadily to the value shown below and examine the external surface of the can for at least 2 min for bubbles of gas escaping from leaks. A leak is indicated by a continuous flow of gas bubbles and should not be confused with occasional bubbles of air which often adhere to the double seams of the can.

Can diameter (mm)	Maximum gauge pressure (kPa)
154	70
99	100
84	170

Flexible and semi-rigid containers are prepared for leak testing by removing their contents through a cross-shaped cut made in a panel of the container away from seams or heat seals. The containers are rinsed and if appropriate they are boiled with a non-ionic detergent for 1 h and again rinsed. The containers are dried for 1 h at 100°C or overnight at 60°C and the cut is repaired with a air-setting silicone rubber cement. A hypodermic needle connected to a controlled supply of clean compressed air is then pushed through the silicone rubber seal. The containers are then pressurized to about 10 kPa and examined for escaping air while held under water.

Net mass

Net mass is determined by weighing the intact container of the product and subtracting the mass of the clean, dry, empty container and closure.

Procedure. The gross mass of containers of the product weighing up to 2 kg should be determined to ± 0.25 g and for large retail containers the gross mass should be determined to ± 1.0 g. The mass of the empty container and closure should be determined using the same balance. Ice crystals and moisture on the outside of frozen products should be removed with a towel before the gross mass is determined. The mass of the container is subtracted from the gross mass to give the net mass.

Oxygen permeability

The oxygen permeability of flexible packaging films is measured by exposing one side of the film to a glass stream containing a known concentration of oxygen and the other side to an oxygen-free atmosphere. The rate of increase of concentration of oxygen in the gas that was initially oxygen-free is measured and those data are used to calculate the permeability of the sample.

Equipment. The test cell comprises two cylindrical compartments between which the test sample is held. The cell and its fittings are made from stainless steel and the clamping device from mild steel (figure 24). Each of the two cell compartments is fitted with a gas sampling port, two ball valves and a support for the film under test. Each support consists of concentric circles of wire fixed to four radial arms which are positioned inside the compartments in order to ensure that the sample of film remains flat. The sealing surface of the lower compartment is flat, whereas the other surface is rounded to a radius of 2 mm. Both surfaces are accurately machined and polished so that when a sample of film is clamped between the surfaces, the force from the clamping device is applied to a narrow circular area on both sides of the sample and gives an effective seal.

The two compartments of the cell have volumes of about 1000 ml and 500 ml. Another component of the unit is a stainless steel disc which may be placed in the lower compartment to reduce its volume to about 130 ml. The permeability of the sample to be measured determines which of the three volumes (1000, 500 or 130 ml) is used for the measuring compartment.

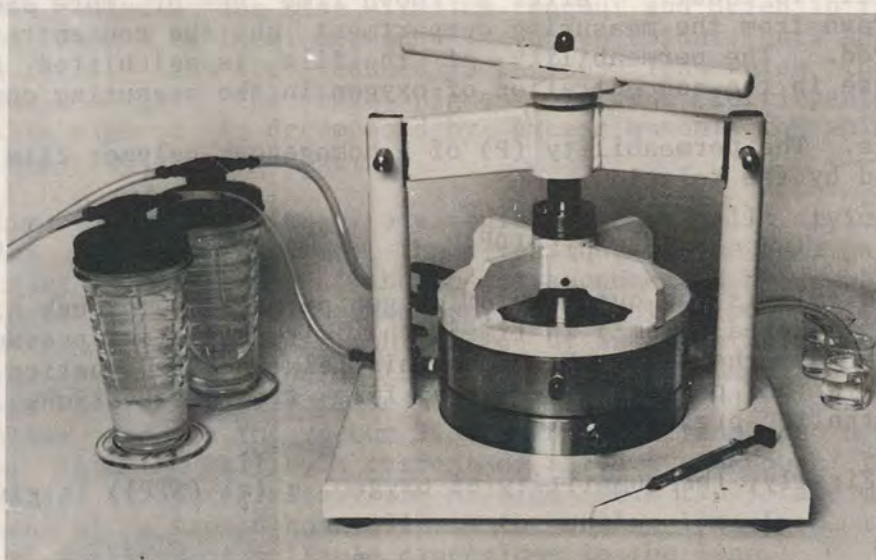


Figure 24. Equipment used for measuring the oxygen permeability of flexible packaging films.

Calibration. The volume of each compartment is determined by sealing a laminate of aluminium foil and polyethylene in the cell and flushing the compartments with nitrogen. The residual oxygen content of the nitrogen is measured by analysing samples of the atmosphere from the cell by gas chromatography. The inlet and outlet valves of the cell are then closed, and a known volume of nitrogen is withdrawn from each compartment and replaced by the same volume of oxygen. After an equilibration period of 1 h, the oxygen contents in the cell are redetermined. The volume (V) of each compartment is calculated from the relation:

$$V = (V_a \times 100) / (O_1 - O_0), \quad (1)$$

where V_a is the volume of oxygen added and O_0 and O_1 are respectively the percentages of oxygen before and after the addition.

Procedure. With the film to be tested clamped between the compartments, one compartment is flushed with oxygen and the measuring compartment is flushed with an inert gas such as nitrogen. The relative humidity of the gas streams may be controlled by placing desiccants or saturated salt solutions in the gas stream before the inlet cocks to the cell. The outlet lines are immersed to a depth of about 2 cm in a beaker of water, thereby avoiding back diffusion of air into the cell.

At a flow rate of 75 ml/min the cell may be flushed free of air in less than 30 min, but with hydrophilic films it is important to continue the flushing operation until the moisture content of the film has reached equilibrium with respect to the test conditions. The time taken to reach equilibrium can only be determined by repeating the determination of permeability on the same sample until constant results are obtained.

After the concentration of residual oxygen in the measuring compartment has been measured, the gas flows from the supply cylinders are stopped and the inlet and outlet cocks of the cell are turned off, in that order. This procedure ensures that the pressure of the two gases in each compartment of the cell is equal. After a known time one or more samples of gas are withdrawn from the measuring compartment and the concentration of oxygen is measured. The permeability of the film is calculated from the observed increase in the concentration of oxygen in the measuring compartment.

Results. The permeability (P) of a homogenous polymer film to oxygen may be defined by the relation:

$$P = ql/At\Delta P, \quad (2)$$

where q (ml) is the quantity of oxygen permeating through a film of thickness l (μ m) and area A (m^2) in time t (h) with a partial pressure difference ΔP (kPa) across the film. Based on this relation, an equation may be derived to calculate the permeability of a film from observations made, using the concentration-pressure method.

Firstly, the quantity of oxygen, q (ml (STP)) is given by:

$$q = \frac{V}{100} (G_1 - G_0) \frac{P_a}{101.3} \times \frac{273}{\tau} \quad (3)$$

where V (ml) is the volume of the measuring compartment of the cell, G_0 and G_1 (%) are the initial and final concentrations of oxygen in the measuring

compartment, P_a (kPa) is the atmospheric pressure, and τ ($^{\circ}\text{K}$) is the test temperature.

For cases when $G_1 - G_0$ is small, i.e. less than say 1%, the mean partial pressure, ΔP in kPa of the test gas across the film sample is given by:

$$\Delta P = 1/2 \frac{(P_a G_c - P_a G_0 + P_a G_c - P_a G_1)}{100} \quad (4)$$

where G_c (%) is the concentration of the oxygen supplied to the cell.

Simplifying (3) and (4) and substituting in equation (2) gives:

$$P = \frac{5.390 V l (G_1 - G_0)}{A \tau (2G_c - G_1 - G_0)} \quad (5)$$

in the units $\text{ml (STP)} \times \mu\text{m} \times \text{m}^{-2} \times \text{h}^{-1} \times \text{kPa}^{-1}$. With heterogeneous materials, such as laminates and coated films, it is not valid to calculate the permeability for a unit thickness, so the thickness term is omitted. The calculated value then becomes a transmission rate which is expressed in the units $\text{ml (STP)} \times \text{m}^{-2} \times \text{h}^{-1} \times \text{kPa}^{-1}$ together with a description of the composition of the test material.

Pinhole test for packaging films

A paper coated with an ammonia-sensitive reagent is used to detect the presence and position of pinholes in packaging films.

Equipment. The ammonia-sensitive reagent is prepared by dissolving 100 g tartaric acid in 600 ml water and adding 15 ml 60% ferric chloride, 120 ml 20% ammonia in water and 500 ml 12% potassium ferricyanide. This solution is stable for several weeks when stored in the dark. Sheets of white, semi-absorbent paper are coated on one side with the reagent and dried in the presence of light. The sheets at first are an olive-green colour, but upon exposure to light the ferric ions are reduced to ferrous ions which then react with ferricyanide to give the blue pigment ferrous ferricyanide (Turnbull's blue). This pigment is decomposed by excess ammonia to white ferrous hydroxide and ammonium ferricyanide.

The apparatus (figure 25) consists of a circular Perspex cell divided into two compartments by the test specimen; the dimensions of the cell are not critical. The outlet from the top of the cell is connected by rubber tubing and glass T-pieces to a mercury manometer and a water pump. The vacuum applied to the top compartment is controlled by an adjustable hose clip downstream from the manometer. The bottom compartment is connected on one side to a Drechsel bottle partly filled with concentrated ammonium hydroxide and on the other side to the vacuum line downstream from the hose clip. Ammonia is swept into the cell by a current of air drawn through the ammonium hydroxide solution at approximately 150 ml/min. This flow rate may be achieved by adjustment of a second hose clip on the outlet line from the cell, or by inserting a capillary of suitable dimensions in the line.

The test specimen is placed on the flange of the cell and covered with the moistened reagent paper so that the reagent-coated side faces the

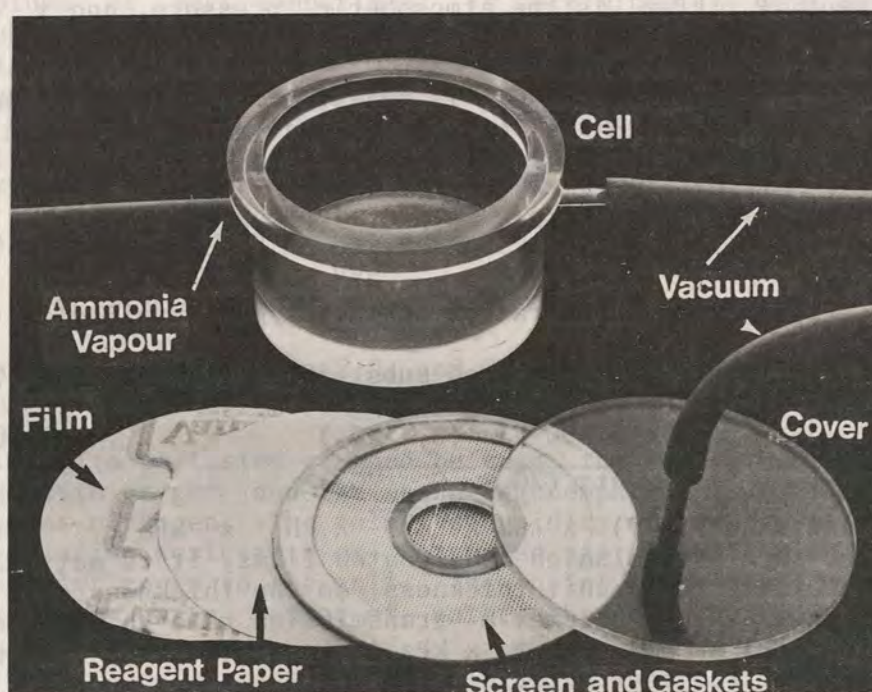


Figure 25. Apparatus for detecting pinholes in flexible packaging materials. The components in the foreground are placed in order from the left (the sample under test, the moistened reagent paper, support screen and gaskets and top cover) onto the cell, and ammonia vapour is drawn through the system by applying a vacuum to the tubes leading to the right.

specimen. With coated materials, the coating on the specimen must face the reagent paper. A neoprene gasket is positioned on the paper, and a metal screen having a diameter approximately 2 mm less than the internal diameter of the cell and gasket is fitted. The screen is used to prevent the moistened paper from sealing on to the flat surface of the cell cover when a vacuum is applied. To prevent distortion of the specimen, the thicknesses of the screen and gasket should be similar. The cell cover is then fitted and clamped by four spring-back paper clips. With the water pump turned on, the hose clip is adjusted to give a gauge pressure of about minus 70 kPa (approximately 20 cm Hg) in the top of the cell. The tubing to the ammonia supply is then connected and ammonia drawn through the bottom of the cell. At the end of the test period, the ammonia line is disconnected and air drawn through the cell for approximately 30 sec to remove residual ammonia. The cell is then dismantled and the sensitized paper examined for white spots, which indicate discontinuities in the test specimen.

The time required for a test at a specific vacuum level depends on the nature of the film material; it is usually in the range 15-240 sec. The optimum test time is one that gives small, sharply defined spots on the sensitized paper, and it should be pre-determined for each type of test material. Test times that are inconveniently short or long may be adjusted by decreasing or increasing, respectively, the vacuum on the top of the cell. However, when long test times are used, ammonia may permeate through an organic film free of pores by the solution-diffusion mechanism, resulting in a uniform discoloration of the reagent paper.

Temperature of frozen foods

The temperature of frozen foods should be measured with a battery-powered instrument which uses a resistance thermometer or a thermocouple to sense temperature.

Equipment. The temperature measuring instrument should comply with the following specifications:

- . it should be accurate to $\pm 0.5^{\circ}\text{C}$ over the range -30°C to 30°C and it should be sensitive to changes of 0.5°C ;
- . the accuracy of the measurements should not be affected by the temperature of the surroundings;
- . the scale should be readable to at least 0.5°C ;
- . the half-value period, i.e. the time needed for the reading to change from the initial temperature halfway to the final temperature, should not exceed 0.5 min;
- . the sensitive part of the measuring probe should be constructed so that it makes good thermal contact with the product;
- . the equipment should be protected from moisture and other undesirable factors; and,
- . it should contain a device for checking the battery voltage to indicate when replacement or recharging is required.

A sharp pointed metal instrument, which can be easily cleaned, is required to make holes in the product for insertion of the temperature measuring probe; the holes should be just larger in diameter than the probe. Hand operated wood drills are suitable.

The calibration of the temperature measuring equipment should be checked in melting ice (0°C) and in salt/ice mixtures at lower temperatures. In the latter case the temperature produced by the salt/ice mixture should be measured with a standard thermometer. The melting ice and the salt/ice mixture should be held in a thermos and temperature readings should be delayed at least 5 min after the thermometers are placed in the thermos to allow a steady state to be attained. The calibration should be carried out with the circuit and power supply of the temperature measuring instrument exposed to the range of temperatures likely to be encountered in practice, say ambient, 0°C and -18°C .

Procedure. Temperature may be measured in the product, between individual containers or in the air in cold rooms and refrigerated transport systems. The probe should be shielded from extraneous radiation when air temperatures are being measured. It is difficult to obtain accurate measurements of temperature of the exposed surface of packages and these measurements should not be attempted with the equipment described here. Internal product temperatures should be measured at a point 25 mm below the centre of the largest surface whenever possible. The tool used for making the hole in the product for the probe should be pre-cooled and the hole should be at least 50 mm deep to ensure that as much of the probe as practicable is in the product. The measurements should involve as little disturbance of the product and

storage conditions as possible. The temperature between packages should be measured by inserting the probe as deeply as possible into the load. In all instances the temperature is recorded after it reaches a steady value.

Tin coating mass

The tin coating mass on tinplate may be determined gravimetrically after removing the coating from one or both surfaces of a specimen of plate of known area using acidified antimony trioxide.

Procedure. The specimen, of known area, usually 50-100 cm², is cleaned with a solvent or by cathodic treatment in 1% sodium carbonate for 1-2 min at about 6 V. The specimen is then rinsed and dried. If the coating mass on one surface only is to be determined the other surface is protected with a brushing lacquer. The specimen is then weighed to the nearest mg or less and the exposed tin coating and the tin-iron alloy is removed from the base steel by immersing the specimen in a solution of 20 g antimony trioxide in 1 L concentrated hydrochloric acid. The specimen is allowed to remain in the acid for about 1 min after gas evolution has ceased; it is then removed, washed immediately in running water and the loosely adherent deposit of antimony is removed by mopping with a soft cotton wool swab or paper tissue. The specimen is then dried and weighed again.

Results. For matt-finish electrolytic tinplate the weight of tin coating is calculated directly but for bright finish electrolytic plate and hot-dipped plate a correction should be made for the amount of iron in the tin-iron alloy layer. A correction of minus 1.4 g/m² is appropriate for most hot-dipped tinplates and minus 0.5 g/m² is appropriate for most bright electrolytic tinplates.

Vacuum

The vacuum in rigid containers such as metal cans and glass jars is measured with a Bourdan tube vacuum gauge.

Procedure. The vacuum gauge is fitted with a 3 mm dia hypodermic needle which is used to pierce the can or the closure of the glass jar. The needle is surrounded by a flexible rubber skirt which forms an air tight seal as the point of the needle is being forced into the container. The contact surface of the rubber skirt should be moistened before the measurement to assist in obtaining a satisfactory seal. The gauge should be applied towards the edge of the can end or the closure of the glass container to minimize the risk of altering the headspace volume and hence the vacuum in the container. Some commercially available vacuum gauges are fitted with a device which compensates for the air in the gauge as it reduces the true vacuum in the container during the measurement.

Vacuum gauges should be calibrated from 0 to minus 100 kPa (750 mm Hg) in intervals of 5 kPa. The vacuum is reported as a "Gauge pressure of minus a number of kPa" or as "mm Hg".

Washed, drained mass

The washed, drained mass of products which comprise solid particles in a thick sauce or gravy is determined by weighing the solids retained on a fine

sieve after the covering material is washed away with hot water.

Procedures. Wash the contents of the container on to a sieve which has been previously weighed or for which a tare has been established. The sieve should have a square mesh with openings of 0.30 mm and should be 20 cm diameter. The material on the sieve is washed with running cold water and then hot water until the retained solids are free of adhering substance. The solids are then spread over the sieve, allowed to drain for 5 min and the total mass is determined. The drained mass is then determined by subtracting the mass of the sieve.

Water vapour transmission rate

The water vapour transmission rate of flexible packaging films and packages is determined by weighing the amount of water which diffuses through a known area of the film in a specified time and under the influence of a known water vapour pressure gradient at a specified temperature.

Procedure. For measurements at temperatures above 20°C a circular sample of the material under test is sealed with wax across the opening of an aluminium dish containing a desiccant such as calcium chloride granules; the assembly is then stored under controlled conditions of temperature and relative humidity and the uptake of moisture followed gravimetrically. The temperature of the test system is controlled with a thermostat, the humidity is controlled with saturated salt solutions (see "Equilibrium relative humidity", p. 46) and the storage atmosphere is stirred by an electric fan. For measurements at temperatures below 0°C the aluminium dish is stored in stirred air over ice in a sealed metal box in a constant temperature room. Specially formulated wax may be needed to seal satisfactorily the circular sample of film to the aluminium dish for measurements at low temperatures.

The water vapour transmission rate of packages is determined gravimetrically by sealing a desiccant in the test package.

Results. The water vapour transmission rate is given by:

$$\frac{q}{A t}$$

where q is mass of water diffusing through a film of area A in time t where the relative humidity on each side of the film, the temperature and the type and thickness of the film are specified. The commonly used units are $\text{g/m}^2/24 \text{ h}$ for films and $\text{g}/24 \text{ h}$ for packages. The need to specify the relative humidity on each side of the film arises because the permeability of hydrophilic materials varies markedly with humidity.

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ANNEX

STORAGE LIFE OF FRESH FRUITS AND VEGETABLES INTENDED FOR PROCESSING

In addition to the temperature, humidity and composition of the storage atmosphere, the storage life of fresh fruits and vegetables is influenced by many factors including variety, maturity, growing conditions and the methods of harvesting and handling. The following table gives what are widely accepted as conservative estimates of the storage life of many common fruits and vegetables at their best storage temperature. The items are listed in alphabetical order and comments on special aspects of the storage of some items are given as footnotes to the table.

PRODUCT	TEMPERATURE (°C)	Storage Life
Apples	-1 to 3 ^d .	8 weeks
Apricots	-0.5	2 weeks
Asparagus	0	2 days
Avocados	10	1 week
Bananas (green)	12.5	2 weeks
Beans (green)	7	2 days
Beetroot (topped)	0	12 weeks
Berry fruits	-0.5	2 days
Broccoli	0	2 days
Brussels sprouts	0	2 days
Cabbage (Chinese)	0	1 week
Cabbage (late varieties)	0	6 weeks
Cabbage (except large late)	0	4 weeks
Carrots (topped)	0	12 weeks
Cauliflower	0	2 days
Celery	0	6 days
Cherries	1	2 weeks
Coconuts	0	8 weeks

Corn (immature)	-0.5	1 day
Cucumbers	7-10	2 weeks
Custard apples	7	2 weeks
Figs	-1	2 weeks
Grapefruit	10 to 12	10 weeks
Grapes (most varieties)	-0.5	4 weeks
Grapes (some late varieties)	-1	7 weeks
Guavas	7 to 10	2 weeks
Jackfruit	12	3 weeks
Lemons	12	12 weeks
Lettuce	0	2 days
Limes	10	6 weeks
Loquat	1	1 week
Longan	12	1 week
Lychee	2	4 weeks
Mandarins	5 to 7 ^a .	3 weeks
Mangoes	10 to 12	2 weeks
Mangosteen	5	2 weeks
Marrows	10 to 12	6 weeks
Melons (water, cantaloup)	5 to 7	2 weeks
Melons (honeydew, casaba)	7 to 10	4 weeks
Mushrooms	0	1 week
Nectarines	-0.5 ^b .	4 weeks
Onions (early varieties)	0 ^c .	6 weeks
Onions (later varieties)	0 ^c .	12 weeks
Oranges	5 to 7	6 weeks
Papaya (Papaws)	7	2 weeks

Parsnips (topped)	0	12 weeks
Passionfruit	7	3 weeks
Peas (green)	-0.5	1 week
Peas (green, mechanically harvested)	0 to 2	8 hours
Peaches	-0.5 ^b .	2 weeks
Pears	-1 ^e .	8 weeks
Peppers (sweet), capsicums	7	2 weeks
Persimmons (to ripen slowly)	10 to 12	6 weeks
Pineapples	10	2 weeks
Plums	-0.5 ^b .	2 weeks
Potatoes	7	16 weeks
Pumpkins	10 to 12 ^c .	12 weeks
Quinces	0	8 weeks
Radish (topped)	0	2 weeks
Rambutan	10	1 week
Rhubarb	0	2 weeks
Silver beet	0	1 week
Spinach	0	1 week
Squash	10 to 12	6 weeks
Swede Turnips	0	16 weeks
Sweet potatoes	12	16 weeks
Tomatoes (coloured)	7 to 10	1 week
Tomatoes (mature green)	10	3 weeks

-
- a. According to variety.
 - b. Very perishable at higher temperatures.
 - c. Dry air, relative humidity 70 to 75%.
 - d. Depending on variety and climate.
 - e. Very perishable at temperatures above 10°C.

12 weeks	0	Parsnips (topped)
(autumn) 12 weeks	1	Parsnips (topped)
3 weeks	7	Parsnips (topped)
1 week	01-7	Parsnips (topped)
8 hours	0 to 2	Parsnips (topped)
2 weeks	1	Parsnips (topped)
2 weeks	10-21	Parsnips (topped)
2 weeks	-1-5-0	Parsnips (topped)
2 weeks	7	Parsnips (topped)
2 weeks	10 to 12	Parsnips (topped)
2 weeks	10	Parsnips (topped)
2 weeks	-0-5-21	Parsnips (topped)
10 weeks	7	Parsnips (topped)
12 weeks	10 to 12	Parsnips (topped)
8 weeks	0	Parsnips (topped)
2 weeks	0	Parsnips (topped)
1 week	10	Parsnips (topped)
2 weeks	0-7 to 5	Parsnips (topped)
1 week	91 to 91	Parsnips (topped)
1 week	0	Parsnips (topped)
6 weeks	10 to 12	Parsnips (topped)
10 weeks	0 to 5	Parsnips (topped)
10 weeks	12 to 1	Parsnips (topped)
1 week	7 to 10	Parsnips (topped)
2 weeks	10 to 12	Parsnips (topped)

- a. According to variety.
 b. Very perishable at higher temperatures.
 c. Dry air, relative humidity 70 to 75%.
 d. Depending on variety and climate.
 e. Very perishable at temperatures above 10°C.

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