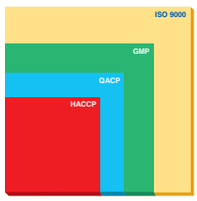


Long-Life Products:

Heat-Treated, Aseptically Packed:

A Guide to Quality

Long-Life Products:
Heat-Treated,
Aseptically Packed:
A Guide to Quality

Publisher: Dr. Bernhard von Bockelmann, Linnévägen 4, SE-232 52 Åkarp, Sweden.

ISBN 91-630-6695-5

Printed by Fälth & Hässler, Värnamo, Sweden

The intention of this book is to present some of the knowledge acquired during many years of practical work on the quality control and quality assurance of long-life products. Different factors have an impact on product quality – the standard of the raw materials, the equipment involved, the processes applied, operational procedures, installation, and so on. In order to produce a high quality product, a thorough understanding of these factors is necessary. An attempt has been made to discuss and present the material in a comprehensible way concentrating on microbiological aspects. The HACCP (QACP) concept has been used as a model for process control.

UHT-treated and aseptically packaged products have been produced in large quantities for quite some time. So far, the literature available concentrates on particular aspects of product and equipment characteristics and the technology involved. A more general presentation appears to be lacking. Hopefully, our work will help close the gap.

To a large extent, the available "old" literature has been consulted. The new literature is readily accessible through data profile searches. In addition, more than 25 years of practical field experience has gone into this study.

The different topics are arranged in such a way that each chapter stands as an independent entity. Of necessity, this has led to some repetitions. We hope that the reader will accept the problem and make allowances for the consequences. It is also our hope that this study will contribute to the production of long-life products with a high level of quality.

Finally, we would like to express our appreciation to all the Tetra Pak people who have helped to prepare this book and who have contributed with valuable comments on the contents. This applies particularly to Gösta Odelberg B. Sc., Regional Manager FiSQA (Field Service Quality Assurance) and Dr. Charles Sizer.

Åkarp, August 1998

Dr. Bernhard von Bockelmann *Dr. Irene von Bockelmann*

Contents

Contents

Introduction

Commercially sterile products have been on the market for a long time. Direct UHT (**U**ltra **H**igh **T**emperature) processing was introduced at the beginning of the 20th century. At that time, the technology was used only as a pre-treatment for products to be filled into cans for subsequent retorting. The first UHT-treated and aseptically packaged products were in cans and were shown at an agricultural exhibition in London in the mid 1920s. The product (milk) was not a commercial success. During the early 1930s, aseptic canning was developed in the USA (69).

In 1961, aseptic packaging procedures were introduced (74, 93, 243) using flexible packaging material (a laminate of wax, paper and polyethylene): the Tetra Pak system ("Tetra Classic Aseptic" or TCA system). The paperboard carton and black colouring provided protection against the entry of light. However, gas barrier characteristics were rather poor. In the meantime, UHT processing systems were in more general use. After initial problems, the combination of UHT processing and aseptic packaging in the TCA system gained market success ("long-life products").

In 1969, a brick-shaped package was introduced ("Tetra Brik Aseptic" or TBA system). The structure of the packaging material was more complex: a polyethylene-paper-polyethylene-aluminium foil-polyethylene laminate. Such a material provides protection against the entry of light and acts as an effective gas barrier. The shape of the container offered considerable advantages in storage, transportation and handling. It also provided the real breakthrough for UHT processing and aseptic packaging technology. Today, larger quantities of both low and high-acid, shelf-stable food products are processed by inflow (UHT) methods and subsequently packaged under aseptic conditions. Milk and milk-based products were the first long-life food products and still remain the largest commodity in terms of volume.

Processes that stabilise food thermally have been a major benefit to mankind by providing stability and safety in food. Although commercial canning is probably the most reliable and safest method of food preservation, it is not perfect. In trying to improve the technology, the present low risk of contamination present in simple straightforward technologies must sometimes be sacrificed to gain better quality and economy.

Aseptic technology is considered to have the following advantages over conventional technologies (254):

a) new package forms;

b) savings in energy and packaging costs;

c) convenience; and

d) improved product quality.

A comparison of the price of different packaging alternatives is shown in table 1 (278).

In table 2, the total cost of canning is compared to the production of a long-life food product (table 2, 179).

Figure 1. *Canning*

Figure 2. *Tetra Classic Aseptic Package*

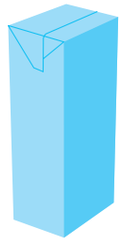


Figure 3. *Tetra Brik Aseptic Package*

Type of Package	US Dollar/1000
Metal can	100
Composite can	80
Glass bottle	75
Aseptic cup	60
Aseptic carton	50

Table 1. *Cost of Containers Made of Different Materials*

	Cans, DM/1000	Aseptic Carton, DM/1000
Packaging material	225	140
Outer wrapping	13	12
Processing and filling	11	21
Total	249	173

Table 2. *Cost of Canning and Aseptic Cartons Made of Paper-Based Laminates*

1. UHT Processing

Summary

Food products are processed by UTH Methods in order to obtain a commercially sterile product. Rapid heating, short holding and rapid cooling minimise the occurrence of chemical change. A short description is given of the principles involved in indirect and direct heating systems.

1. General

Purpose of UHT Treatment

Ultra-high-temperature treatment is a continuous inflow process. It is based on the rapid heating of the product to the sterilisation temperature and short holding at that temperature, followed by rapid cooling. The purpose of UHT treatment is to achieve commercial sterility of the product. Full sterilisation efficiency requires rapid heat transfer which is only possible in liquid systems. If powders are used in the formulation of a product to be UHT-treated, special attention has to be paid to proper soaking: all powder particles must be completely wetted through.

1.1 Low-Acid, Liquid-Food Products

Low-acid food products are characterised by a pH value of > 4.5 or > 4.6, depending on local food legislation. These products require careful treatment because:

a) microorganisms can grow and multiply. Bacterial spores are also able to germinate and cause product spoilage;

b) practically all pathogenic (disease-causing) microorganisms can develop. Consumption of products thus spoiled may lead to food poisoning and/or food-borne infections.

Low-Acid Products: Processing Conditions

Typical processing temperatures for low-acid foods are 130 - 150°C with holding times of a few seconds (60), usually 4 seconds.

1.2 High-Acid Liquid-Food Products

High-acid food products have a pH value equal to 4.5 or 4.6 or less. These are mainly fruit juices, fruit juice drinks and “belly washers”. High-acid food products are safer than low-acid foods because:

a) they are not prone to pathogenic bacteria and are therefore regarded as safe from the point of view of public health;

b) bacterial spores cannot germinate under high-acid conditions and, consequently, cannot cause food spoilage;

c) the sterilising efficiency of any heat treatment increases with decreasing pH. Thus, lower temperatures can be applied in order to achieve commercial sterility;

d) in addition, some organic acids common in fruits specifically decrease the temperature resistance of possible spoilage organisms;

e) the main spoilage microorganisms are yeast, mould, and a few bacteria (*Lactobacillus, Streptococcus,* and some others).

Product	pH
Apple Juice	3.3 - 3.5
Apple Sauce	3.4 - 3.5
Apricots	3.5 - 4.0
Blackberries	3.0 - 4.2
Blueberries	2.5 - 2.7
Cranberry Juice	3.0
Currant Juice	3.6 - 4.0
Fruit Cocktail	3.6 - 4.0
Grapefruit Juice	2.9 - 3.4
Grapes	3.3 - 4.5
Lemon Juice	2.2 - 2.6
Orange Juice	3.0 - 4.0
Tomato Juice	3.9 - 4.7
Pineapple Juice	3.4 - 3.7

Table 1. *ph of Fruits and Fruit Juices*

High Acid Products: Processing Conditions

Processing temperatures for high-acid foods are rather low. With few exceptions temperatures of 85 - 95°C with holding times of 30 to 15 seconds, sometimes a few minutes, are sufficient (60). However, there are some exceptions, particularly tomato products which may require considerably higher temperatures, often above 100°C. The pH values of a number of fruits and fruit juices (131) are given in table 1.

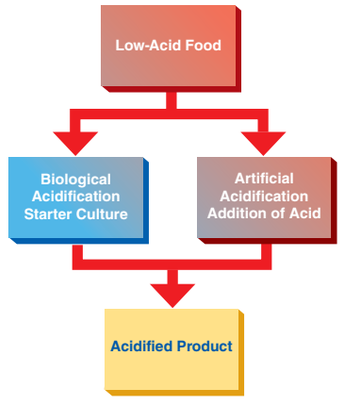


Figure 1. *Flow Chart: Production of Acidified Food Products*

1.3 Acidified Liquid-Food Products

Acidified foods are low-acid products, the pH of which has been lowered into the high-acid range by pre-processing. Such products have the same microbiological advantages as high-acid food products. The acidification process is crucial. It is necessary to achieve an even and low pH throughout the product. Acidification can be carried out biologically by the addition and growth of a starter culture, usually *Lactobacillus* and/or *Streptococcus* followed by a ripening period at a suitable temperature. Another possibility is an artificial or chemical procedure in which an acid (usually citric or lactic acid) is added to the product which has to be mixed thoroughly afterwards (Figure 1).

To achieve commercial sterility, the acidified product has to be processed after the acidification process, usually by heat treatment, and then packaged.

2. Indirect UHT Systems

A heat exchange surface separates the product from the heating or cooling media in indirect UHT systems. The heating medium may be either steam or super-heated water (Figure 2).

A time-temperature diagram for indirect systems is shown in figure 3.

Typically, the product enters the steriliser via a balance inlet tank and a centrifugal feeding pump at about 4°C. Subsequently, it is heated to 70-75°C, at which temperature the product is homogenised. For milk homogenisation, pressures of 200 to 250 kg/cm^2 (150 to 200 at the first stage, and about 50 kg/cm^2 at the second) are often used. The homogeniser is a positive piston pump which pushes the product through the remaining equipment directly to the aseptic filling machine or into an aseptic tank for subsequent filling. Sterilisation temperatures are usually between 135°C and 140°C. Holding times between 2 and 6 seconds at the sterilisation temperature are common. Though more expensive, downstream (aseptic) homogenisation results in better flavour characteristics for some products.

In order to lower the oxygen content of the product, deaeration can be introduced prior to upstream homogenisation. Milk entering the steriliser is normally saturated with oxygen. Since indirect working sterilisers are closed systems, the incoming and outgoing oxygen content of the product is the same. Depending upon temperature, milk may contain 6 to 9 ppm of oxygen (ca 7 ppm), (168). Passing a vacuum chamber (deaerator) at high temperature prior to homogenisation (~ 70°C), the oxygen content can be reduced to about < 1 to 3 ppm (0.3 - 0.9 ppm), (168). The degree of deaeration depends upon the temperature and the underpressure applied in the process.

During heat processing, deposits form on heat exchange surfaces, particularly in

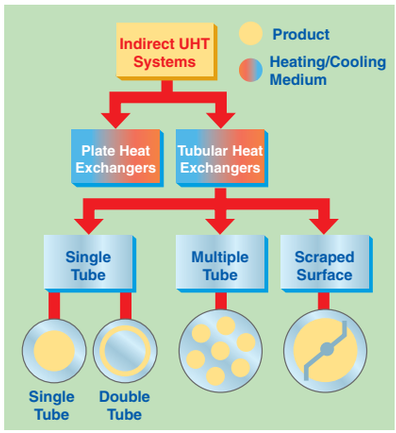


Figure 2. *UHT Treatment: Indirect Systems*

Indirect Heating

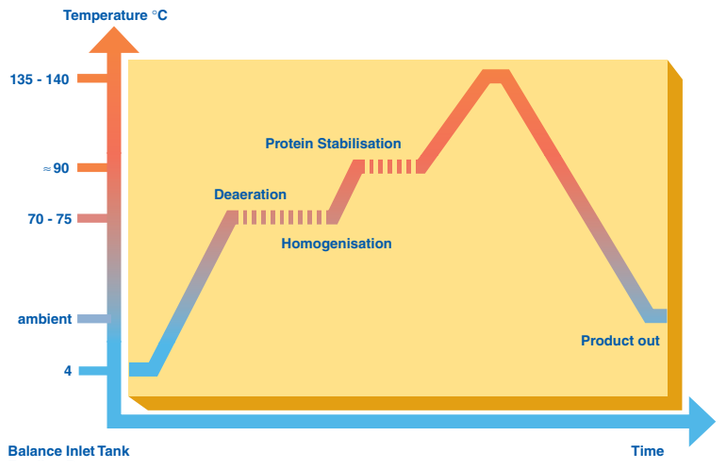
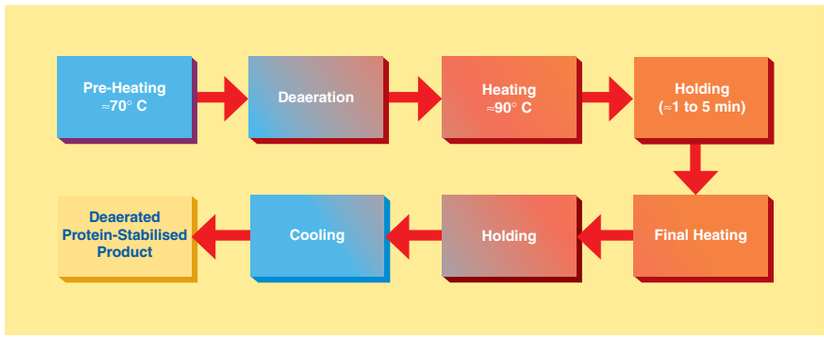


Figure 3. *Time-Temperature Diagram: Indirect Heating*



Deaeration
Stabilisation

Figure 4. *Flow Chart: Indirect Heating*

the temperature range > 80°C. In order to reduce this deposit formation and thus prolong the running time, a holding cell can be introduced: the product (milk) is held for a few minutes at a temperature of about 90°C (Figure 4). Indirect systems offer good possibilities for regenerative energy recovery: the incoming product is heated by the outgoing.

In the equipment shown in figure 5, a pressure drop takes place. As a consequence of this, the pressure of the incoming product is higher than the pressure of the commercially sterile outgoing product. Leakage in the regenerative section and the final cooler may lead to reinfection. This risk can be eliminated by introducing a "booster pump" after the final heating section figure 6).

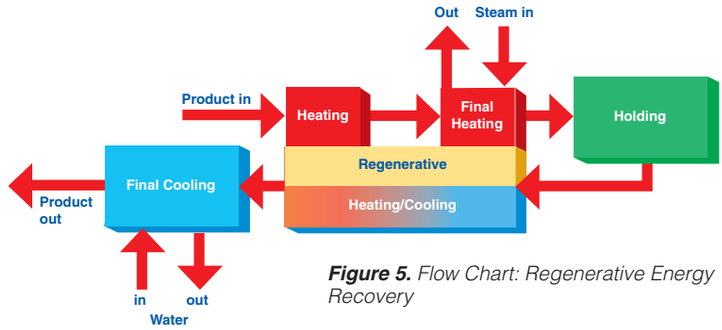


Figure 5. *Flow Chart: Regenerative Energy Recovery*

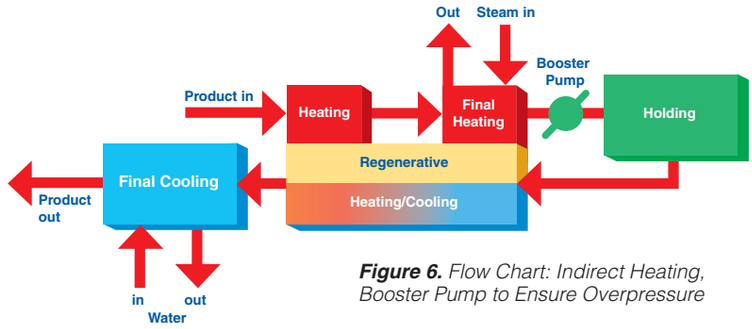


Figure 6. *Flow Chart: Indirect Heating, Booster Pump to Ensure Overpressure*

Advantages of indirect UHT systems are:

a) technically, they are relatively simple;

b) they are comparatively inexpensive in terms of investment cost;

c) they permit high values of regenerative energy recovery (for plate heat exchangers up to 93%);

d) they require limited service;

e) they have comparatively low running costs.

Advantages
Indirect Systems

For both plate and tubular (single and multiple tube) systems for commercial production, the capacity range available is from 2,000 to 30,000 litres/hour or even more.

3. Direct UHT Systems

Direct UHT plants feature direct contact between the heating medium and the product. The heating medium is usually steam but electrical heating ("Elecster", "Ohmic") has been introduced to a very limited extent (figure 7).

Electrical heating features the passage of an electrical current through the product. Although it is also used for liquids, the system is mainly intended to be applied in the sterilisation of products containing particles. Commercial application is very limited although a number of plants have

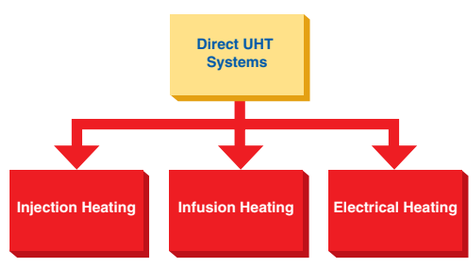


Figure 7. *UHT Treatment: Direct Systems*

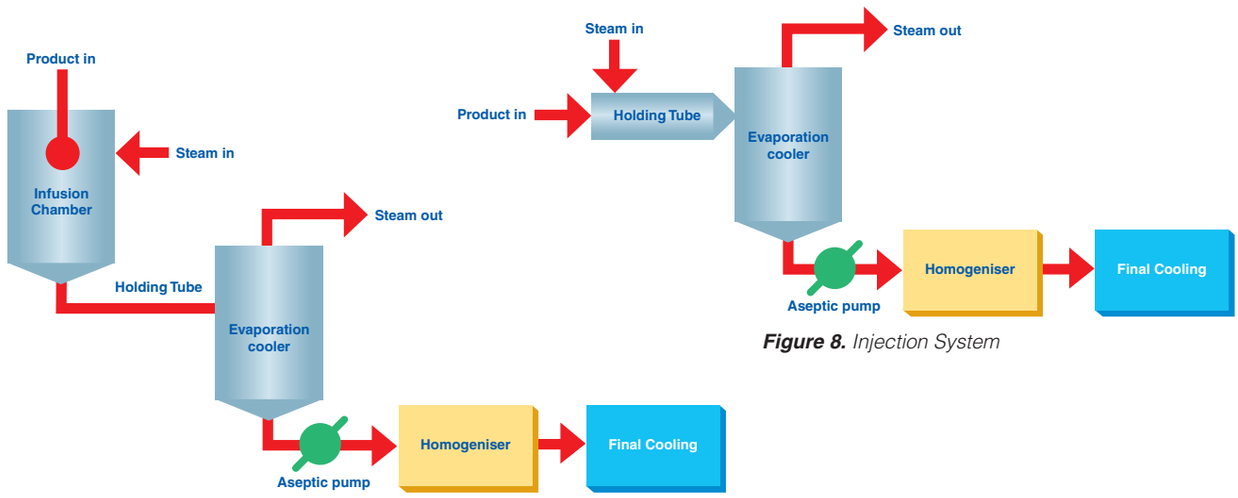


Figure 8. *Injection System*

Figure 9. *Infusion System*

Electrical Heating

been installed in the research and development departments of some major food and animal-feed processing companies. One problem is the difference in electrical resistance which can exist between the liquid and solid phases. Other problems generally associated with the processing of products containing particles are those of separation during processing and aseptic transfer, and mechanical damage to particles softened by processing.

Injection implies direct steam injection into the product. In infusion plants, the product is infused into a steam chamber.

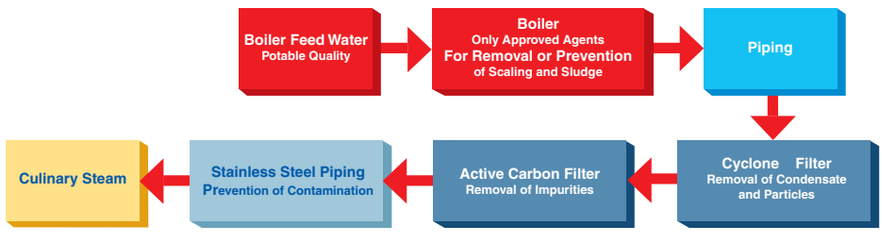


Figure 10. *Flow Chart: Culinary Steam*

Steam Requirements

Injection and infusion systems must be operated with culinary steam.

The minimum requirements outlined in figure 10 must be observed for the steam in such plants (not necessary for electrical heating). Indirect working plants should also use culinary steam especially if they are sterilised with steam.

The US Department of Health and Human Services (17) lists the following requirements for culinary steam:

1. *Boiler Feed Water:* If boiler feed water is treated, the process must be under the supervision of trained personnel. Only compounds complying with Section 173.310 of 21 CFR (258) may be used to prevent corrosion and scale.
2. *Boiler Operation:* A supply of clean, dry saturated steam is necessary for proper equipment operation. It is recommended that periodic analysis be made of condensate samples.
3. *Piping Assemblies:* The steam supply line should be as shown in figure 11.

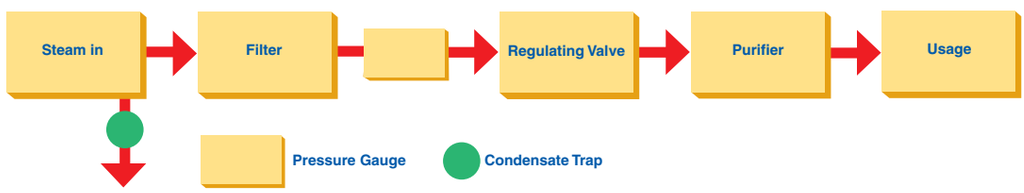


Figure 11. *Flow Chart: Steam Purification*

A typical time-temperature diagram for direct heating is shown in figure 12.

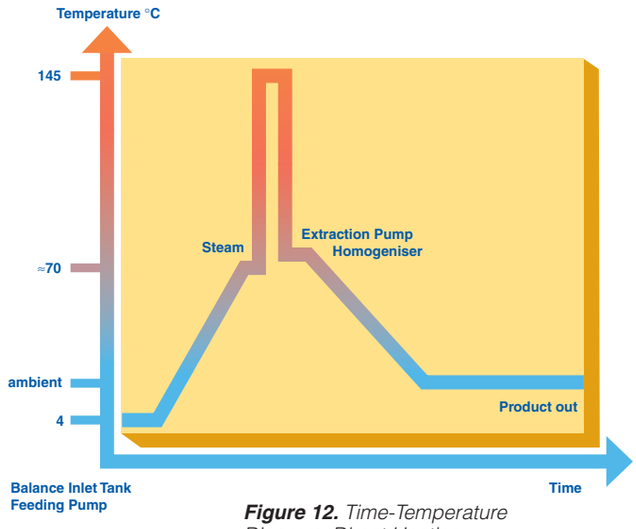


Figure 12. *Time-Temperature Diagram: Direct Heating*

Direct Heating

The product enters the steriliser via a balance inlet tank and a centrifugal feeding pump at a temperature of about 4°C. It is heated by plate or tubular heat exchangers to about 70°C. At this stage, steam is injected into the product or the product is infused into a steam chamber. Steam condensation increases the temperature almost instantaneously (~ 0.1 sec in injection and ~ 0.25 sec in infusion systems) to the sterilisation temperature which is typically between 145°C and 150°C. The average holding time at the sterilisation temperature is usually around 4 seconds. In both the injection and infusion processes, water condenses in the product and dilutes it. Depending on the temperature difference, an increase in volume of about 10% results. This water must be subsequently removed. The outlet of the holding cell connects to a vacuum chamber. To prevent boiling in the product holding cell, a sufficient overpressure by a suitable restriction device must be introduced. Being exposed to underpressure, the product starts boiling vigorously and steam is flushed off. Careful adjustment of the injection (infusion) temperature and the underpressure in the vacuum chamber guarantees the same dry matter content of the incoming and outgoing product. The resulting pressure drop requires the installation of an aseptic extraction pump for further product transportation. In order to avoid an accumulation of product in the expansion cooler or its emptying, the capacity of both the product feeding pump and the extraction pump at the outlet of the expansion cooler must be carefully matched.

Cavitation forces during steam condensation and the boiling in the expansion cooler destabilise milk protein and fat. To compensate this effect requires downstream homogenisation which has to be done under aseptic conditions. Homogenisation pressures for milk are usually 200 to 250 kg/cm^2 (150 to 200 kg/cm^2 at the first stage and about 50 kg/cm^2 at the second). The homogeniser pushes the product through the final cooling section of the steriliser, either into a sterile tank or directly to the aseptic filling machine.

Removal of Gas: Deaeration

In the expansion cooler, not only water is removed from the product but also all other volatile compounds. In addition, the vacuum chamber functions as a very effective deaerator that removes oxygen and other dissolved gases, mainly carbon dioxide (CO_2). As a consequence, the freezing point increases. At the outlet of the expansion cooler, the oxygen content of milk is down to ~ 0.1 ppm.

Advantages Direct System

Advantages of injection and infusion heating are:

a) a lower total heat load, as a result of which fewer chemical changes are inflicted on the product;

b) less scaling, particularly in the temperature range of 70°C and above resulting in longer production runs (less frequent cleaning and sterilisation);

c) the low oxygen content in the product increases the stability of some vitamins and, during storage, reduces flavour changes caused by oxidation;

d) more suitable for viscous products.

Combination: Direct and Indirect Heating

Recently, special UHT heat exchangers have been developed combining direct and indirect heating. In one assembly, tubular heating and steam injection have been combined. The product enters at ~4°C and is heated to 95°C by tubular heat exchangers. It then passes a holding cell at the same temperature to stabilise the protein. Steam injection raises the temperature almost instantly to 140-150°C. The product is held at this temperature for a few seconds before being cooled down. Pre-cooling is performed in a tubular heat exchanger where

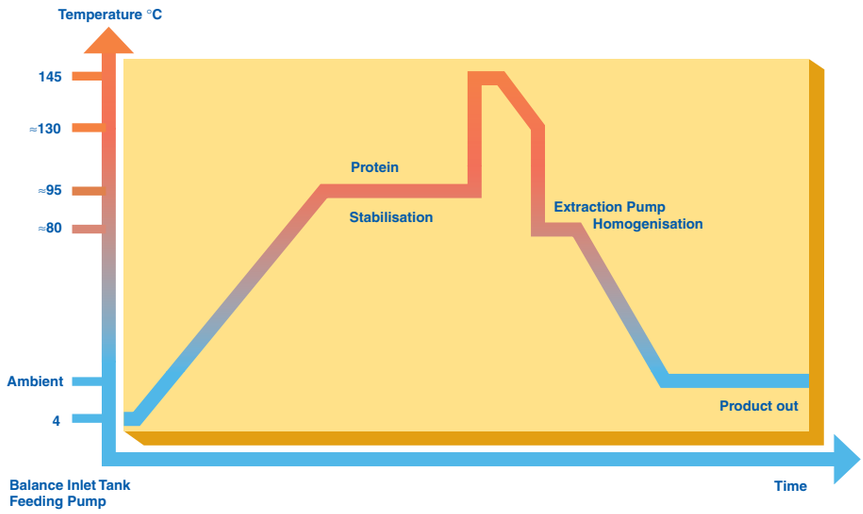


Figure 13. *Time-Temperature Diagram: Combined Indirect and Direct Heating*

the heat energy is utilised for regenerative heating. The injected steam is flushed off as vapour in a vacuum cooler. The temperature drops to 80°C. Aseptic homogenisation is needed. Subsequently, the product is cooled down to ambient and filled aseptically.

4. Plant Sterilisation

Prior to production, the equipment must be sterilised. This can be achieved either by superheated water or by steam. Because of the expansion and infusion vessels, direct working systems must be sterilised by steam. It is essential that the temperature controller/guarding sensor is placed at the most sensitive spot, usually somewhere in the return.

If superheated water is used to sterilise the plant, the temperature, time, and flow rates are critical (142). Possible air pockets and the interface between the product steriliser and the aseptic tank circuit are significant. Air is effectively removed from the system if the flow rate is > 1.5 m/second.

An additional sterilisation circuit is required in installations operating through an aseptic tank. The sterilisation medium is always steam. In steam sterilisation processes, a possible problem is condensate which must be removed from the system.

Sterilisation of the aseptic filling equipment is always performed separately either by heat alone or by chemicals and heat. Attention should be paid to the interface between the product line and the filler.

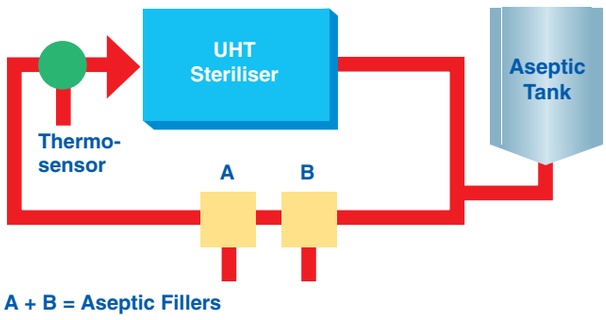


Figure 14. *Flow Chart: Plant Sterilisation*

2. Aseptic Packaging

Summary

The chapter describes different principles of aseptic packaging, primarily those for packaging systems using paperboard-based packaging materials and hydrogen peroxide (H_2O_2) as a sterilant. Attention is also paid to the sterilisation of the packaging material, the sterile environment in which the packages are formed, filled and sealed, and the production of tight containers.

1. General

UHT processing results in a commercially sterile product. The task of the aseptic packaging operation is to:

a) maintain the high microbiological quality of the product for the length of its intended shelf life; and

b) retain consumer acceptance with regard to the flavour, texture and nutritional value of the product during its promised shelf life.

Four different principles of aseptic packaging are distinguished (74):

a) filling in a sterile working area;

b) aseptic filling into pre-formed containers;

c) aseptic filling into pre-formed sterile containers; and

d) the aseptic form-fill-and-seal method.

By and large, only the aseptic form-fill-and-seal method will be covered, which is also the best and most successful technology. Such an aseptic packaging operation requires several functional steps (figure 1) (60, 61, 74, 86).

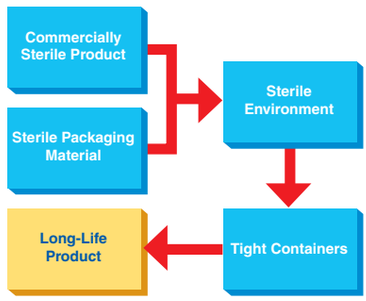


Figure 1. *Functional Steps Involved in Aseptic Packaging*

2. Sterilisation of the Packaging Material

In aseptic packaging procedures, sterilisation of the packaging material (food contact surface) is achieved with few exceptions by chemical means (61). By far the most common chemical used for this purpose is hydrogen peroxide (H_2O_2) (93, 142). Of importance are the microbiological efficiency of the sterilisation process and the elimination of the chemical which might get into the filled product as a residue.

Depending on the make of the aseptic packaging equipment, different means of applying the sterilant are used:

Application of Hydrogen Peroxide

1) spray;

2) vapour;

3) roller systems;

4) immersion bath, etc.

2.1 Spray Application

The spraying ("fogging") of hydrogen peroxide is used in some aseptic packaging systems which operate intermittently and use pre-formed blanks (61, 74, 93, 171).

A certain amount of H_2O_2 is sprayed into each pre-erected container through a spray nozzle (figure 2). For proper function (sterilisation), the food contact surface of the container needs to be covered completely with the spray solution. Because of the hydrophobic characteristics of plastic materials in general and polyethylene in particular, it has been found (91, 145) that only 20-30% of the carton surfaces are wetted. In spite of this, good killing effects were achieved: up to 6 log reductions when tested with *Bacillus subtilis* spores have been reported (145),

Efficiency of Spray Application

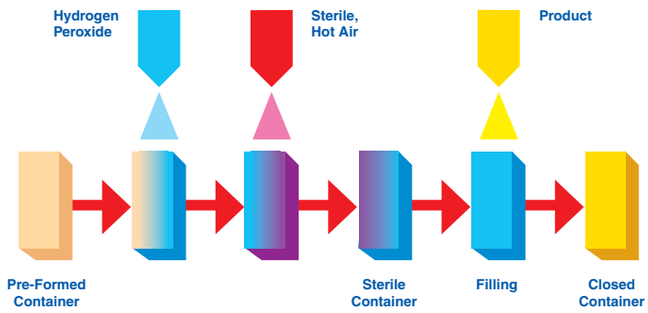


Figure 2. *Flow Chart: Spray Application of Hydrogen Peroxide in the Sterilisation of Packaging Material*

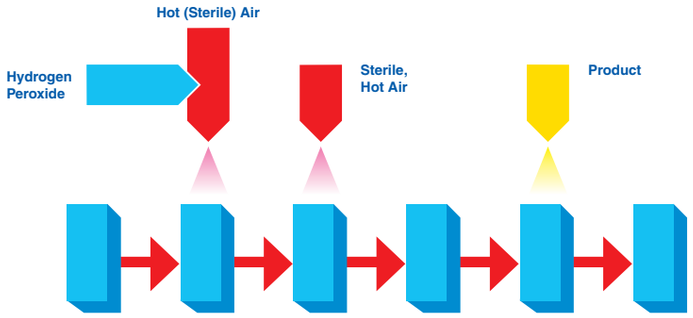


Figure 3. *Flow Chart: Evaporation and Condensation of Hydrogen Peroxide in Packaging Material Sterilisation*

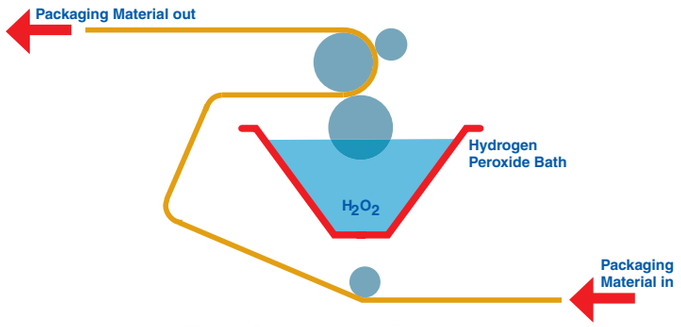


Figure 4. *Application of Hydrogen Peroxide by a Roller System*

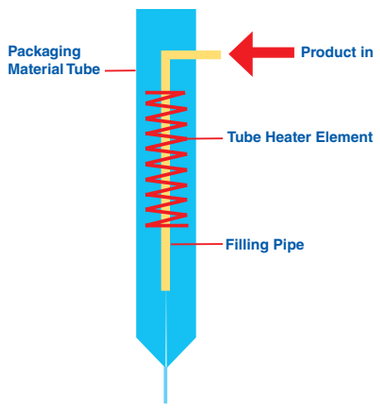


Figure 5. *Heating and Elimination of Hydrogen Peroxide*

probably because of the subsequent evaporation. Hot sterile air is blown into the container to attain the temperature necessary for the sterilisation process and to remove the H_2O_2 from the food contact surface. Using sterile air at 180°C for drying, 5 to 7 decimal reductions of *Bacillus subtilis* spores were attained (91, 113).

The killing efficiency of low concentrations (~ 0.5% or less) of hydrogen peroxide was greatly enhanced by the simultaneous use of ultraviolet radiation (44, 45).

2.2 Application by Vapour

In systems applying hydrogen peroxide by vaporisation, liquid H_2O_2 is injected into a stream of hot (sterile) air, then vaporised and condensed on the surfaces to be sterilised (93). A better coverage of surfaces is obtained since the condensing droplets are smaller. Subsequently, the hydrogen peroxide is heated and evaporated by blowing hot, sterile air into the containers (figure 3).

2.3 Application by Roller System

Roller systems (figure 4) permit the application of liquid hydrogen peroxide on to flat food contact surfaces (59, 60, 61, 74, 93). Packaging material sterilisation thus becomes possible before the containers are formed (figure 4).

In order to obtain a film of the water-like H_2O_2 liquid covering the total food contact surface, a wetting agent - about 0.2 to 0.3% of PSM, (polyoxyethylene-sorbitan-monolaurate), or equivalent is recommended - has to be added to the hydrogen peroxide. After application of the sterilant on to the food contact surface, the packaging material web is formed into a tube and sealed longitudinally. The actual sterilisation requires the hydrogen peroxide covering the packaging material food contact surface to be at a high temperature. An electrical element ("tube heater") provides the temperature necessary (105-110°C) for the sterilisation process and, simultaneously, eliminates the H_2O_2 (figure 5).

2.4 Use of an Immersion Bath

Likewise, the use of a hydrogen peroxide bath permits sterilisation of a plane packaging material web (59, 61, 69, 73, 74, 93, 171) prior to the actual forming of the container. The packaging material is sterilised by its passage through liquid hydrogen peroxide. The temperature necessary for the sterilisation process is obtained by heating the hydrogen peroxide solution indirectly by means of a water bath which is placed inside the H_2O_2 bath (figure 6). A concentration of 30%, a temperature of 70°C and an exposure time of about 10 seconds are necessary to achieve sufficient killing of bacterial endospores (74). In some models, the hydrogen peroxide is removed from the packaging material:

a) by a pair of pressure rollers that squeeze the excess chemical back into the hydrogen peroxide bath; and

b) by a pair of air knives that blow hot, sterile air on to both sides of the packaging material web.

After passage through the bath and removal of the hydrogen peroxide, the sterile, flat packaging material web is formed into a tube and sealed longitudinally.

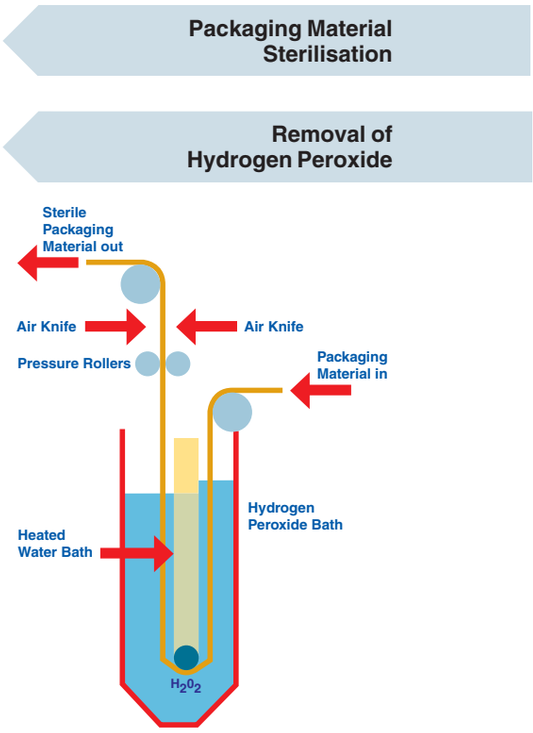


Figure 6. *Hydrogen Peroxide: Dip-in Bath*

3. Sterile Environment

After sterilisation of the product and packaging material (food contact surface), reinfection has to be avoided. The containers have to be formed, filled and sealed in a sterile environment. To achieve this, the area in which the process is executed has to be sterilised, and sterility has to be maintained during the entire length of the intended production run.

3.1 Sterilisation of the Filler

Repeatedly successful sterilisation requires clean surfaces and thus proper cleaning of the equipment prior to sterilisation. Depending upon the construction and complexity of the aseptic filling equipment, sterilisation of the machine can either be performed by:

1) heat alone; or by

2) a combination of heat and chemicals.

3.1.1 Sterilisation by Heat Alone

Some total systems or parts thereof permit sterilisation by heat alone using steam and/or heated air. An example is given in figure 7.

In such systems, a combination of steam and dry air at high temperature is often used. Sterile air is produced by incineration at a temperature of 330-360°C (75) and the hot, sterile air is admitted into the machine via a valve cluster. The filling pipe and the "sterile area" are thus sterilised by dry heat at a minimum temperature of 240°C for 30 minutes. A steam barrier separates the aseptic filler from the product supply line. The steam barrier area as well as the product valve are sterilised by steam at a temperature of about 130°C.

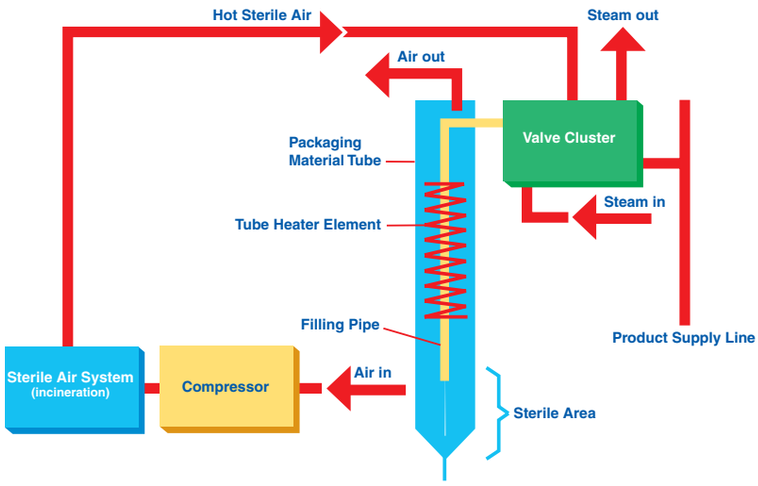


Figure 7. *Flow Chart: Sterilisation of the Filler by Heat Alone*

3.1.2 Sterilisation by a Combination of Heat and Chemicals

More complex aseptic filling systems cannot be sterilised by heat alone. Structures such as form rings, guiding rollers, etc., might not tolerate the temperatures necessary. The construction may be so solid that the time required for heating and subsequent cooling becomes prohibitive. Such systems demand a combination of heat and chemicals for sterilisation. For this purpose, the most commonly used chemical is hydrogen peroxide (73, 75).

The hydrogen peroxide may be applied in two different ways:

- by spraying as a fog; or
- by evaporation as a gas which condenses on the surfaces to be sterilised (75).

In any event, warm or hot sterile air has to be admitted to the system in order to achieve the temperature necessary for the sterilising effect needed and to remove the hydrogen peroxide from the system (figure 8). Areas not included in the hydrogen peroxide sterilisation cycle have to be sterilised by either dry or wet heat.

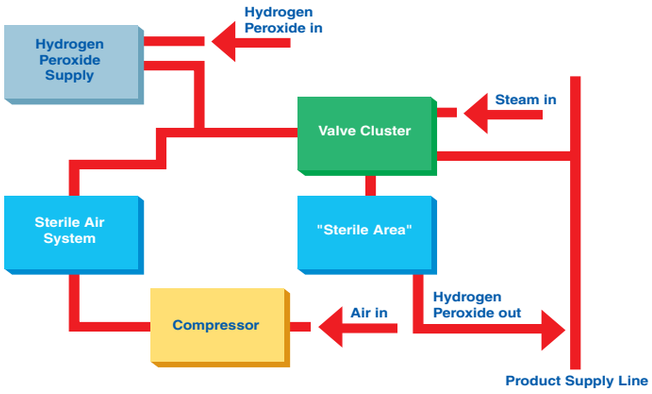


Figure 8. *Flow Chart: Sterilisation of the Filler by a Combination of Heat and Chemicals*

Air Sterilisation by Filtration

3.2 Maintaining Sterility during Production

In most aseptic packaging systems, an overpressure of sterile air is used to seal the area in which the containers are formed, filled and sealed from the non-sterile surroundings (73, 142) (figure 9).

Sterility of the air may either be obtained by filtration (142) or incineration, or a combination of both. For filtration, HEPA (high efficiency particular air) filters are often used. The following features are recommended:

- air supply by low-pressure blowers;
- air speed 0.5 m/sec (laminar flow); and
- retention of 99.99% of all particles larger than 0.3 µ.

If incineration is used, a temperature of about 340°C is needed for sterilisation. The air can be cooled by a water cooler or by a regenerative heat exchange system where the incoming air is pre-heated by the air leaving the incinerator.

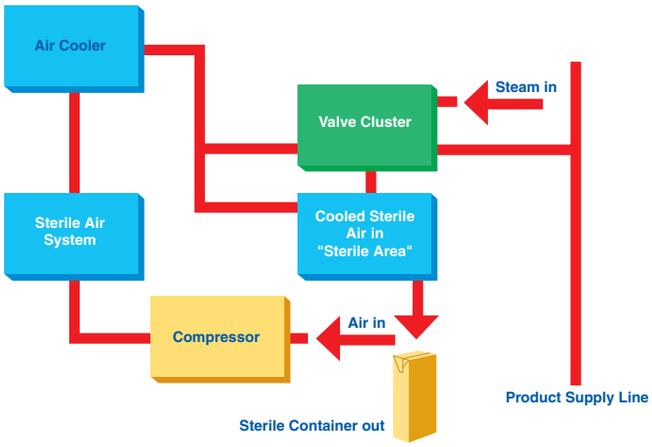


Figure 9. *Flow Chart: Maintaining Sterility by Overpressure with Sterile Air*

4. Production of Tight Containers

Package Integrity Problems

The tightness of the containers produced depends upon:

- the correct top and bottom seals (transversal seals);
- a tight longitudinal seal; and
- an undamaged packaging material structure.

Problems of package integrity can be caused by the quality of the packaging material as such, wrong adjustments, the maintenance and operation of the aseptic filling equipment, and transport damage. In order to minimise the impact of these factors, suitable procedures should be introduced such as:

Prevention of Package Integrity Problems

a) a functional test of each new shipment of packaging material;
b) proper maintenance of the filling equipment;
c) machine operator training;
d) competent supervision;
e) appropriate outer-wrapping equipment which needs to be properly maintained;
f) outer-wrapping materials which provide sufficient protection of the containers during transportation and handling;
g) information to all sections and parties concerned with the handling of the aseptic containers; and
h) storage of the packaging material prior to use under suitable, hygienic conditions (temperature and humidity) and times (as recommended by the manufacturer).

Package Integrity: Methods of Checking

A number of procedures for testing and checking package integrity are available today:

a) dye test procedures (12, 86);
b) increase in oxygen content (12);
c) conductivity measurement; and
d) tear-down procedures.

Bio-testing (the submersion of packages into a solution rich in microorganisms, figure 10) is not recommended since the procedure affects the packaging material: artificial defects are created and interpretation of the results obtained becomes impossible!

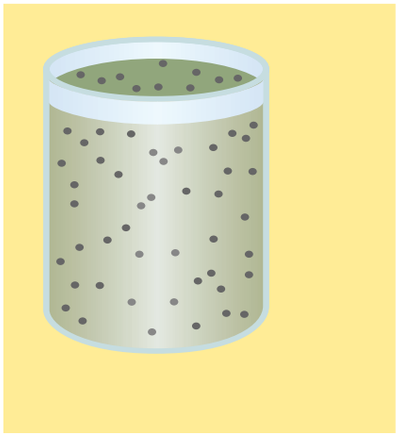


Figure 10. *Bio-Testing: Not Suitable for Paper-Based Plastic Laminates*

Some bacteria are able to pass through minute channels which will not permit visible leakage of the product (86). In model experiments using plastic materials, it was observed that the motile bacteria *Escherichia coli, Pseudomonas aeruginosa* and *Bacillus subtilis* could move actively through capillaries filled with liquid and with an inner width of 10 µ or more at a velocity of more than 10 mm within 24 hours. The movement was slowed down considerably when the diameter was reduced. The minimum transversal inside diameter permitting passage was between 1.3 and 2 µ. The non-motile bacteria *Staphylococcus aureus* and *Bacillus megaterium,* as well as the yeasts *Debaryomyces hansenii, Candida parapsilosis* and *Torulopsis candida* moved very slowly. Their speed was less than 0.5 mm in 24 hours in capillaries with inner diameters of up to 10 µ. Depending on the cell size, the smallest capillaries which could still be penetrated by non-motile bacteria had an inner diameter of 2 to 5 µ (275).

3. Sterilising Effect

Summary

The killing of microorganisms is not an absolute process; not all bacteria are killed at the same time. Rather, using the same lethal procedure, an equal fraction is killed at distinct time intervals. It is important to understand this! As a consequence, the result of any sterilisation operation is determined by the process parameters applied to, *and* the microbiological load subjected on, such a process. Sometimes the process parameters cannot be altered in order to render the process more efficient. In such a situation an improvement in the result can only be obtained by reducing the microbial load – often the bacterial spore count – in the product to be sterilised.

1. General

Killing of Bacterial Spores

The subject of the "sterility" of food containers and products is controversial. On the one hand, legislation is - more or less - compelled to define sterility on an absolute basis, i.e., "the absence of ...". On the other hand, it is generally accepted that microorganisms when subjected to a lethal process are killed in a semi-logarithmic fashion (60, 69). The killing of microorganisms in general and bacterial endospores in particular has been studied extensively. For most micro-organisms, a semi-logarithmic death rate is found (figure 1) irrespective of whether a thermal or chemical sterilisation process is applied:

Logarithmic Death Rate

$$\log (N_0/N) = \text{contact time with sterilant}/D$$

where N_0 = the initial number of viable organisms, N = the viable count after a given time of contact with the sterilant, and D = the decimal reduction time (i.e., the time needed to achieve one logarithmic reduction in the count of surviving organisms). The sterilant may either be heat or a chemical.

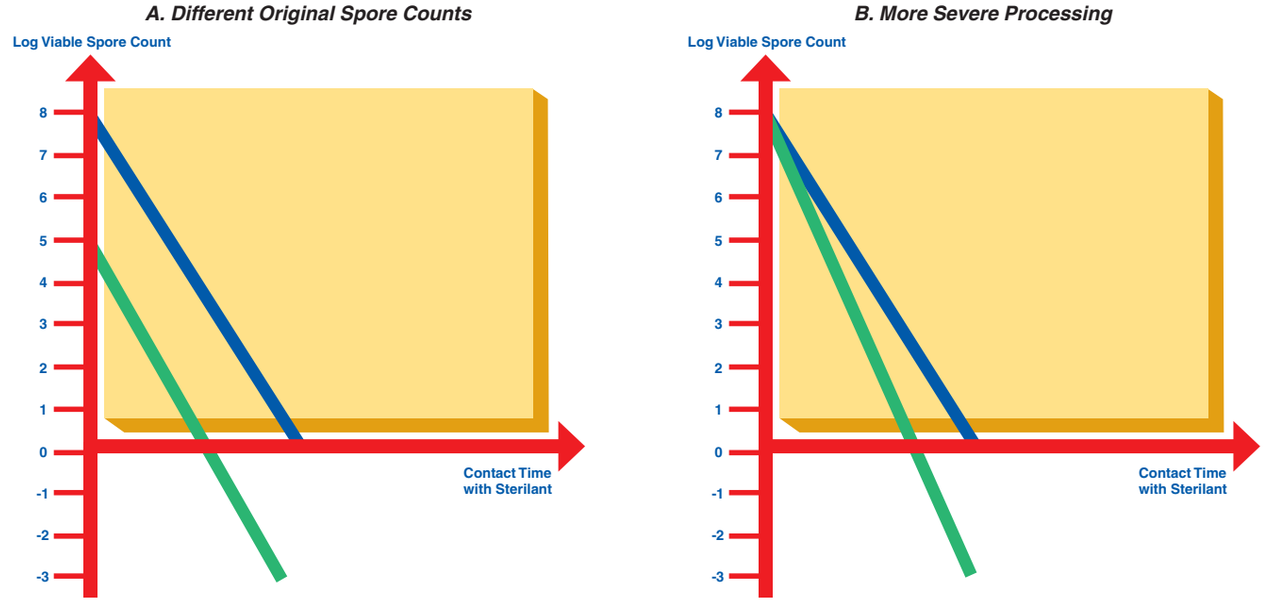


Figure 1. *Death Curves: Effect of Spore Count and Processing*

An improved result can be obtained either by reducing the bacterial spore load in the product (figure 1 A) or by applying more severe sterilisation conditions (figure 1 B), i.e., a higher sterilisation temperature and/or longer holding times at the sterilisation temperature.

Total Absence is Total Nonsense

Sterility implies the total absence of *all* living microorganisms in any volume of product. Expressed in terms of the semi-logarithmic death rate of microorganisms, this would imply log x = 0. However, as the equation log x = 0 ($10^x = 0$) does not exist, sterility in an absolute sense cannot be achieved; it can only be approached. Consequently, every sterilisation process must have survivors (91, 176)! Each sterilisation procedure is characterised by a sterilisation effect or efficiency. This sterilisation efficiency can be expressed by the number of logarithmic (decimal) reductions achieved by the process. For example, it is safe to assume that a normal UHT process achieves nine decimal reductions in milk, i.e., out of 10^9 spores fed into the process one will survive.

10^9 bacterial spores → UHT → 10^0 = 1

This is true irrespective of volume.

D–Value, F–Value and z–Value

The D-value is the time needed at a given temperature to achieve one decimal reduction in the bacterial spore count. The F-value for a process is the number of minutes required to kill a known population of microorganisms in a given food under specified conditions. This F_0-value is set at 12 D. When the symbol F_0 is used, a z-value of 18°F (10°C) is assumed with an exposure temperature of 250°F (121.1°C). The z-value states the increase in temperature which is necessary to reduce the decimal reduction time (D-value) by one power of ten, i.e. from 1 to 0.1 minute. The Q_{10} value is the increase in the speed of a reaction if the temperature of the system is raised by 10°C. Assuming a z-value of 10.5 and a Q_{10}-value of 9, direct heating by UHT to 140°C with a holding time of 4 seconds resulted in an F_0-value of 4.2 seconds (105).

Sterilising Effect

In the United Kingdom, the Department of Health and Social Security has issued guidelines for the processing of low-acid canned foods which specify that a satisfactory minimum thermal process must reduce the probability of survival of *Clostridium botulinum* spores to less than 1 in 10^{12} containers (70). This is usually interpreted in terms of a lethality corresponding to heating for 3 minutes at 121°C (F_0 = 3) (23). In a plate heat exchanger, a holding time of 2 seconds at 142°C resulted in a sterilising effect (number of log reductions) of 7 using *Bacillus stearothermophilus* endospores as a test organism (70). However, bacteriological results obtained by heat treatments using *Bacillus stearothermophilus* spores and milk have to be corrected for an inhibitory factor (78), since the heat treatment of milk may result in the formation of compounds which inhibit the germination of bacterial spores in general and those of *Bacillus stearothermophilus* in particular.

Sterilising Effect Achieved by UHT

In milk and cream infected with large numbers of spores of *Bacillus licheniformis,* the z-value was found to be 7.4-8.0°C (46). In the UHT temperature range, a Q_{10}-value of 11 is given for endospores of *Bacillus stearothermophilus* while the corresponding value for *Bacillus subtilis* was 30 (70). Heating *Bacillus subtilis* spores to UHT temperatures (129-135°C) in skimmed milk, resulted in z-values ranging from 6.7°C to 18°C (111).

Linear semi-logarithmic death curves were obtained if *Bacillusstearothermophilus* spores were heated between 104.4°C and 137.8°C at pH 6.0 and 7.0. The corresponding z-values were 8.7°C and 10.3°C respectively (70).

Sterilising Effect: Bacillus

Using direct steam injection, a holding time of 4 seconds and different temperatures, the following logarithmic reductions (sterilising effect) were found (table 1) (250, 257):

In a pilot UHT plant, skimmed milk was heated to 135°C using endospores of

	Bacillus subtilis			*Bacillus stearothermophilus*		
Temp. °C	**Initial Spore Count**	**Final Spore Count**	**Log. Red.**	**Initial Spore Count**	**Final Spore Count**	**Log. Red.**
140°C	45,000	0.0004	9.0	10,000	0.0004	7.4
135°C		0.0004	9.0		0.0004	7.4
130°C		0.0007	8.8		0.45	4.3
125°C		0.45	6.0		2.5	3.6
135°C	4,000,000	0.0004	10.0	250,000	0.0004	9.0
130°C		0.0004	10.0		0.095	6.4
125°C		0.04	8.0		2.5	5.0
120°C		4.5	6.0		2.5	5.0
135°C	75,000,000	0.0004	11.0			
130°C		0.025	9.5			
125°C		4				

Table 1. *Sterilising Effect: Direct Steam Injection*

different strains of Bacillus stearothermophilus as test organisms, "the z-value never dropped below 6.7°C (12°F) and never exceeded 18°C (32°F)" (82).

The inactivation of *Clostridium perfringens* type A spores at ultra-high temperatures has been studied. Aqueous spore suspensions were heated to 85-135°C by the capillary tube method. At temperatures above 100°C (1), the results indicated a rapid inactivation of the spores. The decimal reduction time (D-value) of the most resistant *Clostridium botulinum* spores at 140°C is of the order of 0.1 of a second. Over a temperature range of 120-140°C, a z-value of 11°C was found for spores of *Clostridium botulinum* suspended in a phosphate buffer (pH 7) (23). Using the capillary test method, a significant increase in the z-value resulted in an increase in temperature in the range of 85-160°C (70). Twelve decimal reductions of *Clostridium sporogenes* could be achieved within 3.42 seconds at 148.9°C. This was from a spore suspension characterised by a z-value of 19.4°C and a D-value (at 148.9°C) of 0.285 of a second (70).

Sterilising Effect: Clostridium

The number of *Cl. botulinum* spores in raw materials may vary from 10^4 per container of product, which may occur in some mushroom products, to 10^{-1}, which may be the level for meats, to perhaps as low as 10^{-5} for the packaging material needed to make one aseptic container (201). Decimal reduction values in themselves are of very limited interest unless the possible microbiological load is also taken into consideration. Generally, food products will carry higher spore loads than packaging material. There is a potential hazard in expressing process requirements as "12 D", since only the spore reduction is specified, whereas the final result will vary with the initial concentration of *Cl. botulinum* and other spores (201). The sudden changes in temperature caused by the direct mixing of steam with milk and vacuum evaporation by direct heating, gives rise to a "heat-shock" destructive effect on bacterial spores. Spore destruction could be higher than expected in comparison with results obtained in the laboratory by exposing spore suspensions to lower temperatures. Therefore, data obtained in the laboratory should not be extrapolated to conditions prevailing under UHT operational conditions (86). All the experimental results were obtained with models using pure bacterial spore suspensions of more or less well defined heat resistance. In addition, these spores were used in very high concentrations. It is questionable to what extent the results obtained can be transferred to conditions that exist in reality.

Temp. °C	**Initial Colony Count/ml**	**Final Colony Count/ml**	**Decimal Reductions**
130.5	7.2×10^6	0.9	7
	6.9×10^6	0.6	7
132.0	4.8×10^6	0.2	7
	4.8×10^6	0.09	7
133.0	4.7×10^6	0.001	8
	7.3×10^6	0.001	8
135.5	4.9×10^6	<0.0004	>8
	8.2×10^6	<0.0004	>9

Table 2. *The Effect of Temperature on* Bacillus subtilis *786: Spore Destruction in Milk in a UHT Plant (120)*

Effect of Heat Shock

4. Hydrogen Peroxide as a Sterilant

Summary

In aseptic packaging systems, hydrogen peroxide (H_2O_2) is often used for the chemical sterilisation of the packaging material (food contact surfaces). Aspects relating to residues in the packaged food product, to environmental conditions, as well as to the microbiological efficiency of the chemical, are discussed.

1. General

In most aseptic - and some extended shelf life (ESL) - packaging systems, hydrogen peroxide is used either to sterilise or to sanitise packaging material food contact surfaces. Such chemicals should fulfil certain requirements. A perfect sterilant should be (61):

Requirements for a Chemical Used in the Sterilisation of Food Contact Surfaces

a) easy to apply;
b) part of an in-line application;
c) residue-free after application;
d) effective in killing potential spoilage microorganisms such as bacterial endospores;
e) inexpensive;
f) non-toxic;
g) non-corrosive; and
h) generally available.

Of major importance are microbiological effectiveness and the presence of residues in the packaged product. Although other, and in some aspects, more effective chemicals are available, hydrogen peroxide appears to offer the best compromise.

2. Microbiological Aspects

The microbiological efficiency of hydrogen peroxide has been studied with *Bacillus subtilis* (NCDO 736, ATCC 95244) spores at different temperatures and relatively low concentrations (table 1) (247).

Temp. °C	10%	15%	20%
25	1640	780	570
50	192	128	66
60	96	53	45
70	60	39	26
80	36	23	15

Table 1. *Time (seconds) needed to achieve 4 log. cycles reduction in the number of* Bacillus Subtilis *spores at different concentrations and temperatures of hydrogen peroxide.*

A review of the literature on the sporicidal action of hydrogen peroxide shows the effects of temperature and concentration (63). For a 10% solution of hydrogen peroxide at 60°C, the Q_{10}-value was about 1.6. Increases in concentration from 10% to 15% and from 15% to 20% each gave an increase of about 50% in the rate constant (247). At temperatures of 80°C, a 30% hydrogen peroxide solution achieved several decimal reductions in the bacterial endospore count (91) within seconds.

Microbiological Effectiveness of Hydrogen Peroxide

Comparing the resistance of *Clostridium botulinum* and *Bacillus* against the sporicidal action of hydrogen peroxide, the results shown in table 2 were obtained (148).

The sporicidal properties of hydrogen peroxide were evaluated at concentrations of 10-41% and at temperatures of 24-76°C. *Bacillus subtilis* SA 22 was the most resistant and *Staphylococcus aureus* the most sensitive of the organisms tested (251).

	Time (sec)			
	71.1°C		87.8°C	
Organism	**+**	**-**	**+**	**-**
Cl. botulinum	0	5	2	3
B. subtilis A	5	10	2	4
B. globigii			14	16
B.stearothermophilus			10	14

\+ = survival, growth;
\- = no growth

Table 2. *Comparison of the Resistance of* Cl. botulinum *and* Bacillus *Spores at 35% H_2O_2, 71.1°C and 87.8°C, 1 x 10^4 Spores*

3. Residual Hydrogen Peroxide

Hydrogen Peroxide Residues: Legal Limits

In packaging material (food contact surface) sterilisation, hydrogen peroxide concentrations of between 15% and 35% are typical (74). Whatever the system, whenever a chemical is used it cannot be removed entirely. Thus, a certain though minute amount of residue is unavoidable if hydrogen peroxide is applied. The question really becomes: What is an acceptable level of residue? In this respect, the legal authorities can be consulted. Some countries have passed legislation on the subject, others have not. Basically, the following situations are found:

- No regulations at all: the hydrogen peroxide residue is a matter of interpretation for the public health inspector.
- Existing legislation says: "hydrogen peroxide can be used in connection with food products providing the amount of residue in the product does not exceed the 'technically unavoidable level'". But what is 'technically unavoidable'?
- Hydrogen peroxide can be used in the food industry "providing it is eliminated or dissipated before the process is terminated". Two questions arise: a) What does "eliminated" mean? Obviously, it should be interpreted as meaning "not detectable by a reference method". The permitted residue of hydrogen peroxide is thus determined by the sensitivity of the official test method. If no such method exists, how much is permitted? Again, this is more or less a matter of interpretation for the public health officers. b) What does "terminated" mean? In the case of the TBA system, does it refer to the moment the transversal seal is effectuated, or to the moment the package leaves the final folder, or the storage area, or reaches the sales outlets, or to the moment its contents are consumed?
- A specified maximum permissible amount of hydrogen peroxide residue is regulated together with a reference method or methods (260).

At the present time, the US FDA regulation (21 CFR 177.1520) on residual hydrogen peroxide, if applied to the sterilisation of plastic surfaces of packaging material, is the most complete and best. The regulation was originally passed in 1981 (110) and states that hydrogen peroxide may be used, provided that (259):

- only those plastic surfaces listed in the regulation are treated;
- all additives used in the hydrogen peroxide (such as stabilisers and wetting agents, etc.,) are either permitted by a regulation or are GRAS (generally regarded as safe);
- the concentration of the hydrogen peroxide is within the regulated limits; and
- immediately after packaging, the residual amount of hydrogen peroxide does not exceed a regulated level (at present 0.5 ppm) as tested by one of two stated reference methods (67) (or an equivalent method).

All investigations carried out so far do not show any toxicity if reasonably small amounts of hydrogen peroxide are consumed. Larger quantities may lead to irritation of mucous membranes, but the effect is not chronic. Because the human tissues contain catalase and peroxidase, hydrogen peroxide is decomposed:

$$2\,H_2O_2 = 2\,H_2O + O_2$$

Hydrogen Peroxide: Toxicity

The toxicity of hydrogen peroxide has been studied extensively. The breakdown products of water and oxygen are harmless substances. Hydrogen peroxide does not accumulate in the body; chronic toxicity does not exist. Hydrogen peroxide can bleach hair and temporarily irritate the mucous membranes. Because of gas formation, the ingestion of large amounts of hydrogen peroxide may cause problems.

A test method for hydrogen peroxide residue based on peroxidase has been recommended (270).

The decomposition of hydrogen peroxide residue has been studied in raw and pasteurised milk and examined in detail in long-life milk (table 4).

Hydrogen Peroxide: Decomposition

Milk:	Raw					Pasteurised					Long-Life	
H_2O_2 µg/ml	5	10	20	40	80	5	10	20	40	80	2	5
0 min	-		+	+	+	-		+	+	+	+	+
5 min		-	+	+	+		-	+	+	+	+	+
10 min				+	+			+	+	+	+	+
20 min			-	+	+			+	+	+	+	+
40 min				+	+			-	+	+	+	+
60 min				-	+				+	+	+	+
90 min					+				+	+	+	+
120 min					-				+	+	+	+
180 min									+	+	+	+
240 min									+	+	+	+

+ = present (detectable); - = absent (not detectable) µg/ml = ppm = mg/litre

Table 3. *Decomposition of H_2O_2 in Milk*

H_2O_2 µg/ml	20	40	70	100	150	200	250
1 day	-		+	+	+	+	+
2 days		-	-		+	+	+
3 days				-		+	+
4 days					-	+	+
7 days						+	+
8 days							+
35 days							+

+ = present (detectable); - = absent (not detectable) µg/ml = ppm = mg/litre

Table 4. *Breakdown of H_2O_2 in Long-Life Milk at Ambient Temperature*

4. Hydrogen Peroxide in the Environment

Hydrogen Peroxide in the Environment

Another area of concern is environmental hydrogen peroxide, i.e., hydrogen peroxide present in the working environment. Again, in some countries this issue has become the object of regulation. The legislation usually states that:

- hydrogen peroxide in the air in areas where people visit or work for longer periods of time shall not exceed a maximum amount of 1 ppm.

Inhaled hydrogen peroxide, though not really toxic, irritates the mucous membranes (eyes, lungs, etc.,). Also, hydrogen peroxide is a bleaching agent and may affect the colour of the hair.

5. Packaging Material

Summary

Only paper-based laminates are discussed in this chapter. Whenever a food product comes into contact with a surface, be it packaging material or any other object, an interaction takes place: material from the surface enters the food product, and compounds of food can be taken up by the contact surface. The different interactions between packaging materials and packed food are also discussed. In aseptic packaging, attention must also be paid to the microbial load on the packaging material (food contact surface).

1. General

Different materials are used as aseptic packaging, such as:

- metal cans;
- glass bottles;
- pure plastic materials (bottles, cups, pouches);
- paper-based laminates, etc.

In the present chapter, only paper-based laminates will be discussed. From a functional point of view, the multi-layered structure (60, 69) is usually that shown in figure 1 (237).

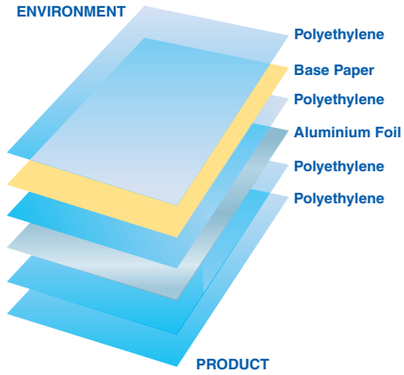


Figure 1. *Paper-Based Laminate: Packaging Material Structure*

Packaging Material: Structure and Function

An outer polyethylene layer provides protection against moisture from the environment. The base-paper layer gives stiffness and strength to the packaging material. The next layer of polyethylene ("laminating layer") serves to bond the base paper to the aluminium foil, which in turn functions as a barrier against the entry of light and gas and the passage of materials and product components from the inside. The first inner layer of polyethylene binds the last layer to the aluminium foil. To achieve proper adhesion, a high degree of oxidation is needed whenever polyethylene is used. Finally, the innermost layer, usually polyethylene, serves as sealing and provides the contact surface with the food. For it to function properly, the packaging material should be stored at the correct temperature and humidity as recommended by the manufacturer (142). Special composition of the packaging material structure may be required for some products, such as:

- fruit juices;
- tomato-based products;
- edible oils;
- mineral water;
- wine, etc.

The materials selected must be correlated with product and process factors (85). It is advisable to carry out storage tests in order to establish the practical shelf life of the products and packaging. Accelerated storage tests, i.e., using higher temperatures than those normally encountered, are usually not recommended since reactions that do not take place at the usual ambient temperatures may, and often will, be essential to product change.

Packaging Material: Selection

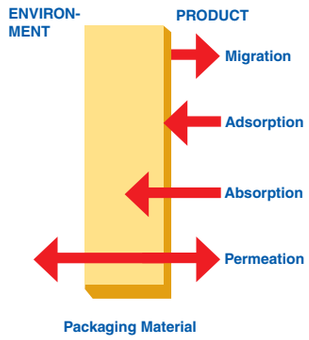


Figure 2. *Packaging Material and Product Interactions*

2. Interactions between the Product and the Packaging Material

Interaction always takes place between the packaging material and the packaged product. Four different types of interaction can be distinguished (figure 2).

2.1 Migration

Migration refers to compounds which originate in the packaging material and dissolve in the product. If aluminium foil is used in the composition of the packaging material, migration takes place mainly from the inside coatings. Migration may result from the:

1) polyethylene coating;
2) print; and/or
3) base paper.

Migration: Compounds into the Product

Migration: Legal Requirements

Existing legal requirements relate to toxicity, recognisable flavour changes and "global migration", i.e., the total amount of material which passes under specified conditions from the packaging material into test liquids (water or oil).

2.1.1 Migration from the Polyethylene Coating

Global Migration Legal Limit: ~ 60 ppm Actual: 0.5 – 5 ppm

Organic polymer materials such as polyethylene are very strictly regulated regarding the amount and kind of migrating products. As a limit for global migration from polyethylene into food after 10 days of storage at 40°C, a level of 60 ppm has been set in a European Union directive. A typical level found for aseptic packaging material is 5 ppm maximum (237). However, global migration values of 0.5 ppm are not uncommon. Such values are well below the legally established limits.

Migrating Products: Toxicity and Flavour

In addition, migrating products must be non-toxic. Polyethylene has been studied extensively in this respect. So far, neither acute nor chronic toxicity has been shown. Actually, ethylene-oxide has been used for a long time to prolong the storage time of fruit, especially apples. Migrating products should not have a recognisable effect on the flavour of the packaged product. The lower the flavour profile of a food commodity, the more sensitive it is. Recognisable flavour changes are caused by small amounts of migrating compounds already. In flavoured products, the aroma of the food masks or covers the change in flavour. In this respect, water is the most difficult of products. Special packaging materials may be needed in order to prevent flavour defects.

Migration: Effect of Treatment Prior to Use

Polyethylene grades without additives which are used in the manufacture of packaging materials do not create problems with regard to the specific migration of components such as slipping agents, plasticisers or antioxidants.

If, prior to use, the packaging material is to be sterilised, either by heat alone or by a combination of heat and chemicals, as for example in aseptic packaging, the effect of the sterilisation process on migration has to be fully understood (234), tested and demonstrated. Such procedures may alter the contact surfaces of the material which, in turn, may affect migration.

2.1.2 Migration from the Print

Printing may form part of the packaging material structure in different ways. Two examples are shown below (figure 3).

Migration from the Ink

Depending upon the kind of packaging material (blanks, reels, etc.,) and its structure, direct or indirect migration from the printing layer is possible. Consequently, attention has to be paid to the composition of the ink as well as to the solvents used. Aspects of toxicity and possible sensory effects must be considered. This is especially true if the packaging material structure does not include aluminium foil.

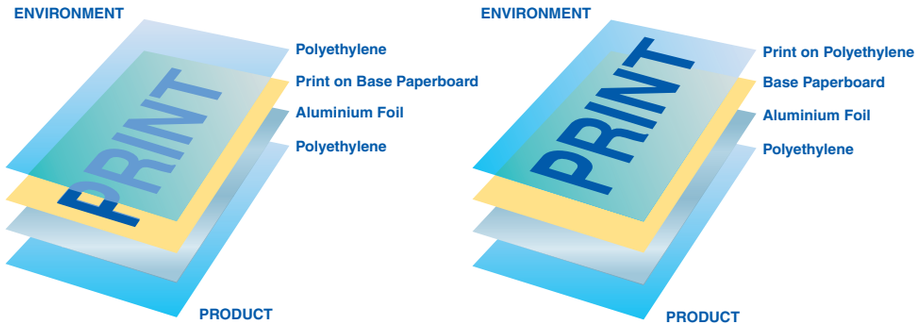


Figure 3. *Packaging Material Structure*

2.1.3 Migration from the Base Paperboard

Migration from the Base Paper

Even though direct contact between the layer of base paperboard and the product is prevented by plastic coatings and, where applicable, by the aluminium foil, the possibility of indirect migration should not be excluded. Two factors should be considered:

a) the water content of the paperboard; and

b) chemicals.

High moisture (above ~ 9%) may result in recognisable flavour changes due to compounds from the base paperboard migrating via the inner plastic coating into the packed product.

Chemicals may originate from recycled fibres, biocides used in the paper mill, and other sources. Greater concern for the environment has led to the extended use of recycled paper and the introduction of closed water circuits in paper factories. This requires careful attention in the choice of return paper to be used as well as to standards of hygiene in the manufacture of the board.

2.2 Adsorption

Adsorption = Compounds Sticking on to a Surface

Adsorption is the adherence of product ingredients to the inner surface of the packaging material. This is often observed with lipid materials, especially milk fat, and may cause problems with products having a high fat content, particularly whipping cream. Proper homogenisation of the product reduces or eliminates this fault.

2.3 Absorption

Absorption = Compounds Dissolved into the Packaging Material

In the case of absorption, compounds of the product are dissolved into the packaging material structure. An example of absorption is the loss of limonene in orange juice (108, 181, 237). Such a phenomenon is characterised in figure 4. At the beginning of the storage time, the compound dissolves into the inner coating. As time passes, more and more of the compound is taken up by the material. On the other hand, as the concentration of the dissolved compound increases in the plastic layer, more and more of it is released back into the product. Eventually an equilibrium is reached and no further changes in concentration can be registered. The amount and kind of absorbed material depend upon the thickness and type of the plastic layer (237). Adding the compound in question to the product prior to processing and packaging can compensate for the amount that has dissolved into the plastic coating.

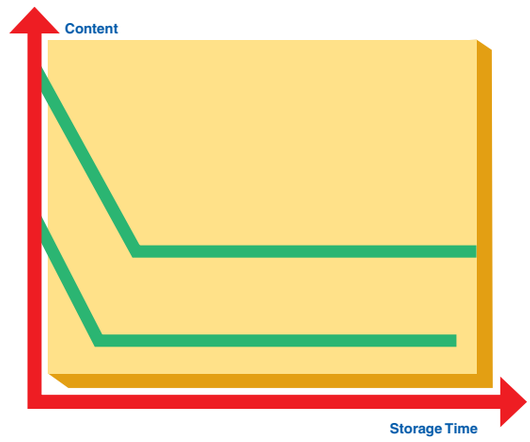


Figure 4. *Typical Absorption Curve*

2.4 Permeation

Permeation = Compounds Passing through the Packaging Material

Permeation describes the passage of product components through the package from within and without. As far as penetration and the passage of gas in particular are concerned, a distinction should be made between the tightness of the packaging material and the tightness of the package. Often the packaging material is much tighter than the package.

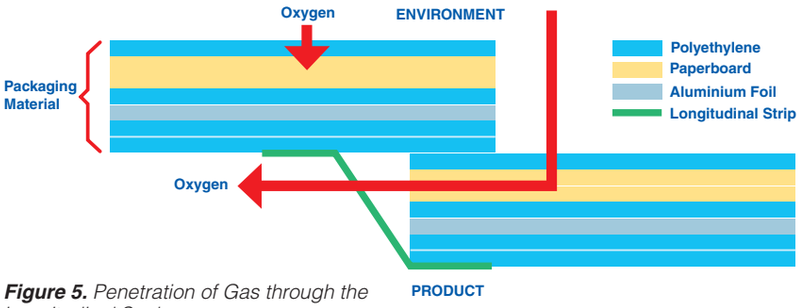


Figure 5. *Penetration of Gas through the Longitudinal Seal*

Permeation of Gas (Oxygen)

Oxygen Permeation into Aseptic Packages

Gas permeation is possible through the seams of a container. In the structure shown in figure 5, oxygen permeation becomes possible through the longitudinal seam. The barrier characteristics of the package are determined more by the tightness of the longitudinal strip material than by that of the packaging material itself (128). A polyethylene - polypropylene - polyethylene strip provides better barrier characteristics than a low density - high density - low density polyethylene strip. Multi-layered laminates with good gas barrier characteristics limit the passage of, for example, oxygen.

Gas permeation through the transversal seal is less important. The bonded plastic material in the transversal seal area has a very thin diameter and a rather large thickness (figure 6). Gas permeation is slow.

A method has been suggested for checking the permeation of gas through materials lined with aluminium foil. Oxygen permeability of the packages produced (250 ml) was found to be less than 0.17 ml O_2 per month (85). The oxygen permeation values for different aseptic packages are given in table 1 (198).

The transmission of limited amounts of oxygen through the carton is possible mainly through folds, seals, lids (if applicable) and creases. 250 ml Tetra Brik aseptic cartons were filled with deaerated water and the increase in oxygen content was measured at different storage temperatures (figure 7). The rate of transmission increased over time and at higher temperatures (237).

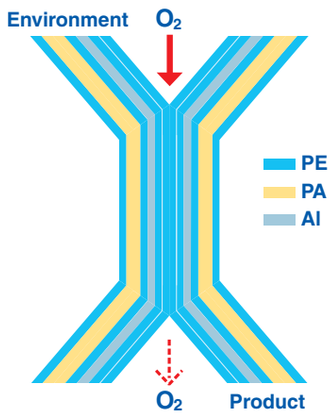


Figure 6. *Permeation of Gas through the Transversal Seal*

Package Design	Volume	O_2 Penetration mg/l/ year
Tetra Brik	1,000 ml	3.7
Combibloc	1,000 ml	2.2 - 7.5
Hypa S	700 ml	1.5 - 2.2
Glass Bottle	1,000 ml	1.5 - 7.3

Table 1. *Oxygen Permeation Values*

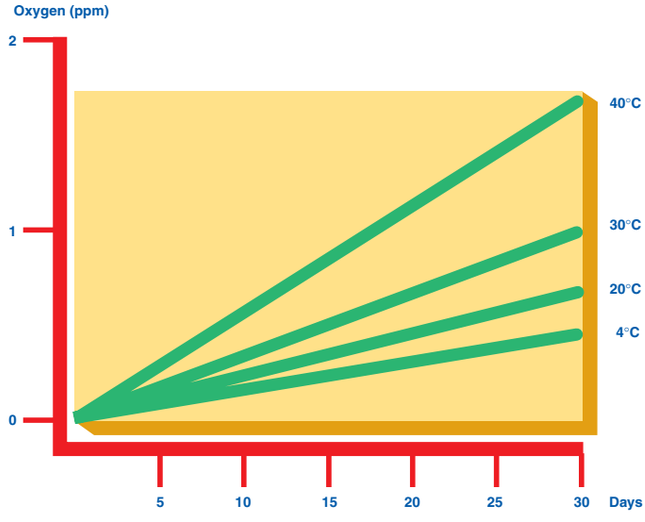


Figure 7. *Oxygen Permeation at Different Temperatures*

3. Microbiological Aspects

Microbiology of the Base Paper

The microflora of the base paper used in the manufacture of laminated packaging material is dominated by Gram positive bacteria. Gram negative microorganisms have not been found. In the production process, the Gram negative flora is practically eliminated during the drying phase of carton. More than half the samples tested had a total count of <200/g (64). Closed water systems in paper mills, limitations on the use of biocides, and the reprocessing of waste paper cause considerable microbiological loading in the process water. Dissolved material leads to the proliferation of microorganisms. Aeration of the water system, integrated use of biological filters, improved hygiene and the well-balanced use of biocides allowed the control of colony counts in the process water, which alleviated operating problems and avoided a deterioration in paper quality (123).

Extruded plastic materials and laminates are usually sterile (74, 136) because of the heat applied during the converting process (86). However, after processing, a limited airborne infection takes place. The amount and kind of micro-organisms depend upon the level of hygiene prevailing at the converting plant. The microbiological load of the packaging material's food contact surface should not exceed a total colony count of 5/100 cm^2 (142) or should be less than 1/100 cm^2 (12). On plastic materials, total counts of 0.4 to 10/100 cm^2 (12), 0 to 10/100 cm^2 (76) and 2 to 10/100 cm^2 have been mentioned. Total counts on plastic food-contact surfaces of less than 1/m^2 have also been reported (230). If "aseptic" conditions after extrusion are observed, counts as low as 0.2 to 0.8/100 cm^2 could be obtained (263). The total microbial count found on food contact surfaces of paper-based plastic laminates ranged from 0 to 5 colony forming units/100 cm^2. This flora had the composition of that shown in table 2 (60, 61). The results were obtained immediately after the packaging material had been produced.

Microbiology of the Food Contact Surface

Attention should be paid to the effects of storage and additional infection taking place at the site where the filler is operated and the material used. Changes in the amount and composition of this bacterial flora during storage have not been studied. Often, hydrogen peroxide is used for the sterilisation of packaging material (food contact) surfaces (86). The sterilising effect necessary for the sterilisation of the packaging material (food contact surface) depends upon the size of the pack (contact surface), the surface contamination and the acceptance quality level (the AQL) (76). The minimal decimal reduction values necessary for sterilising the packaging material (food contact) surface vary between 4 and 6, as tested with spores of *Bacillus subtilis* A (134). Four decimal reductions have also been suggested (91). *Clostridium botulinum* spores are reported to be 2.5 times more sensitive to the lethal action of hydrogen peroxide than spores of *Bacillus subtilis*: 4 decimal reductions obtained with *Bacillus subtilis* corresponded to 10 for *Clostridium botulinum* (76). To achieve a result of one failure per 10,000 packages, a minimum sterilising effect of 4 is needed (76). Such statements are meaningless unless attention is also paid to the quality of the intermediate product (bacterial spore counts) as well as to the general hygienic conditions prevailing in the area where sensitive materials such as the packaging material are handled and used. Defect rates achieved by an aseptic production line are not determined solely by the aseptic packaging operation.

Microorganisms	Percentage
Yeast	10.6
Moulds	20.6
Bacteria	68.8
Micrococci	44.4
Bacterial spores	3.1
Streptococci	3.7
Pseudomonas	1.2
Gram Positive Rods	6.9
Gram Negative Rods	9.4

Table 2. *Composition of the Microflora on Plastic Packaging Material*

6. Application of Microbiology to UHT Processing and Aseptic Packaging

Summary

Microorganisms are used by the food industry in the production of certain food products. Some microorganisms can cause disease in human beings (pathogenic microorganisms cause disease or food poisoning). Depending on the kind of food, most fungi and bacteria can cause food spoilage.

Microorganisms of interest to aseptic technology are fungi (moulds and yeast) and bacteria. These groups of organisms are discussed in general. Some aspects of product and packaging material sterilisation are touched upon.

1. General

Microbiology covers a wide range of organisms invisible to the human eye. These organisms can be divided into the following main groups: protozoa, algae, fungi (moulds, yeast), bacteria and viruses. The groups of protozoa, algae and viruses are of no or very limited interest to aseptic technology and will not be dealt with further.

1.1 Moulds

Moulds are a large group of microorganisms. They can multiply at a wide range of pH (~ 1.5 - ~ 8), at low water activity (a_w), at relatively low temperatures and still have low nutritional requirements. However, oxygen is needed for growth. Moulds form typical structures of growth and visual identification is possible down to genera by microscopy. In the lack or absence of oxygen (as in submersed cultures), growth is slow and spores do not usually form (figure 1).

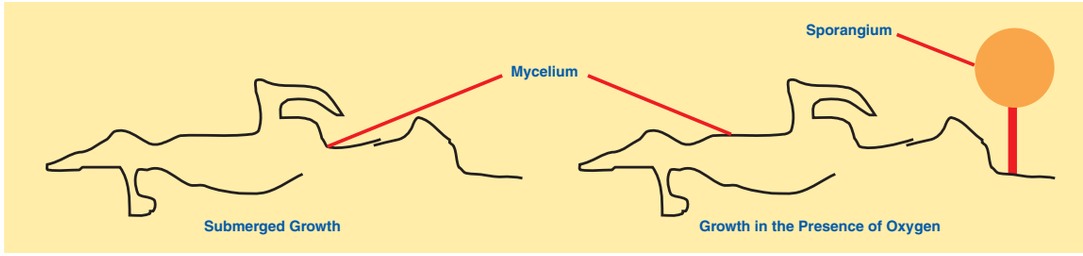


Figure 1. *Typical Growth of Moulds*

Moulds: Growth and Multiplication

Though rather thin (diameter a few µ), the threads (hyphae) can become very long. In the presence of oxygen, specific structures (sporangium) are formed which may contain a large number of spores. After a certain period of time, the spores are released simultaneously into the environment: the spreading of mould is explosive! Thus, whenever a small spot of mould growth is observed in a factory it must be removed immediately. These spores are not especially resistant to heat or chemicals.

Under certain conditions, some species can form other relatively resistant spores (ascospores, chlamydospores) that have been the cause of problems in the fruit processing industry for a long time.

In the food industry, moulds are used in the production of some cheeses (Camembert, blue cheese and some others). They are also an important source of antibiotics. However, certain species produce toxins (myco-toxins) which accumulate in the human body and can cause cancer.

1.2 Yeast

Yeast: Growth and Multiplication

Yeasts usually form oval cells with a diameter of about 3 to 6µ and a length of 5 to 10µ. They grow in the temperature range of 10-30°C, and are capable of development at low pH. Some tolerate very low water activity (a_w 0.6), but their nutritional demands are higher than those of moulds. Typically, they multiply by budding, i.e., forming a bud on the mother cell. This bud increases in size and eventually separates from the mother cell (figure 2). A scar is left on the surface membrane of the mother cell limiting the number of "daughters" that can be produced. One cell can form about 8-10 daughter cells before multiplication stops.

Some yeasts are used in the food industry for cultured milk products ("kefir", and others), and in the production of alcohol.

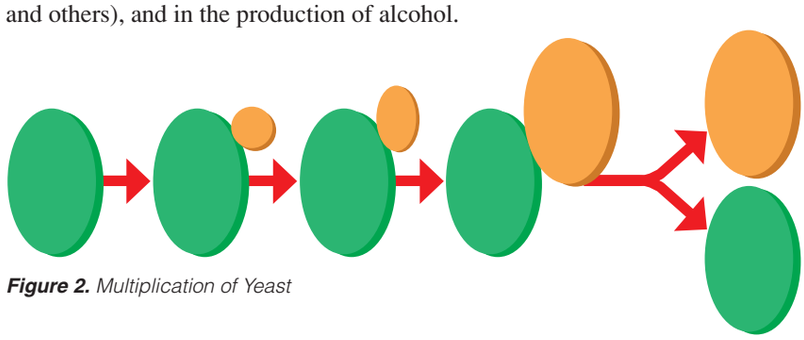


Figure 2. *Multiplication of Yeast*

1.3 Bacteria

Bacteria constitute a very large group of microorganisms. In nature, they are present everywhere. Their need for oxygen differs: some will not grow in the presence of oxygen (they are anaerobic), others require it (aerobic). A third group, the micro-aerophilic bacteria, need small amounts of oxygen. Most bacteria will not multiply at an a_w below 0.9. Their nutritional demands vary widely. In nature, bacteria play a vital role by decomposing organic material. In industry, they can spoil food products. However, some bacteria are needed in the manufacture of foodstuffs such as fermented milk products and others.

Bacteria: Shape of Cells

Bacteria have basically three different shapes:

a) small balls (cocci);

b) straight rods; and

c) bent rods.

Bacteria: Speed of Multiplication

Bacteria multiply by cell division (figure 3).

Under favourable conditions, the speed of multiplication can be very high. It may take only 10 to 15 minutes for one cell to become two. The same length of time is required for 1 million to become 2 million! This unit of time is called generation time.

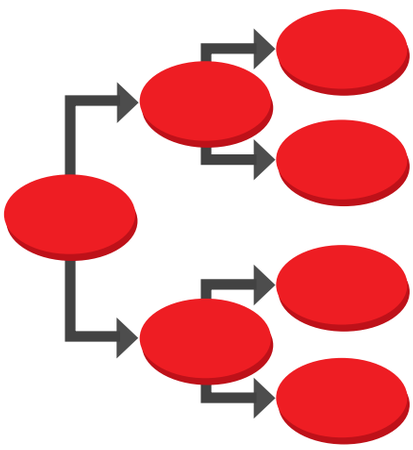


Figure 3. *Multiplication of Bacteria*

For practical reasons, bacteria may be divided into two groups:

a) non-spore forming, vegetative bacteria are usually easily killed by chemical and physical means; and

b) spore-formers. These can develop resting forms - endospores - which are very resistant to heat and chemicals. They can survive long periods in unfavourable conditions.

Gram-Stain: A means of Classification

By using a special staining procedure, a distinction can be made in the classification of bacteria between Gram positive and Gram negative bacteria . (Gram is the name of a Danish physician who, at the beginning of this century, tried to colour bacteria in human tissue). The difference in staining behaviour is due to differences in the cell membrane structure. The cell membrane of Gram positive bacteria consists mainly of protein, while the membrane of Gram negative organisms contains lipids. Generally, Gram positive bacteria are more difficult to kill than Gram negative.

1.4 UHT Processing and Aseptic Packaging

Sterilisation: Low-Acid and High-Acid

In UHT processing and aseptic packaging technology, a number of different sterilisation processes are involved (figure 4) (61).

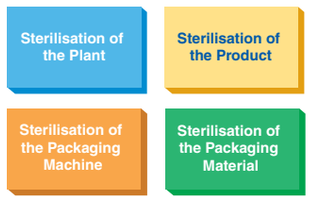


Figure 4. *Production of Long-Life Products: Sterilisation Cycles Involved*

The purpose of the sterilisation process is to achieve commercial sterility of the product, equipment or any other object. To this end, either physical (thermal, radiation) or chemical processes can be used: all microorganisms which can multiply in the product with spoilage as a result should be eliminated. For high-acid products, the killing of yeast, moulds and some vegetative bacteria is sufficient. Low-acid food products require even the elimination of bacterial endospores produced by two genera: *Bacillus* and *Clostridium*. Spores are characterised by high resistance to thermal and chemical sterilisation processes. Spores are resting forms which do not multiply. However, they can survive for very long periods of time even under very unfavourable conditions. Deeper and deeper dormancy combined with increasing resistance results from persistently detrimental conditions (figure 5). If the environment becomes more acceptable, the spores start the germination mechanism, a transition from the resting (sleeping) spore to the vegetative state (figure 5) which is capable of multiplication.

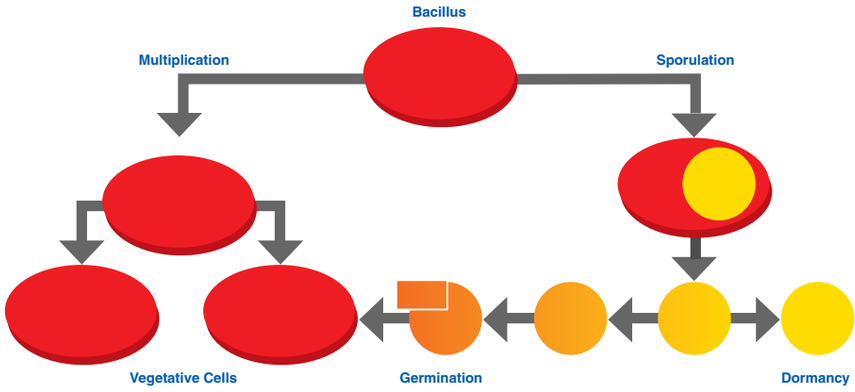


Figure 5. *Bacterial Endospores, Sporulation, Dormancy and Germination*

The first step of spore germination is the intake of water. This process renders the spores more sensitive, particularly to thermal sterilisation.

Since sterilisation processes achieve a certain number of decimal reductions in bacterial endospore counts (which in turn depend upon the process parameters *and* the resistance of the spores), the microbiological result which is obtained from such a process is determined by:

Thermal Process Parameters: Time and Temperature

- the process parameters;
- the microbiological load, i.e., the number; and
- the kind and resistance of bacterial spores fed into the sterilisation process (224).

For thermal processes, the following parameters need to be considered:

- time; and
- temperature.

With this background, line performance guarantees can only be given if a large number of essential parameters are clearly specified. Not all of these are controlled by the equipment supplier. Nevertheless, microbiological performance guarantees have been given by manufacturers of aseptic packaging equipment of 1 defective unit per 10,000 units produced (14).

2. Product Sterilisation

Q_{10}-Value: Chemical: 2–3 Killing: 10–30

The Q_{10}-value of chemical reactions is usually between 2 and 3. The killing of bacterial spores is characterised by a higher Q_{10}-value, reportedly between 8 and 30 (81): with increasing temperature, the killing rate of microorganisms increases

much more rapidly than the chemical changes inflicted by the process. Consequently, for microbiologically equivalent processes (same number of decimal reductions, same sterilising effect), the higher the sterilisation temperature and the shorter the holding time, the less chemical changes are encountered.

The sterilising effect obtained by a given, defined UHT treatment is determined by the process parameters (time and temperature), the kind and dormancy of the spores and by product characteristics such as pH, a_w, viscosity, etc. For a given product, the result obtained can be expressed by a simple equation:

Process Survivors = Process Parameters + Microbial Load.

Raw Materials: Ingredients + Other Materials

The bacterial load (spore count) is determined by:

a) the quality of the raw materials used; and

b) the level of infection, multiplication and sporulation during pre-treatment.

Raw materials are the ingredients used in the formulation of a product and the materials required for its manufacture. This includes the packaging material. The microbiological effectiveness of the UHT process can be improved by increasing the temperature and/or the holding time. Because of increased deposit formation, production times are reduced and the severity of chemical change increases. (It should be borne in mind that the efficiency of the packaging material sterilisation process normally cannot be increased without causing negative side-effects which surpass by far the microbiological benefits.)

Bacterial Spores in Milk

Product	Fat %	Spore Count	Fat %	Spore Count
Raw Milk	4.1	249/g	4.2	89/g
Skimmed Milk	0.1	224/g	0.1	86/g
Cream	47.0	29/g	55.3	20/g
Sludge	-	12,800/g	-	6,100/g

Table 1. *Mesophilic Spores in Milk Products*

Product	Fat %	Spore Count	Fat %	Spore Count
Raw Milk	4.3	4.3×10^6	4.2	9.5×10^3
Skimmed Milk	0.1	9.1×10^5	0.04	9.3×10^2
Cream	33.8	1.8×10^5	32.0	1.0×10^2
Sludge	-	5.0×10^8	-	2.8×10^9

Table 2. *Thermophilic Spores in Milk Products*

The mesophilic spore count in untreated milk (England) exhibited a seasonal variation, the counts being higher in winter than in summer. Spore counts in untreated milk ranged from 0 to 700/100 ml. The incidence of the different species was: *B. licheniformis*: 62%; *B. brevis*: 15%; *B. subtilis*: 7%; and others: 9% (5).

3. Packaging Material Sterilisation

The term "aseptic packaging" has not been clearly defined. It has been stated (93) that any company can market a filler equipped with a packaging material sterilisation system as an aseptic one. How effective the sterilisation process is, another question.

The death rate of microorganisms in general and bacterial endospores in particular often, but not always (87), follows a semi-logarithmic function regardless of whether thermal or chemical processes are used (63, 90, 247). As with thermal treatment, the result achieved by a given chemical sterilisation procedure is determined by the process as such and the number, kind and resistance of microorganisms (bacterial spores) subjected to the process (13):

Process Survivors = Process Parameters + Microbial Load

Bacterial Spores: Resistance to Hydrogen Peroxide

Variable survivor curves were found for a number of Bacillus and Clostridium spores exposed to hydrogen peroxide (148). Tailing of death curves has been observed, which is explained by lump formation (88). Bacillus subtilis A, Bacillus subtilis SA 22, and Bacillus subtilis (globigii) have a high resistance against hydrogen peroxide. Therefore, these have often been chosen as test organisms (149). The relative resistance at 24°C to 25.8% hydrogen peroxide was: Bacillus subtilis SA22 >Bacillus subtilis var. globigii >Bacillus coagulans >Bacillus stearothermophilus >Clostridium sp. >Staphylococcus aureus, with "D"-values of 7.3, 2.0, 1.8, 1.5, 0.8 and 0.2 minutes respectively (251). Age of the spores did not affect their sensitivity but dry spores were about twice as

resistant as wet ones (166, 167). The opposite effect, however, has also been reported (251). Nevertheless, the efficiency of a specific sterilisation process is characterised by a sterilising effect which can be expressed in the number of decimal reductions in bacterial spore counts achieved by the procedure. The methods and procedures for such tests have been described (21).

Reactivation of *Bacillus subtilis* spores treated with a 15-35% solution of hydrogen peroxide at 24°C has been observed. More than 90% of the spores could be reactivated by subsequent heat treatment at 80°C for 5 minutes. However, heating to 100°C resulted in permanent inactivation (89), a temperature which is reached in most packaging material sterilisation procedures.

In aseptic packaging systems, with few exceptions, the packaging material (food contact surface) is sterilised by the application of hydrogen peroxide (93), a chemical process. The following parameters need to be considered:

- the kind of chemical used;
- the concentration of the chemical;
- the contact between the chemical and the object to be sterilised;
- the contact time; and
- the temperature during contact.

Chemical Process Parameters: Chemical Concentration Contact Contact-Time Temperature

As pointed out elsewhere, spray application does not result in total coverage of the food contact surfaces. Depending upon the construction of the spray nozzles, only 30-40% of the surface is covered with hydrogen peroxide. However, subsequent heating using hot (~ 180°C) sterile air in order to eliminate the sterilant from the packages, thus reducing hydrogen peroxide residue to an acceptable level, results in satisfactory sterilisation because of the increase in temperature (93) and the development of hydrogen peroxide vapour which facilitates the necessary contact.

For optimum microbiocidal effects, hydrogen peroxide solutions in concentrations of about 50% (w/v) are recommended. A rapid destruction of the most resistant *Bacillus* spores has only been observed at temperatures around or above 80°C. Mould spores, vegetative bacteria and yeast cells are easily killed by hydrogen peroxide at far lower temperatures (90). D-values of *Bacillus subtilis* spores decreased rapidly with increasing concentration and temperature (table 3) (167).

% H_2O_2	20°C	30°C	40°C
5.9	38.9	10.7	3.1
11.3	16.2	5.1	1.6
17.7	9.3	2.9	1.0
23.6	5.6	2.0	0.9

Table 3. *D-Values for* Bacillus subtilis *Spores*

Another, earlier study (247) showed a similar result (table 4).

For different temperatures and concentrations, Q_{10}-values reported are fairly constant ranging from 1.36 to 1.74 (247).

Clostridium botulinum was found to be more sensitive to the action of 35% hydrogen peroxide than *Bacillus subtilis* (table 5) (148).

Temp. °C	10% H_2O_2	15% H_2O_2	20% H_2O_2
25	1640	780	570
50	192	128	66
60	96	53	45
70	60	39	26
80	36	23	15

Table 4. *Time (sec) for Reduction of 4 log Cycles of* Bacillus subtilis *spores.*

Spores	71.5°C	71.5°C	87.8°C	87.8°C
Cl.botulinum 169B	0	5	2	3
B.subtilits A	5	10	2	4
B.subtilits (globigii)	-	-	14	16
B.stearothermophilus 1518	-	-	10	14

Table 5. *Time in Seconds Necessary to Kill 10^6 Spores at Different Temperatures (35% H_2O_2)*

Using hydrogen peroxide as a sterilant, the following D-values and z-values were found (table 6) (252).

Organism	% H_2O_2	Temp.°C	D-Value	z-Value
B.subtilis A	26	25	7.3	24
B.subtilis ATCC 9374	26	25	2.0	
B.coagulans	26	25	1.8	
B.stearothermophilus	26	25	1.5	
B.subtilis ATCC 95244	20	25	1.5	47
B.subtilis	25	25	3.5	
Cl.botulinum 169B	35	88	0.03	29

Table 6. *D-Values and z-Values for Different Microorganisms*

The US FDA requires a minimum of four decimal reductions for packaging material sterilisation as tested with *Bacillus subtilis* spores (personal communication).

Synergistic Effect: H_2O_2 + UV-Light

The sterilisation efficiency of low concentrations of hydrogen peroxide can be drastically increased through combination with ultraviolet irradiation. UV-irradi-ation of spores in the presence of hydrogen peroxide produced a kill which was about 2,000-fold greater than that produced by irradiation alone. Ultraviolet irradiation of spores in the presence of 2.5g H_2O_2/100 ml water followed by heating to 85°C produced a kill of at least 99.99% (4 decimal reductions) in all 15 strains of *Bacillus* and *Clostridium* examined (44, 45). The combined effect of UV-light and hydrogen peroxide was less pronounced in systems in which the chemical is sprayed on to plastic surfaces. The explanation is found in the partial coverage of these surfaces (148, 251).

7. Installation

Summary

The installation of equipment is important not only with respect to available space and appearance, but also with regard to the operative and qualitative results obtained. Attention should be paid to *all* aspects, some of which are discussed bellow: the factory surroundings, the layout of the plant, the choice of equipment and its installation. Factors having an impact on microbiological performance are also discussed.

1. General

When planning, erecting and installing a plant, all legislative requirements pertaining to the surroundings, building exteriors and interiors, and equipment have to be followed.

The planning of any plant, and in particular a UHT plant, demands very careful consideration of the following factors (32):

Considerations When Planning a UHT Plant

a) a feasibility study to ascertain whether long-life milk and/or UHT dairy products are likely to fulfil a demand and whether there is a possibility of greater demand in the future;

b) the capital sum which is available or can be provided for the project, and an estimate of the likely expenditure in order to assess the viability of the project;

c) an economic assessment of the installation and running costs to provide information on profitability;

d) the location of the plant in relation to: (i) the area from which the milk is collected or from which it is supplied, and (ii) the distribution area of the packaged long-life milk or milk products taking into account climatic conditions, possible difficulties in terrain, seasonal variations in milk supply, etc.;

e) the availability of technical, skilled and unskilled personnel and professional advice in connection with any contractual work which may be involved, and with the operation and maintenance of the processing plant and ancillary equipment;

f) the facilities which exist in the area for water, fuel and electrical supplies, and for the disposal of effluent, both with regard to immediate needs and possible future requirements.

The fundamentals of quality assurance are laid down at the drawing board stage of a project and, to a large extent, become integrated when the plant is erected and installed. The design, capacity and siting of the individual units, and the way in which they are integrated and connected with one another to form a whole plant, are some of the many factors that can have a permanent effect on the standards of quality (33).

Optimal Installation Requires Definition of the Goal

An installation should be adequate for its intended purpose. Thus, an optimal installation can only be designed if the goal is clearly specified. This is often done with respect to quantity but not always as far as quality aspects are concerned. Product quality specifications should preferably be decided upon even before a decision has been made concerning the site where the factory is going to be built. Obviously, the requirements placed on the surroundings and installation depend upon what ever it is one wants to achieve, i.e., among other things, the AQLs (acceptance quality levels).

2. Factory Surroundings

External Requirements

The external environment will have an impact on the quality of the manufactured product(s). Processing and manufacturing premises should be located in areas which are free and likely to remain free from flooding, objectionable odours, smoke, dust or other contaminants (10). Attention should be paid to:

- dust which, depending upon prevailing wind directions, may enter the plant in unacceptable quantities causing microbiological and other quality problems;
- polluted water from sewage, and other household and factory effluents which may form heavily infected aerosols, sometimes even with pathogenic microorganisms;
- water purification plants producing massively infected aerosols which concentrate microorganisms;
- the presence of a slaughterhouse, the effluent from which is usually seriously infected, even with pathogenic microorganisms, and thus may imply a public health risk. In addition, the smell may also affect the quality of the products;
- pests (insects, rodents, etc.,) which may enter the factory and cause unnecessary product losses.

The Immediate Surroundings

The immediate surroundings of any food-producing factory and particularly of plants producing long-life products should be clean and kept so by proper housekeeping procedures. The area immediately adjacent to the factory should be paved with concrete or other suitable material to give a hard, cleanable surface. All unnecessary pollution must be avoided. Waste material, pallets, market returns, spoiled products, etc., should be removed quickly from the vicinity of the building. The handling of product returns, spoiled and/or outdated products should be performed in a specified area separate from the immediate surroundings of the plant.

3. The Plant Layout

The Building: Requirements

It is important that the building is designed and finished in such a manner that it facilitates cleaning and that it can be kept tidy at all times. Internally, the walls should be smooth, non-porous, and crevice-free. The floors should be drainable and resistant to attack or corrosion by acid and alkaline solutions as well as by product residues. The building should be adequately protected against the entry of rodents, insects, birds, and other animals (34).

Housekeeping

The plant should be laid out in such a way that the floor and walls around the processing equipment, storage tanks, etc., are easily accessible for cleaning and housekeeping. Wherever possible, equipment should be raised above floor level to permit underside surfaces and the floor itself to be cleaned (34). Equipment should not be placed on top of drains. Consideration should be given to the sewage system, the placement of gullies, the product flow and ventilation. “Open” and “closed” operations should be clearly identified. In open operations, the product is not separate from the environment; the surrounding air has free access to the product. In closed operations the product is protected and separated from the environment. “Open” operations should be connected to the main effluent duct upstream to “closed” operations (figure 1).

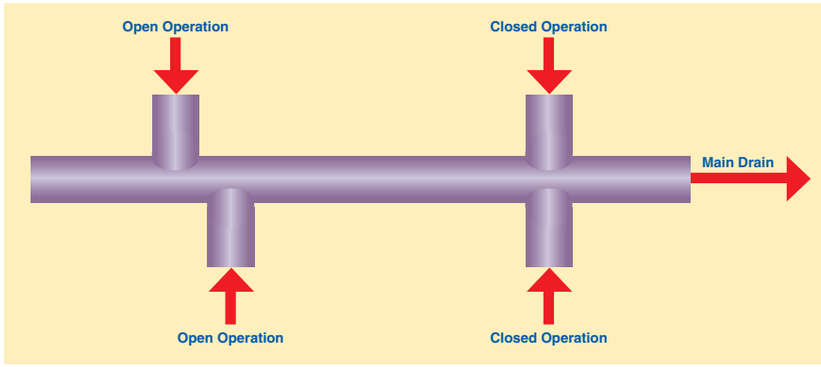


Figure 1. *Drainage System*

Drainage System

Effluent, waste products, etc., should be removed from the aseptic filling area via open ducts covered with a removable grate. In the aseptic filling room, gullies should be avoided as much as possible. They must be placed and constructed in such a way that they are easily accessible for cleaning and disinfection, and not covered by equipment.

Dry floor conditions are of importance in order to avoid aerosol formation by splashing and/or injury to personnel by slipping. For the installation of aseptic filling machines, the following arrangement (figure 2) could be considered.

The filling machine could be surrounded by a small "bulwark" which guides any waste water on the floor to the drain.

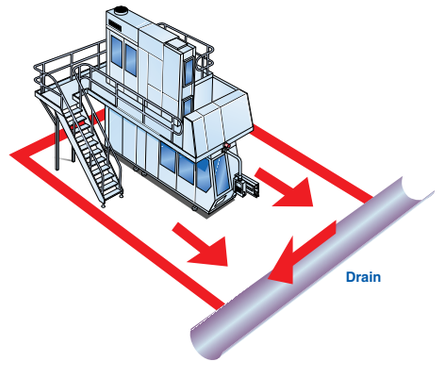


Figure 2. *Filling Machine: Installation to Reduce Water*

Packaging Material

Packaging material should be stored in areas which are specifically designed for the purpose and in accordance with the recommendations of the supplier. Raw material (ingredients) storage and handling as well as the formulation of products, particularly if these contain powders, must be kept separate from the storage of the intermediate product and from UHT processing and aseptic packaging. Any installation for the production of long-life products should be kept distinctly separate from other activities in the factory (32). The product flow should be arranged not only with economy in mind but also with regard to hygienic requirements. It is highly recommended that a separate room for the aseptic filling operation (figure 3) be provided. Proper ventilation with reasonably clean, filtered air should create a slight overpressure in the aseptic filling area.

Filling Room

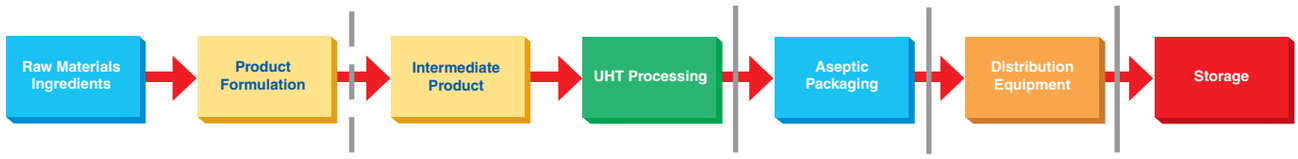


Figure 3. *Flow Chart: Separation of Areas*

Ventilation

For ventilation, the air intake should be located in such a way that any unnecessary contamination from microorganisms, dust and other pollutants is kept to the minimum.

The air intake should be situated in the wall (not the roof) of the factory in a position opposite to the prevailing wind direction (figure 4). An air filter for the removal of dust and other particles should be provided.

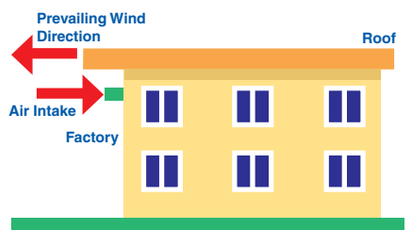


Figure 4. *Air Intake: Wind Direction*

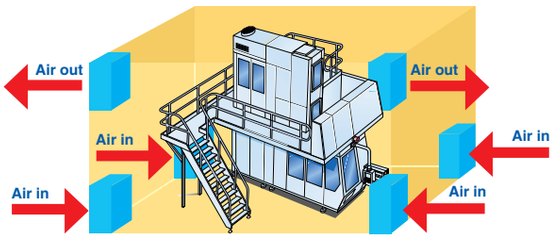


Figure 5. *Filling Room: Air in and Air out*

For the aseptic filling area, a filter size of 2 to 5 µ is recommended and is normally sufficient. In the filling room the air should enter at a low level and leave at a high level (figure 5).

The air flow should be slow, and laminar and should not exceed 0.8 metre/second. If necessary, air conditioning can be installed in the filling room. Space for later extensions of production capacity should be provided.

The floor of the filling room should be covered with acid-resistant tiles and have an incline of 1:50 towards an open drain. Recommended drain measurements are 100 mm in depth and 300 mm in width.

A disinfection mat at the entrance is recommended. For very ambitious quality requirements (low microbiological AQL), "clean room" conditions may be necessary.

Clean Room?

Installation requirements in general and for the filling room in particular depend upon the AQL (acceptance quality level). The higher the demands on the microbiological performance of a production line, the more requirements have to be placed on the quality of the installation.

Lighting

Lighting must be adequate to permit machine operators to check package integrity effectively and carry out other necessary duties. 500 lux at a working height of 4 meters is a recommended minimum value.

The area in which the distribution equipment is installed should provide stacking space for packages which may be needed during stops for servicing and breakdowns, etc.

The storage area for the finished product should have cleanable floors and walls. Its size should be sufficient to permit production planning.

Microbiological Conditions

The microbiological conditions prevailing in sensitive areas such as the filling room should be checked by adequate methods. Special sampling tools are available for this purpose. Fallout (settle) plates may be used. Several Petri dishes containing appropriate media are exposed for a given time and incubated. The colonies are then counted (222). Acceptable counts are determined by the AQL, i.e., microbiological performance requirement, but (for low-acid food products) they should not exceed 50 cfu (colony forming units) on a normal Petri plate (diameter: 9 cm) after 15 minutes of exposure.

4. The Equipment

When choosing equipment for pre-processing, UHT treatment, aseptic packaging, and outer wrapping, etc., attention should not only be given to the cost factor. Service availability, quality performance levels, etc., should also be considered.

The equipment must be of hygienic design and suitable for "cleaning in place" (CIP). It must also be as "closed" as possible.

For hygienic reasons, all product contact surfaces must be neutral to the food, cleaning agents and disinfectants. Stainless steel is the ideal material for most foodstuffs. Two main grades should be used for dairy products (249):

- AISI 304; and
- AISI 314, an acid-proof steel quality.

Rubber and plastic hoses should be avoided to the greatest extent possible; they present cleaning problems. Where flexible connections are needed, the hoses should be as short as possible.

5. Installation of the Equipment

The installation should be performed by competent staff. Flexible connections by means of plastic or rubber hoses should be avoided as much as possible (they present cleaning problems). However, such solutions may be necessary in the reception area when liquid raw materials are delivered by tankers and used in the product composition or formulation. If unavoidable, tubing should be short.

Pipe connections (permanent joints) should be welded as much as possible (139), and by professional welders only.

If the equipment should have to be dismantled, the pipe connections should be in the form of a threaded union with a male end and a retaining nut with a joint ring inbetween, or a clamped union with a joint ring. This type of connection is used for process equipment, instruments, etc., that need to be removed for inspection, cleaning, repair or replacement.

Pipe Connections

Attention must be paid to dead ends, both in the pre-processing and final processing area. The length of the dead end (measured from the centre of the main pipe to the end of the dead end) must not exceed 1.5 times its diameter (figure 6) (129). When placed in a bend, the flow of cleaning solution should be into the dead end (figure 6).

Dead Ends

Certain basic rules of hygiene should be followed when installing aseptic filling equipment (60). When setting up pipelines, unnecessary “ups” and “downs” should be avoided (figure 7).

Supports for auxiliaries, such as water and air pipes, electrical cables, etc., should be accessible for cleaning (housekeeping: removal of dust).

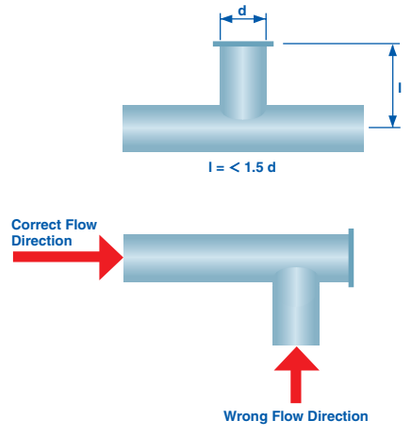


Figure 6. *Dead Ends: Length and Flow Direction*

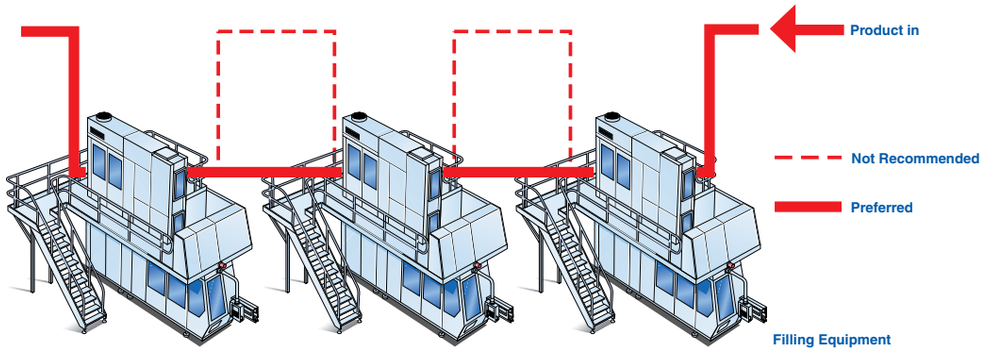


Figure 7. *Installation of Piping*

Aseptic Tanks

An installation should be as straightforward as possible and as flexible as necessary. Some situations require complicated installations (262). An example of a rather flexible installation is given below (figure 8) (24).

Incorporation of (aseptic) tanks may also add to flexibility. Is the flexibility really necessary? Everything installed beyond the outlet of the steriliser holding cell increases the risk of microbiological recontamination! However, there are some clear advantages connected with operations that include an aseptic tank:

1) product overflow (reprocessing) is avoided; this is of increasing interest as the number of filling machines in a production line is increasing and particularly if fillers with different volumes are included in one and the same line;
2) longer production runs may become possible since the product steriliser can be cleaned while the filling operation continues from the aseptic tank;
3) operational stress is reduced since the sterilisation of the product becomes independent of the packaging operation;
4) during start-up of the filling operation, an abundant surplus of product is available. This results in a reduction of packaging material and product losses and minimises pressure drops during the production stage.

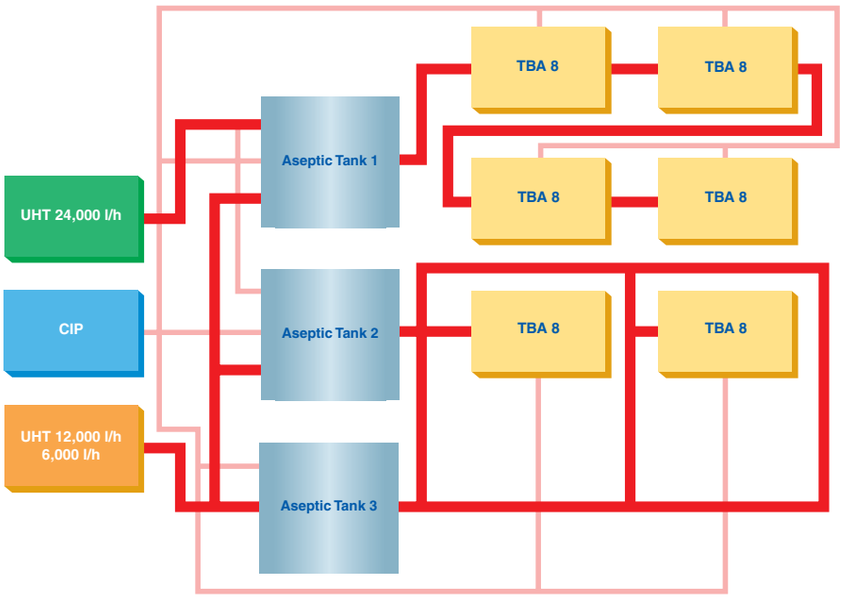


Figure 8. *Complex, Flexible Installation*

Production Flexibility

Flexibility can be obtained by automatic valve arrangements, swing bend or manifold panels, etc. Sometimes it is less expensive to replace versatility by production planning, provided that the necessary storage space is available and that the market department delivers, at least one week in advance, a forecast of the amounts of the different products required.

If manifolds are used, they should be firmly mounted and placed in such a way as to minimise "ups" and "downs" of piping because of air pockets and/or condensate which lead to cleaning and plant sterilisation (disinfection) problems. Raising the manifold above floor level may require the installation of a platform for operation, an additional cost. On the other hand, piping becomes much shorter, and this constitutes a saving (figure 9).

In a manifold, the position of the connections can be controlled, interlocked and recorded by automatic devices.

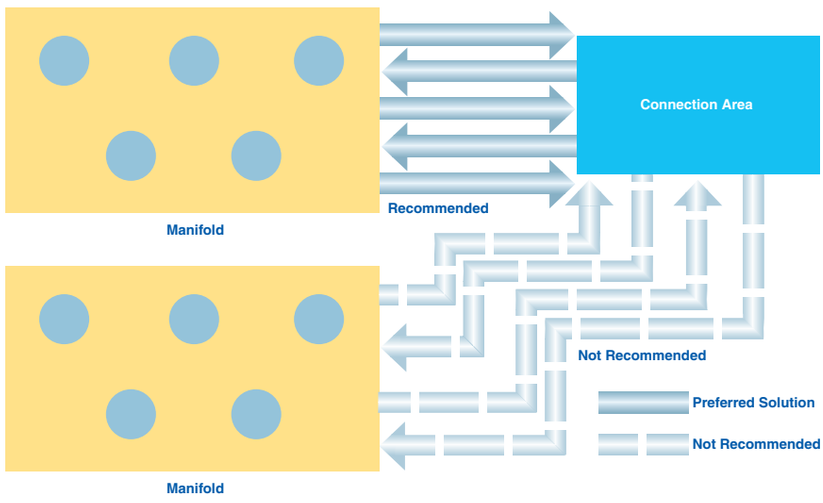


Figure 9. *Installation of Swing Bend Panels*

8. Cleaning and Housekeeping

A. Cleaning

Summary

In any food producing plant, but particularly in those producing long-life products, proper cleaning is one of, if not the, most important operation. The purpose of cleaning is to remove all visible dirt from equipment surfaces, whether they are in contact with the product or not. Efficient sanitation and/or sterilisation of equipment surfaces is possible only if they are free from dirt. In this respect, two aspects need to be considered: the repeatability and the adequacy of the process involved. Repeatability requires full, preferably automatic, control. Knowledge and experience are necessary to achieve adequacy. The different parameters involved in cleaning are also discussed.

1. General

The purpose of cleaning is to render equipment physically clean, i.e., to remove all dirt. No aspect of processing is more important than cleaning and sterilisation of the equipment (42). A consistently good result from an operation can only be expected if the equipment is reasonably clean.

Food residues are bound to surfaces by a number of forces, or may even penetrate into materials (plastics, rubber, etc.,). In cleaning, these forces have to be overcome by either a mechanical and/or chemical effect (figure 1).

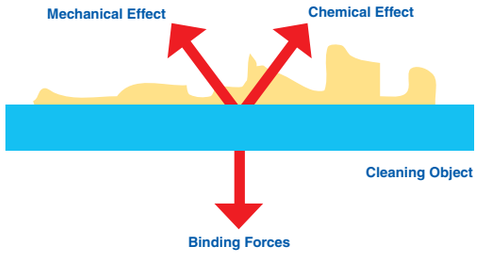


Figure 1. *Forces Binding Dirt to Surfaces*

The cleaning of a plant and the equipment installed there must be carried out with the utmost care and attention if the end-product quality of the processed materials is to be entirely satisfactory (34). To this end, not only procedures, but also the design and installation are of great importance (152) (see the section on Installation).

Whether it is carried out manually or by circulation, the wet-cleaning of equipment involves three basic steps (34).

Steps Involved in Wet-Cleaning

- *Pre-rinsing*, the purpose of which is to remove by water any loosely adhering residues from plant surfaces. It should be done as soon as production is completed to prevent dirt from drying on the surfaces. It must be applied in the correct manner relative to the plant, (e.g., burst-rinsing for vessels and continuous flow for components and pipelines), the product and at the correct pressure and velocity for both. The time taken must be sufficient to ensure that the discharged water is free of product residues (42). If possible, the temperature of the rinse should be above the melting point of any fat residues but should not denature protein.

Pre-Rinse

- *Cleaning* entails the removal or elimination of any residues from equipment. It is accomplished by a process of soaking and/or scrubbing involving the use of detergent solutions (emulsification, hydrolysis, saponification, dispersion, etc.,) by plain alkali and/or acid or by formulated cleaning agents. Since the actual cleaning is a chemical process, correct temperatures are essential.

Actual Cleaning

- *Final rinsing*, the aim of which is to remove or reduce to an acceptable level all traces of residues and detergent from the equipment by means of clean water. The quality of the water used is of great importance with regard to physical (damage to equipment), chemical (corrosion) and microbiological aspects.

Final Rinse

2. Manual Cleaning

The Human Factor

The result of manual cleaning depends to a large extent on the mechanical effect. Appropriate tools must be made available. Only mild chemicals may be used. The pH value should not exceed 10 or be below 4. The human factor is of the utmost importance in manual cleaning. The importance of motivation in the staff (operators) involved in manual cleaning cannot be overestimated. Training, education and supervision are necessary to ensure a satisfactory result.

3. Cleaning in Place (CIP)

In CIP cleaning, solutions are pumped and circulated through the equipment "in place" (248). The chemical effect dominates.

In a food factory, there is usually more than one cleaning cycle in operation. Attention should be paid to areas where two or more cleaning cycles meet (interface, figure 2). Often these areas are not included in the cleaning operation.

The "mechanical" effect of circulation cleaning requires a turbulent flow for the removal of air from the system and transportation of particles through the circuit. A flow rate of > 1.5 m/sec in pipes is recommended.

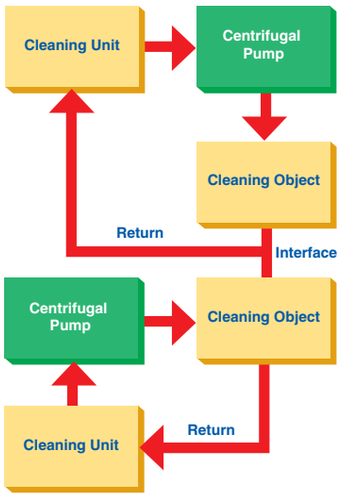


Figure 2. *Cleaning Circuits: Interface*

Flow Rate = 1.5 m/sec

The result of any process, including cleaning operations, is determined by its repeatability and adequacy (table 1).

A repeatable process is not necessarily adequate!

In chemical processes such as cleaning, five (critical) control parameters need to be controlled *and* must be adequate:

1) the chemical;
2) the concentration of the chemical;
3) the contact (= flow rate) between the chemical and the cleaning object;
4) the contact time; and
5) the temperature during contact.

Repeatability	Adequacy
Process Control	Knowledge
Maintenance	Experience

Table 1. *Processes: Repeatability and Adequacy*

3.1 Cleaning Parameters

3.1.1 Chemical

Chemicals used in cleaning food plants in general and dairies in particular may either be simple or formulated cleaning agents. If formulated cleaning agents are used, the recommendations of the suppliers must be followed meticulously.

Cleaning Agents

In the dairy industry, the use of pure caustic soda and acid (nitric or phosphoric) is common and usually quite adequate. However, for some milk-based and other products containing emulsifiers, stabilisers, thickening agents, etc., formulated cleaning agents may be required to obtain an acceptable result in the cleaning process. Poor quality of the raw milk (intermediate product) may also cause problems.

3.1.2 Chemical Concentration

Concentration

The concentration of the detergent depends upon the kind of dirt to be removed. Heated surfaces often require higher concentrations than cold ones. For the cleaning of heat exchangers (pasteurisers, UHT equipment) used for processing milk, an alkaline (NaOH) concentration of 1.5-2.0% is usually sufficient. The corresponding concentration of acid (HNO_3 or H_3PO_4) is 1.0-1.6% (11).

If controlled by dosing only, special attention should be paid to the concentration of the detergent(s) used. The dosing device of automatic cleaning units is usually rather sensitive. Service and maintenance are required to ensure proper functioning. A conductivity measurement connected to a guarding and recording function is safer.

Control

Semi-automatic and manual cleaning units (see below, table 2) may present a problem. In such units, the detergents are often added in "one go" directly to the balance inlet tank of the steriliser by means of a bucket (figure 3). Since heat exchangers are designed to give the same residence time to every passing particle, the product (detergent) does not mix. As a consequence, a plug of concentrated detergent passes through the equipment reducing the contact time and causing corrosion, particularly during the acid clean. The detergent needs to be added during the time required for one circulation. The duration depends upon the capacity of the equipment and the installation, but is typically between 5 and 10 minutes. It is impossible to empty repeatedly a bucket for such a long time! Either a "dosing" device has to be constructed or a pre-mix of ready-to-use detergent solution must be prepared in sufficient amounts (the volume needed to fill the entire cleaning circuit).

Problem: Semi-Automatic and Manual Cleaning Units

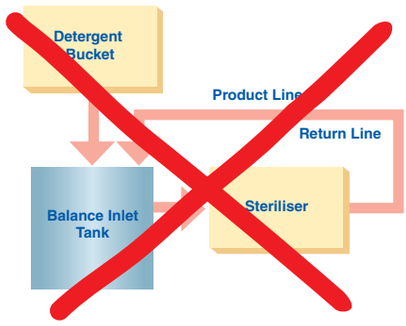


Figure 3. *Wrong Addition of Chemicals*

3.1.3 Contact between the Chemical and the Cleaning Object

Contact between a liquid and the inner surfaces of pipes requires a certain flow rate: turbulent flow is needed in order to remove effectively air pockets from the circuit and transport particles through the system. In pipes, a flow of at least 1.5 m/sec (34) is recommended and usually necessary to achieve sufficient turbulence. In addition, the flow direction should be into, and not past, dead ends (figure 4) (129).

Flow Rate = >1.5 m/sec Flow Direction

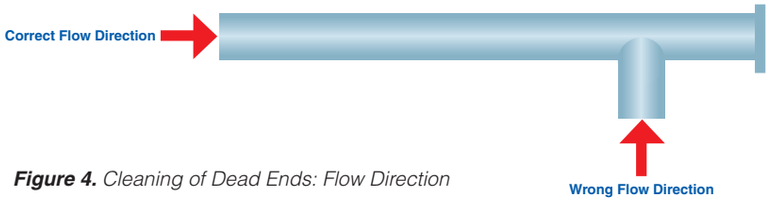


Figure 4. *Cleaning of Dead Ends: Flow Direction*

For cleaning aseptic and other tanks complete coverage of the inner tank surface is needed. 200-250 litres/hour/m^2 for horizontal tanks and 250-300 litres/hour/m^2 for vertical tanks are necessary. In addition, a spray device needs to be installed every second metre in horizontal tanks. The proper functioning of spray devices must be ensured by regular service and maintenance, and they must be controlled regularly.

Cleaning the Tank

Heat exchangers are normally designed to create a turbulent flow but connecting pipes must be taken into account. Due to flow restrictions, a bypass may become necessary for heat exchangers and especially homogenisers. Installation of a special "cleaning pump" with sufficient capacity may be required.

Heat Exchangers

3.1.4 Contact Time

A distinction should be made between contact time and circulation time. The circulation time is given by the effective contact time plus the time needed for one circulation in the circuit to be cleaned. For the alkaline cleaning phase, an effective contact time of ten minutes is usually enough. A somewhat shorter time suffices for the acid-cleaning phase. For the pre-rinse, intermediate and final rinses, 5 to 10 minutes effective contact time is usually sufficient.

Circulation Time Effective Contact Time

3.1.5 Temperature during Contact

Pre-Rinse

The temperature of the pre-rinse should preferably be above the melting point of fat residues (for butter fat, 50°C) but below the denaturation temperature of protein (for milk 70-80°C).

As mentioned above, cleaning is mainly a chemical process. All chemical reactions are very much temperature-dependent. This is particularly true of the alkaline-cleaning phase. It is safe to assume a Q_{10}-value of 2 to 3. Each increase in the temperature of the alkaline phase by 10°C doubles or triples the speed of reaction.

Alkaline Cleaning Phase

Therefore, the caustic-soda cleaning phase should be done at as high a temperature as possible, preferably at processing temperature: higher temperatures make the caustic-soda phase clean more effectively! Corrosion problems at high temperatures of caustic-soda cleaning are negligible. If, however, temperatures in excess of 100°C are used, the equipment (especially plate heat exchangers) must have the necessary protection shielding. The manufacturer of the equipment should be contacted to ensure that such high temperatures are tolerated without their causing corrosion or other problems.

Acid Cleaning phase

The acid-cleaning phase is different. Dissolving minerals is more of a physical/chemical reaction and as such much less temperature-dependent. In addition, acids at high temperatures corrode stainless steel and, above all, reduce the elasticity of rubber. As a result, gaskets and membranes, etc., become increasingly brittle. Preferably, the acid-cleaning phase should be at 60-70°C.
A temperature of 90°C should not be exceeded.

3.2 The Control of Cleaning Processes

Cleaning Units

Whenever cleaning in place is performed, a cleaning unit is in operation. A distinction can be made between three different types of cleaning unit: fully automated, semi-automated and fully manually controlled units (table 2) (42). Depending upon the degree of automation, cleaning units are able to control the cleaning process more or less efficiently. Attention should be paid to the extent at which the different functions of a cleaning operation are controlled, guarded and recorded. Only full control guarantees repeatability. For each cleaning circuit, the type of cleaning unit should be identified and classified with regard to its functions.

The chemical, often NaOH (caustic soda) and HNO_3 (nitric acid) or H_3PO_4 (phosphoric acid), can be controlled by dosing only, or by measuring and recording the pH of the cleaning solutions. Control of the concentration is possible by determining the conductivity. A guarding function may be connected to the measurement of conductivity. The contact is given by the flow rate, which is determined by the capacity of the centrifugal feeding pump and the pressure drop over the cleaning circuit which remains constant until the circuit or the capacity of the feeding pump is changed. A flow meter offers the possibility of using a guarding function and permits recording. The contact time can be controlled and guarded by a timer, and the temperature by a thermosensor. All these functions can (and should) be recorded.

Parameter	Fully Automated	Semi-Automated	Manual
Chemical	+	-	-
Concentration	+	-	-
Contact (Flow)	+	+	+
Contact Time	+	+	-
Contact Temperature	+	+	-

(+ = controlled, - = not controlled)

Table 2. *Control Level of Different Types of Cleaning Unit*

3.3 Cleaning Programme

Standard Cleaning Programme

The sequence of the different cleaning phases is often the following:

a) water rinse;

b) alkaline-cleaning;

c) water rinse;

d) acid-cleaning; and

e) water rinse.

If the water used for rinsing contains chloride (Cl^-), stainless steel surfaces should be left slightly alkaline. Otherwise, the risk of chlorine corrosion increases. The above programme always results in slightly acid surfaces since the acid used for cleaning cannot be rinsed off totally.

The first action of caustic soda on protein is a swelling. This may cause problems particularly in plate heat exchangers. The product to be processed is heated (cooled) by passing it through a number of parallel sections (figure 5). On heat exchange surfaces, deposits are formed which, specifically in the temperature range of ~ 80-115°C, may contain relatively large amounts of protein (referred to as deposit A). At higher temperatures, the deposit consists mainly of minerals (deposit B). It is unlikely that the same amount of deposit is formed in all parallel passages. More likely, some will have more, others less dirt.

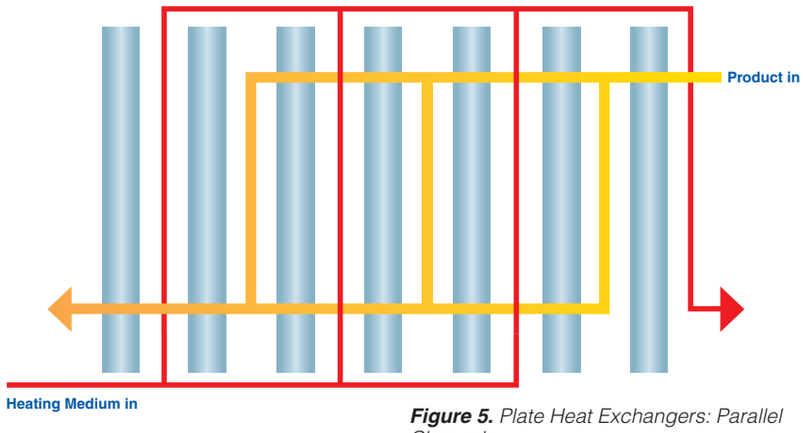


Figure 5. *Plate Heat Exchangers: Parallel Channels*

Swelling of the protein will enhance the difference. Cleaning liquids will seek to pass through sections of low resistance, i.e., those channels with less deposit. Where cleaning is most needed (heaviest dirt), the smallest amount of detergent passes through and is active! In general, deposit formation is favoured by:

Deposit Formation

a) the product as such;

b) the quality, mainly protein stability, of the raw materials used in the formulation of the product and the intermediate product;

c) the actual temperature – in milk, the largest amount of deposit forms in the temperature range 80-120°C;

d) the temperature differential – the lager the difference in temperature between the heating medium and the product, the more deposit will form;

e) the flow rate of the product – laminar or turbulent;

f) the characteristics of the heat exchange surface – more deposit will form on corroded or dirty surfaces;

g) foam in the product – possibly the single most important factor of all.

Acids also dissolve protein. However, the first action is contraction of the protein deposit. Thus, heavily soiled passages are opened. If heat exchangers are soiled with large amounts of precipitates containing protein, a change of cleaning programme may be considered:

a) water rinse;

b) acid-cleaning;

c) water rinse;

d) alkaline-cleaning;

e) water rinse;

f) acid-cleaning; and

g) final water rinse.

Such a programme requires more time, more cleaning agents and larger amounts of water. On the other hand, it may solve difficulties in cleaning which in turn may cause problems in product quality. In both programmes, the surfaces show traces of acid after cleaning.

3.4 Chemistry of Cleaning

Cleaning in place is mainly a chemical process. Reactions are very highly dependent on temperature (Q_t-value: ~ 2 to 3). The mechanical effect is limited, but turbulence from the cleaning solutions is necessary to remove air pockets from the circuit and to transport particles through the system.

Peptides = Wetting Agents

3.4.1 Caustic soda

The effect of the alkaline detergent (caustic soda, NaOH) depends on the pH. An alkalinity corresponding to a pH of 13 is necessary for full efficiency. Caustic soda acts by first swelling and then splitting and dissolving protein. In this process, peptides are formed. Some of these are wetting agents reducing the surface tension of the cleaning solution. This results in emulsifying fat, a better penetration of dirt and wetting of the surfaces to be cleaned. The process is very dependent on temperature. Therefore, the temperature of the alkaline-cleaning phase for heat exchangers, pipes, etc., should be 80°C or more. For cleaning tanks, a lower temperature and the use of formulated detergents may be a better alternative. This, however, requires a separate cleaning unit, i.e., a decentralised CIP-system.

Saponification = Soap = Wetting Agent

Caustic soda also acts on fat. At high temperatures fat is saponified, i.e., soap is formed. Soap also lowers the surface tension of solutions thus improving the emulsification of fat and the wetting effect.

Metal Ions

In alkaline solutions, multivalent metals (mainly calcium Ca^{++} and magnesium Mg^{++}) are precipitated. If hard water is used for rinsing and/or preparation of the cleaning solutions, this will add to the mineral-based dirt in the equipment. Precipitation of metals can be avoided by the addition of sequestering (polyphosphates etc.,) or chelating agents (EDTA, ethylene-diamin-tetra acetic acid or NTA, tri-nitrilo-acetic acid etc.,) to the caustic soda. Such formulated cleaning agents are optimal both for temperature and pH. These must be observed carefully. Because of the strong ability of chelating compounds to dissolve metals, the risk of corrosion should not be forgotten. In addition, their effect on the environment is debatable.

3.4.2 Acid

The main task of the acid (HNO_3 or H_3PO_4) is to remove mineral deposits from the equipment. This process is much less temperature-dependent than the alkaline-cleaning phase. In addition, acids at high temperatures have an apparent corrosive effect on stainless steel but even more so on rubber. The gaskets lose elasticity, they become brittle, and the "pressure-set" decreases. Therefore, the acid should be used at relatively low temperatures, preferably at 60-70°C, but certainly below 90°C.

In addition, acids first contract and then remove protein. This process is much slower and less effective than the action of the caustic soda.

3.4.3 Water

In cleaning, the quality of water is very important and should influence the choice of cleaning agent, and it may be of even more importance as a possible cause of corrosion of the equipment (248). Water treatment or the addition of sequestering (chelating) agents may be necessary. The following water qualities have been recommended (tables 3 and 4):

a) Tetra Pak:
The water must be soft and, if necessary, dehardened, and it must be clean in order to prevent deposits from forming in the plant. It must not contain iron or manganese. Deposits, due to an inferior quality of water circulating in the plant, can cause vital parts to malfunction.
Supply pressure, min. 400 kPa (4 bar), must be constant.

- Total hardness, less than 180 mg per litre of $CaCO_3$ equal to 10 dH or 12.5 e.
- Chlorine content: less than 0.2 ppm.
- Chloride content: less than 30 ppm.
- pH value: higher than 7.

b) IDF (International Dairy Federation) for minimum corrosion: (table 3).

c) The Society of Chemical Industries, London: (table 4).
The Society of Chemical Industries recommends slightly lower pH values and states a pH range of 6.5 to 7.5 (table 4).

The recommendations of Tetra Pak and the IDF (table 3) require a high pH (> 7 and > 8.3 respectively). Because of environmental problems, such water is no longer available in most countries. The high pH values are advocated in order to minimise corrosion of the stainless steel surfaces of the equipment.

The hardness of water is defined (US) in table 5.

Parameter	Value
Hardness	3 - 4 dH
pH	8.3
Chloride	<50 ppm
Sulphate	<100 ppm
Iron	<0.3 ppm
Manganese	<0.1 ppm

Table 3. *Cleaning: Water Quality*

Component	Content
Total Hardness ($CaCO_3$)	<50 ppm
Chloride (NaCl)	<50 ppm
Chlorine (Cl)	<1 ppm
pH	6.5 - 7.5
Iron (Fe)	<1 ppm
Manganese (Mn)	<0.5 ppm
Suspended Solids	Free

Table 4. *Cleaning: Water Quality*

Soft Water	0 - 6 dH
Medium Hard	6 - 12 dH
Hard Water	12 - 18 dH
Very Hard	>18 dH

(1 dH = 10 ppm CaO = 17.9 ppm $CaCO_3$)

Table 5. *Hardness of water*

B. Housekeeping

Introduction

Housekeeping relates to the cleaning of the exterior and interior of a factory. Satisfactory results can only be expected from an operation if both the surroundings and the inside of a plant are in an acceptable condition. In a factory producing long-life products, cleaning activities should be carefully scheduled since they are usually connected with dust and/or aerosol formation. The principles of housekeeping are presented below (221).

1. External Housekeeping

It has already been pointed out that the location of a factory has an impact on the quality of the product(s) produced.

Immediate Surroundings

The immediate surroundings are even more important. A stretch of 20 metres of concrete next to and around the buildings reduces the amount of dust caused by traffic. Mice do not pass an open area of this size and, consequently, the risk for an invasion of the factory by mice is reduced. This area should be kept clean and tidy and must be free from stacks of pallets, old equipment, returned or spoiled products, etc. The planting of bushes and flowerbeds close to buildings should be avoided since they, although attractive, may harbour mice and insects and may attract birds, all of which increase the risk of contamination of the actual production section.

At regular intervals, but preferably not when production is in progress, the premises must be cleaned manually with suitable tools or machines. Great care must be taken to avoid unnecessary airborne contamination of the production area (152).

2. Internal Housekeeping

Inside the Factory

For the actual housekeeping, i.e., the cleaning of the indoor areas, it is necessary to prepare and implement a cleaning schedule which includes all the rooms, storage areas, toilets and offices, etc. The schedule is needed in order to be able to minimise the extra load of particles in the air which occurs whenever any housekeeping activity takes place.

Housekeeping Schedule

Some zones must be cleaned once a day or even more frequently, whereas others need to be cleaned once a week or maybe only once a year. The housekeeping schedule has to be decided on and will be different for each factory. The principle is very simple: minimise the load in the air in sensitive areas! Obviously, the storage area for the finished product is a low-risk section. However, if trucks run between the storage area and, for example, the filling room, the floor in the storage section has, at the least, to be kept clean: frequent cleaning becomes essential.

Once a housekeeping schedule has been prepared it needs to be implemented! The schedule states only when to clean, but the question of how to clean needs also to be addressed. Methods and procedures must be established.

How to Clean?

Cleaning with brushes and brooms, pressurised air, vacuum cleaners, or wiping dust off with a cloth increases the load of dust in the air. Small particles will rise several metres and settle slowly, contaminating horizontal surfaces in particular. Consideration should be given to any materials carrying electrostatic charges, such as plastic-coated packaging material. Often, dust particles are also electrically charged: an active attraction is taking place. The risk of contamination in sensitive areas increases if dust is moistened by condensed water. Growth of mould is likely to result. This is easily overlooked on the ceiling, cable ducts, behind tanks, on piping, etc., and may cause severe problems.

Moulds

It must also be recognised that dust may be transported from one room to another by air streams due to differences in temperature and air currents caused by open doors or windows. The only way to reduce such a risk effectively is to schedule the housekeeping work and adhere to the plan meticulously.

Air Currents

Wet-cleaning may be done manually or with machines, water hoses, high-pressure equipment or by foam or gel cleaning. Although floors and walls in production areas must be made of non-absorbent materials, before a wet-cleaning operation is started it is important to remove all moisture-absorbing materials such as wooden pallets, cardboard boxes, etc., from the section. These come into contact with moisture by being splashed with water or by aerosols which are always formed when a wet-cleaning phase is underway. If such materials do become moist, the risk of growth of microorganisms, particularly moulds, increases. These in turn will contaminate the air and may cause quality problems.

Aerosols

The first step of any housekeeping activity should be to put everything in its proper place. The cleaning of floors may often be difficult because the distance between floor and undercarriage is too narrow. In addition, it is not possible to control by inspection the effects of sweeping or rinsing.

Cleaning Floors

Corners are likely to remain dirty, thus providing ideal spots for insects and microorganisms to breed. Equally, the bottom of tanks, conveyor belts, etc., are critical areas and difficult to keep reasonably clean. Usually, such areas must be cleaned manually with a brush. They are only accessible by crawling on one´s knees and a satisfactory result is difficult to achieve.

The housekeeping schedule should not only state when different rooms are to be cleaned but should also contain information on how often the outside surfaces of the items of equipment in each room should be cleaned and inspected.

Over-Ambition!

Obviously, it is possible and tempting to prepare a housekeeping schedule which is so ambitious that its execution would require many hours every day. This is not necessary. It is important to keep in mind that every housekeeping activity removes a risk but also creates one as well: the risk removed must be higher than the one originating from the activity. It is recommended to clean the production area thoroughly after each production run, at the end of every day, or at the end of the week if it is considered sufficient. All surfaces exposed to dust fallout should be wiped with a towel moistened with water or disinfectant *before* the next production run or use of the equipment commences.

Sewage System, Drains

It is highly recommended that all surfaces which are cleaned with water are carefully dried at the end of the cleaning operation. Small residues of water support the development of microorganisms and increase the microbial load in the air. The importance of a proper installation of the sewage system has already been addressed. All drains in the floors as well as all basins for washing items of equipment by hand should be cleaned manually and disinfected at the end of each working day.

Pests

In spite of all these efforts, it is difficult to prevent the appearance of insects and pests in a food factory. It is common to contract a specialist company for regular control and, if need be, treatment in order to kill mice, insects and other pests. Depending on local regulations, only certain biocides are permitted. To achieve an optimal result, it is usually necessary to locate the breeding spots. Specialist companies should have the equipment and knowledge necessary in order to minimise the risk of an invasion of the factory by pests. They should also know which chemicals are permitted and which chemicals require production to be shut down for several hours or even days.

For cleaning as well as for housekeeping, the goal must be to achieve a result which is as good as necessary and *not* as good as is humanly possible. Every activity in this field represents a compromise between economy and quality.

Cleaning and housekeeping should be:
as good as necessary – no more, no less!

9. Commissioning

Summary

Commissioning is the demonstration that the equipment, production line and/or plant perform as specified or agreed upon. Specified conditions are not negotiable, neither with regard to stated values nor to the methods and procedures to be used in checking. However, some important performance criteria are heavily affected by conditions which are beyond the control of the equipment, production line or plant supplier. If these are included in the commissioning process, values as well as methods and procedures for checking have to be agreed upon between the parties involved, usually the buyer *and* the seller. In the present chapter, the commissioning of microbiological performance criteria is discussed in detail.

1. General

Commissioning is Part of GMP

Before production commences, it must be established that the assembled plant is as specified and capable of working to the standards laid down (142).

Commissioning is the demonstration that the components, processing systems and/or combinations delivered and/or installed operate within their technically specified limits or are capable of producing an agreed upon result. As part of the GMPs (US FDA: 21 CFR 113), every plant should be subjected to a commissioning procedure.

Specifications Performance Criteria

Equipment specifications are prepared by the manufacturer of the machinery and thus not negotiable. This applies to the specification parameters as such and the methods and procedures for testing. However, not all characteristics of interest can be specified by the manufacturer of the equipment since they depend upon factors which are not, or not fully, controlled by the equipment supplier. The details of such performance criteria have to be agreed upon by the commercial processor, the supplier of the machinery, and any other party involved. Microbiological performance criteria present an example and are discussed in some detail below.

2. Microbiological Performance

Pre-Conditions for Microbiological Performance Testing

Prior to any microbiological performance test of the production line for a long-life product, it should be confirmed that the plant is cleanable. In addition, it is recommended that during commissioning, all parts of the plant which come into contact with the sterile product reach the required temperature and/or conditions for the required duration when pre-sterilisation is carried out (222).

Microbiological performance can only be checked on entire production lines running under true operating conditions (142, 222), never on individual components that make up such lines (262). The normal procedure is to sample an agreed number of packages or containers which, after proper incubation, are evaluated for microbial spoilage. One manufacturer of an aseptic packaging system provides a “sterile guarantee” (22) without, however, specifying conditions. For such a guarantee to be meaningful, a large number of parameters need to be specified.

When agreeing on microbiological performance criteria, attention must be paid not only to the equipment but also

- to the environment in which the equipment functions;
- to the installation in general;
- to the microbiological quality (spore count) of the raw materials (ingredients);
- to the intermediate product(s);
- and to operative procedures.

Number of Products

The purpose of microbiological performance testing is to demonstrate that the plant in question *can* perform to defined standards. Because of the costs and time involved, such a procedure should be restricted to a limited number of products, preferably only one. Often, if required, process parameters can be adjusted to apply to specific demands for individual products. The idea of including in the microbiological performance test secondary packaging as well as handling practices and distribution procedures (222) could also be considered.

Microbiological Performance = AQL

Microbiological performance criteria for aseptic production lines could be AQLs (acceptance quality level; maximum acceptable defect rates). Since test procedures are based on statistics, unsterility rates can only be expressed in terms of probabilities. At most, a microbiological performance test only shows that a production line at a certain time, and under specified and existing conditions, has achieved the agreed upon result. This is not a guarantee that the line will deliver the same or comparable results at other times or under other conditions. Factors having an impact on the result change continuously.

Agreement is Required

Before commencing a microbiological performance test, agreement should be reached on the following:

1) number of samples to be tested;
2) sampling procedures;
3) incubation conditions;
4) methods of evaluation;
5) definition of a defective (unsterile) package; and
6) consequences of deviation.

2.1 Number of Samples to be Tested

Tetra Pak's Suggestion

It has been claimed that the number of test package should not be less than 20,000 and should represent a running period of not less than 4 hours (142). Tetra Pak suggests three independent production runs. From each run 2,400 packages are sampled, incubated, and evaluated, thus giving a total of 7,200. A defect rate of 0.1% is recommended as acceptable (262). This corresponds to no more than three defects in the total number of packages tested (7,200), and is based on a 90 % probability level. However, the number of samples which should be drawn for testing depends on the AQL to be checked. This in turn should be defined by the commercial processor and the sampling procedures should be adopted accordingly. Agreement should be reached on the parameters above by the buyer and seller.

Poisson Distribution Applies

Statistics are used in all sampling procedures, including that of checking microbiological line performance. In this instance, Poisson distribution applies. (For a more detailed discussion of sampling statistics, see the section on "Quality Control"). In order to be able to establish a suitable sampling plan, the following two parameters need to be specified:

- the rate of defects to be checked; and
- the probability level.

The Poisson distribution diagram is shown in figure 1 at the end of this section. In the diagram, the y-axis shows the probability. When developing attribute sampling plans, the required probability needs to be decided on in order to be able to use the diagram. The x-axis gives a factor for calculating the number of packages needed to fulfil the requirements of a specified sampling plan. Formulas for this calculation are given at the bottom of the diagram. In addition, the diagram contains a number of C-lines (C = 1, C = 2, C = 3, etc.,) which indicate the number of events which are being checked. If, for example, a defect rate of 1 per 1,000 (0.1%) packages is to be determined at a 90% probability level, the Poisson diagram is entered at 9 (= 90% probability). One proceeds along this line until the curve C = 1 is reached and the corresponding x-value of ~ 2.3 is extracted. Using the formula for n (= number of samples needed), the following calculation results:

n = 100 • x / defect rate to be checked
n = 100 • 2.3 / 0.1 = 2,300

In order to check a defect rate of 0.1% at 90% probability, 2,300 packages are needed. The conditions for the sampling plan are fulfilled if, after proper incubation, no more than one defective package is found. If the defect rate to be checked is 2 per 1,000 (0.2%), the same procedure is used but instead of reading the curve C = 1, the curve C = 2 is read and a value of ~ 4 is obtained. The rest of the calculation is the same: 2.3 is replaced by 4 and the resulting number of packages needed is 4,000. The number of defective packages acceptable is zero, one or two.

On the other hand, if a given number of packages is incubated and evaluated (say 200) and one defective package is found, what are the conclusions that can be drawn? Again the Poisson diagram should be consulted, but this time the corresponding defect rate should be calculated. Based on a 90% probability level, x is again equal to 2.3. The following formula should be applied:

defect rate = 100 • x / n
defect rate = 100 • 2.3 / 200 = ~ 1.1

At a 90% probability level, the defect rate is equal to or less than 1.1%. However, there is a 10% risk that the defect rate is even higher than 1.1%!

Ineffectiveness of Sampling Procedures

Table 1 shows that low defect rates (< 0.1 %) are difficult to detect: a large number of packages is needed to reach any reasonable probability level of detection.

% Spoilage in Batch	Size of sample (No. of packages tested)					
	10	30	100	300	1,000	3,000
5	40.1	78.5	99.4	99.9	99.9	99.9
2	18.3	45.0	86.7	99.8	99.9	99.9
1	9.6	26.0	63.4	95.1	99.9	99.9
0.1	1.0	3.0	9.5	25.9	63.2	95.0
0.01	0.1	0.3	1.0	3.0	9.5	25.9

Table 1. *Percentage probability of detecting one or more spoiled aseptic packages in different sample sizes for a number of spoilage levels in an entire batch.*

2.2 Sampling Procedures

Buyer's Risk Versus Seller's Risk

Sampling procedures are described in more detail in the section on "Quality Control". Agreement should be reached as to whether the sample is the total production run or only parts of it. In addition, if a sampling scheme is applied, should random or aimed sampling procedures be used, or both?

It has been suggested (265) that 300 individual packages be randomly sampled from a production run of 3,000 to 8,000 units. A defect rate of 0.1% is achieved if no more than one defective package is found. This number is based on the buyer's risk, i.e., that production within quality specifications is not claimed incorrectly. The "American Military Standard" is based on the buyer's risk. In commissioning the producer's risk, i.e., that of not releasing a "bad" production, should be considered. Much larger numbers are required: 2,300 packages need to be incubated. If no more than one defective package is found, the unsterility rate of 90% probability does not exceed 0.1%.

The Poisson distribution diagram (figure 1) has been used to calculate of the sampling plans. The conditions of the sampling plan are fulfilled if not more than one defective package is found in the sample. As can be seen in table 2, very large numbers of packages are needed in order to detect low defect rates (0.1% or less) at a reasonable level of probability. In this respect, the space needed for incubation can be a problem. A suitable room has to be provided. Sometimes the boiler room can be used.

Defect Rate	Risk %	Samples Needed
1 : 10,000	10.0	23,000
	5.0	35,000
	1.0	46,000
	0.1	70,000
1 : 1,000	10.0	2,300
	5.0	3,500
	1.0	4,600
	0.1	7,000
1 : 100	10.0	230
	5.0	350
	1.0	460
	0.1	700

Table 2. *Samples (Packages) needed to detect different defect rates. (Acceptance level: one defective unit).*

Large Numbers of Packages are Needed!

Table 2 gives the number of samples needed to check for different defect rates at varying levels of risk. The level of probability is an expression of the likelihood that the conclusion drawn is correct, while the risk indicates the opposite. The risk of an operation is equal to 100% minus the probability of that process.

2.3 Incubation Conditions

Incubation Time and Temperature: A Compromise!

For how long and at what temperatures are the samples to be incubated? The choice of any high incubation temperature introduces a factor of choice. The most correct procedure would be to store the selected packages at ambient temperature for the whole of their intended shelf life. However, such a procedure is prohibitive because of the time factor involved.

Accelerated procedures must be used. In so doing, artificial conditions are created and, as a result, not all (or even more?) unsterile packages will be detected. A compromise between accuracy and time needs to be arrived at. In choosing the most suitable incubation temperature, consideration must be given to the normal climatic conditions prevailing in the area where the plant is located, and the product distributed and marketed. It is recommended that incubation should be at ~30°C for 3 weeks (142) or at 30°C for at least 7 days (Tetra Pak's recommendation). The subject is discussed further in the section on "Quality Control".

During the incubation of packages, temperature changes in the containers will be rather slow. Consequently, *average* incubation temperatures are of importance. During incubation, exact temperature regulation, though desirable, is not really necessary.

2.4 Methods of Evaluation

Evaluation: A Compromise!

Methods of evaluation should be agreed upon. A list of some commonly used procedures is presented in the section on "Quality Control". Again, a suitable compromise has to be reached between cost and accuracy. A large number of units has to be tested and checked. Expensive methods of evaluation, though accurate, may be prohibitive because of cost.

2.5 Definition of a Defect

To a certain extent, a failure is defined by the method of evaluation. Depending on the method(s) of evaluation agreed on and used, clarification is needed as to what deviation from normal is to be regarded as a failure, i.e., an unsterile package.

2.6 Consequences of Deviation

What consequences arise if the result agreed on is not attained: repetition of the test or test sequence? Adjustment of the equipment and/or installation? Return of the equipment? etc. If the test is repeated, who is responsible for what? How are the expenses incurred to be recovered?

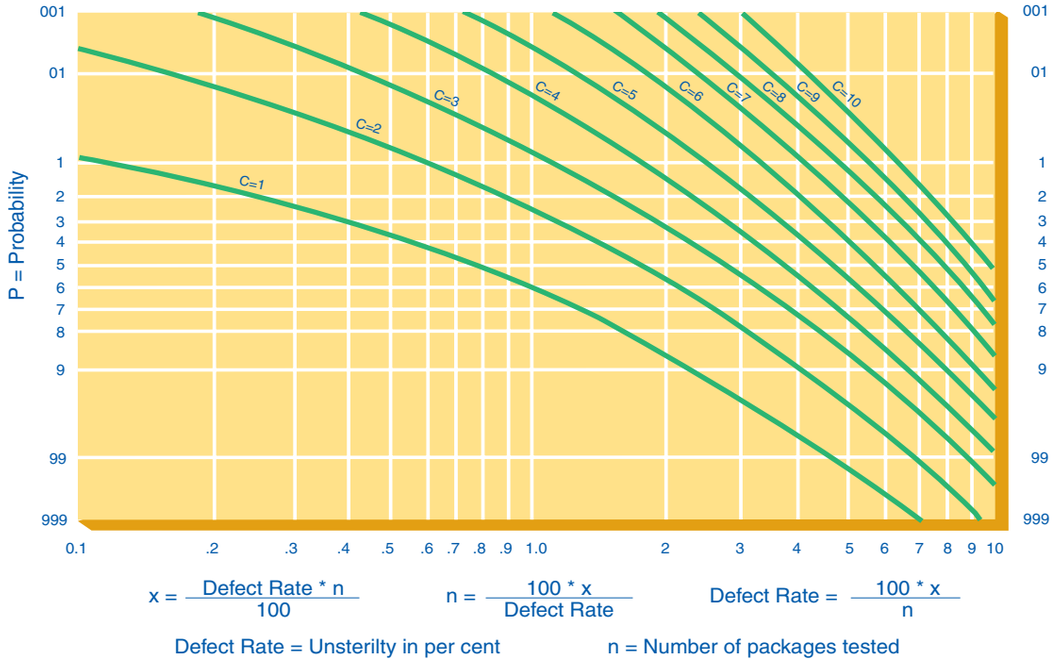


Figure 1. *Poisson Diagram*

10. Changes During Milk Processing

Summary

Each treatment of a food product inflicts changes on it. Depending on the product in question, these changes may be desirable or undesirable. To a greater and greater extent, consumers today are demanding "natural", i.e., unchanged food products. Any effects of commercial processing are regarded as negative. On the other hand, because of rationalisation, distances between food processing factories and consumers are increasing. Concentration of sales outlets (supermarkets and hypermarkets) add to the problem. As a result, an increased shelf life for food products is required. Long-life products represent the best compromise between the demands of unchanged products (limited effects of processing) and increased shelf life.

1. General

Why Food Processing?

In general, food products are processed for a number of different reasons: public health, prolonged shelf life, to increase nutritional values or enhance flavour, etc. Heat treatment of milk is recommended mainly because of public health aspects and to increase its shelf life. A number of thermal processes are either legally recognised and enforced or are already in use in most countries. In principle these processes are:

- pasteurisation;
- retorting (in-container sterilisation);
- UHT treatment (in-flow sterilisation); and
- boiling.

Depending on its severity, heat treatment results in a number of changes in the composition, nutritional value and flavour of milk.

2. Acidity

After UHT treatment, a slight increase in titrable acidity has been found, probably caused by the formation of -SH groups in the heating process (56). However, in a study of 66 UHT-treated samples of milk (250), "sterilisation reduced the titrable acidity somewhat but did not affect the pH".

3. Water

The water content of milk is, of course, not affected by heat treatment provided that no evaporation takes place. If direct (injection or infusion heating) systems are used, it is essential that the vacuum in the expansion cooling vessel is properly adjusted. Otherwise either dilution or concentration of the product takes place.

4. Lactose

The Maillard Reaction

The carbohydrates in milk, mainly lactose, are relatively heat stable. Short boiling, pasteurisation and UHT treatment do not affect the lactose content in a measurable way (155). Depending on the total load of heat, however, retorting may lead to "browning", a product change due to the so-called "Maillard" reaction (118). UHT treatment does not result in browning (205). In this chain reaction, certain sulphurs containing amino acids and lactose are involved. One of the first steps of this reaction is the formation of hydroxy-methyl-furfural (HMF) (204, 205).

Product	HMF-Value
Raw Milk	0.08
UHT Milk	0.07 - 0.09
UHT + Retort	0.16 - 0.24
Retorted Cream	0.38

Table 1. *Different Heat Treatments of Milk, HMF-Values*

UHT Treatment	HMF µMol/litre
Direct	2.16
Indirect	2.56 - 7.64
Retorted	12.04

Table 2. *HMF-Values: Comparison between UHT and Retorting*

In tests, milk was subjected to heating in the temperature range 135-155°C with holding times of 5 to 1 seconds (UHT process). For comparison, in-container sterilisation (retorting) was carried out at 120°C for 15-20 minutes. The HMF-values are shown in table 1.

All retorted products had a significantly higher amount of HMF than the UHT products (table 1) (106). In a comparison between different UHT treatments and retorted milk, the HMF-values shown in table 2 (204) were found.

Lactulose

Any heat treatment of milk results in an increase in the lactulose content. This compound originates from lactose and is also formed in UHT-treated milk. The lactulose value has been used as a measure of the severity of different heat treatments and permits a distinction between various heat treatments of milk.

5. Milk Fat

Unsaturated Fatty Acids Loss of Double Bonds?

The milk fat is also rather heat stable (217). No essential changes in composition are reported by short boiling and pasteurisation. Some reports indicate a slight effect on unsaturated fatty acids from both UHT treatment and retorting: they lose some of the double bonds. From a nutritional point of view, this change is regarded as negligible.

6. Milk Protein

The milk protein, casein (80%) and whey or serum protein (20%) are practically unaffected by pasteurisation.

Denaturation of Whey Protein

UHT treatment results in abnormally long rennetting times which could be corrected by the addition of soluble calcium (205). Short boiling, UHT treatment and retorting denature the serum proteins to a varying extent (table 3). The values in parentheses indicate the percentage gain of casein or loss of whey protein nitrogen inflicted by the different heat treatments.

Heat Treatment	Casein Nitrogen (mg/100g)	Whey Nitrogen (mg/100g)
Raw Skimmed Milk	432 (100.0%)	87 (100.0%)
Pasteurisation	442 (102.3%)	68 (78.2%)
UHT Treatment	470 (109.0%)	37 (42.5%)
Retorting	498 (112.8%)	19 (21.6%)

Pasteurisation: 85°C, 15 seconds; retorting: 116°C, 15 minutes;
UHT treatment: steam injection, 150°C, 2.4 seconds

Table 3. *The Effect of Heat Treatment on Casein and Whey Nitrogen*

Whey Protein Denaturation, Change in Colour and Nutritional Value

Depending on the severity of the heat treatment, the casein nitrogen increases and the whey nitrogen decreases: the serum proteins are deposited on to the casein protein. In an experimental UHT plant, the effects of direct and indirect heating on β-lactoglobulin were studied. Bacteriologically equivalent processes resulted in a 68.4% denaturation for direct UHT treatment, while the indirect system denatured 91.9% (106). Denaturation of the milk serum protein whitens the milk (79),

does not reduce the nutritional value (229), and gives a soft coagulum which actually improves digestibility (6, 205, 213). The coagulum obtained from human milk is soft.

Most of the amino acids remain unaffected by any of the different heat treatments. However, a slight loss in the lysine content (table 4) of milk can be registered (18, 214, 215, 216). Again, the magnitude of this loss depends on the severity of the heat treatment.

Loss of Lysine

Comparing direct and indirect UHT treatment, the differences observed in the loss of lysine were not significant (229).

The coagulation time of milk depends very much on its pH. Milk was heated to 140°C and the time needed for coagulation by heat was determined (39). A minimum heat stability was found at a pH of, or close to, 6.8, the normal pH of milk. In the alkaline range in particular, protein heat stability increased drastically (figure 1).

As found in tests on rats, the effects of pasteurisation, UHT treatment, spray and roller drying, concentration and evaporation on the biological value of milk protein are very slight. Retorting had a more pronounced effect (table 5) (79).

Feeding experiments on rats showed no effects of pasteurisation or UHT treatment but retorting resulted in a clear decrease in the biological value. In a further rat feeding experiment, no major changes in the overall nutritional value of pasteurised or UHT-treated milk were registered (66).

Process	% Lysine Loss
Pasteurisation	1 - 2
Short Boiling	5
UHT Treatment	3 - 4
Retorting	6 - 10

Table 4. *The Effect of Different Heat Treatments on the Lysine Content of Milk*

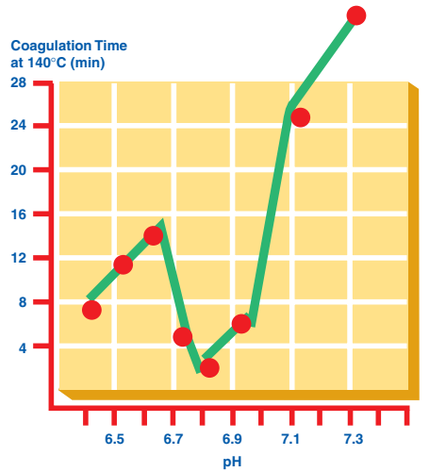


Figure 1. *The Effect of pH on the Coagulation Time of Milk (at 140°C)*

Type of Milk	Biological Value for Rats
Raw	90%
Pasteurised	91%
UHT	91%
Spray-Dried	90%
Roller-Dried	89%
Condensed	89%
Evaporated	88%
Retorted	84%

Table 5. *The Effect of Different Heat Treatments on the Biological Value of Milk*

7. Minerals

Milk minerals are insignificantly affected by heat treatment. The content of soluble calcium and phosphate may decrease slightly, a change which is of no significance for the nutritional value (209, 216).

8. Freezing Point

Depending on the UHT treatment applied, the freezing point of milk may be slightly increased by direct heating and expansion cooling (table 6) (72).

The increase in freezing point (250) is due mainly to the removal of dissolved gas (oxygen and carbon dioxide) during expansion cooling in direct working UHT sterilisers. Under extreme conditions, the freezing point can rise up to 0.010°C. Pasteurisation and indirect UHT treatment may lead to an increase in freezing point of between 0 and 1.7%, while the corresponding values for direct UHT heating may reach up to 4.5% (77). In some countries, the freezing point is not

Heat Treatment	Freezing Point (°C)
Untreated Milk	- 0.514
Pasteurisation	- 0.516
UHT Direct	- 0.519
UHT Indirect	- 0.516

Table 6. *The Effect of Different Heat Treatments on the Freezing Point of Milk*

Increase in Freezing Point!

only used to detect adulteration (dilution with water) in raw milk but also for the finished product. It should be borne in mind that even with correct adjustment of the expansion cooler, an increase in freezing point might occur: freezing point determination is not a suitable method for determining adulteration in finished UHT milk!

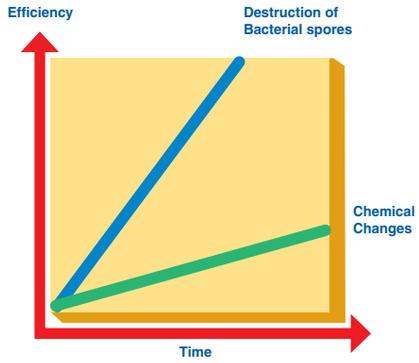


Figure 2. *Destruction of Bacterial Spores Versus Chemical Changes*

Effect of Temperature on Chemical Changes and Microbiological Efficiency

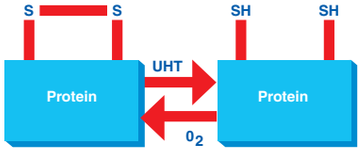


Figure 3. *Effect of UHT Treatment on Di-Sulphide Bonds in the Milk Protein*

Burnt Flavour

9. UHT-Induced Changes in Flavour

The flavour of any food product is probably the most significant quality characteristic in the judgment of the consumer. A product which, for whatever the reason might be, is not consumed has a nutritional value of zero. Therefore, the sensory reception of any food product is of paramount importance. The flavour of long-life milk is determined by three factors:

a) the quality of the raw milk or, more correctly, of the intermediate product;

b) changes in flavour induced by UHT treatment; and

c) changes during storage.

The Q_{10}-value for the change in reaction rate is greater for the destruction of bacterial spores, Q_{10} = 8 - 30, than for chemical changes, Q_{10} = 2 - 3 (121, 219) (figure 2).

Consequently, microbiologically equivalent processes cause less chemical change at higher temperatures and have correspondingly shorter holding times.

Immediately after processing, UHT-treated milk has a pronounced cooked flavour (121, 219) which appears in all types of milk heated above 75 - 80°C (204). A large number of chemical compounds have been identified that contribute to the cooked flavour. However, the main cause is the reaction shown in figure 3 (49, 77, 168, 217).

If milk is heated above 80°C, β-lactoglobulin is denatured and in the process di-sulphide groups are reduced to sulfhydryl groups which have a “cabbage” like flavour (126). This also leads to a slight drop in pH of 0.1 to 0.2 units (56). The occurrence of the cooked flavour could be reduced in intensity or eliminated by the addition of small amounts (30 to 100 ppm) of l-cystine (36, 49, 50).

The formation of SH-groups is reversible (77, 224). The presence of oxygen accelerates this process (56). As a result, the flavour of long-life milk gradually improves during storage until an optimum is reached.

Burnt flavour may result from burnt-on particles which, mainly in indirect heating systems, may loosen from heat exchange surfaces if production times are extended excessively.

Species	Solids	Protein	Fat	Lactose	Ash
Dolphin	55%	10.5%	35.0%	1.0%	0.5%
Fallow Deer	34%	10.5%	20.0%	2.5%	1.5%
Elephant	29%	3.5%	18.0%	5.5%	0.6%
Pig	20%	6.0%	8.0%	5.0%	1.0%
Sheep	19%	6.0%	7.0%	4.5%	0.9%
Cow	14%	4.0%	4.5%	4.5%	0.8%
Goat	13%	3.0%	4.5%	4.5%	0.8%
Human	13%	1.2%	4.5%	7.0%	0.2%
Camel	12%	3.5%	3.5%	5.0%	0.7%
Horse	10%	2.0%	1.5%	6.0%	0.4%

Table 7. *Composition of Milk from Different Animals*

10. Nutritional Value of Milk

Composition of Milk: Different Species

Milk is the lacteal secretion obtained by milking mammals. The composition of the milk varies from species to species; some examples are given in table 7.

Milk is the sole natural source of nutrition for mammals to feed their offspring. It provides all the nutrients needed for growth and development during the first period of life. In its composition, milk differs between different species of mammal (table 7) because of differences in their nutritional needs. Though intended by nature to support the optimal development of infants and suckling animals, cow's milk can provide valuable nutrients for growing children, adults and elderly people. Early in history, milk from domesticated animals was recognised as a valuable food source.

Vitamins, Essential Amino Acids

In addition to the components listed above, minor constituents are also present in milk, such as vitamins (table 10) and trace metals. The term "milk" refers below to cow's milk unless otherwise stated. The protein in cow's milk has a high biological value, i.e., it has a high content of essential amino acids (214).

Essential Amino Acid	Minimal Daily Intake (in mg)	Content in 1 Litre of Milk (in mg)	Litre of Milk Needed (Adult)
Tryptophan	0.25	0.5	0.5
Phenylalanin	1.1	1.8	0.6
Leucin	1.1	3.6	0.3
Isoleucin	0.7	2.2	0.3
Threonin	0.5	1.7	0.3
Methionin	1.1	0.9	1.2
Lysine	0.8	2.7	0.3
Valin	0.8	2.3	0.4

Table 8. *Essential Amino Acids in Cow's Milk*

Nutritional Value: Essential Amino Acids

With regard to the essential amino acids, tables 8 and 9 (4) show the rather close similarity between cow's milk and human milk. Essential amino acids cannot be produced by the human body, they have to be supplied from an outside source. The use of a protein as building material depends upon its content of essential amino acids. The nutritional value of a protein is determined by the essential amino acids present at their relative lowest concentration.

Amino Acid	Cow's Milk	Human Milk	Hen's Egg
Isoleucin	6.4	6.4	6.6
Leucine	9.9	8.9	8.8
Lysine	7.8	6.3	6.4
Phenylalanin	4.9	4.6	5.8
Tyrosine	5.1	5.5	4.2
Cystine	0.9	2.1	2.4
Methionin	2.4	2.2	3.1
Threonin	4.6	4.6	5.1
Tryptophan	1.4	1.6	1.6
Valine	6.9	6.6	7.3

Table 9. *Essential Amino Acids: Comparison between Cow's Milk, Human Milk and a Hen's Egg*

11. Vitamins

Milk supplies a large number of vitamins (table 10) that are essential not only to the development of calves but also necessary to fulfil the nutritional needs of human infants, children and adults.

Opinions differ considerably about the human adult's need for vitamins. Reported variations in the estimated need are 2-30 mg of vitamin E (tocopherol), 0.5-7.0 mg of B_6 (pyridoxine) and 0.5-5µg of B_{12} (cobalamin) per day (214). Milk should be regarded as an important source of some vitamins, especially B_2 and B_{12} as well as vitamin A, B_1, folic acid and panthothenic acid (122, 214).

Some vitamins are affected by heating, the magnitude of loss depending on the severity of the heat treatment applied.

Fat soluble vitamins in milk fat are vitamin A (azerophthol and carotene), vitamin D (calciferol) and vitamin E (tocopherol). The amounts present depend on the fat content of the milk.

Vitamin A is essentially unaffected by pasteurisation and UHT treatment (58, 161, 216). *Vitamin D* is slightly affected by heating, the loss depends upon the severity of the heat treatment (table 11) (55). *Vitamin E* is considered to be stable in all commercially applied heat-processing procedures (209, 214).

Water soluble vitamins present in milk are vitamin B complex, vitamin C, folic acid, biotin and nicotinic acid.

Vitamin	Content in Milk: mg/Litre Average
Caroten A	0.35
Carotin	0.20
Thiamine B_1	0.43
Riboflavin B_2	1.7
Pyridoxine B_6	0.48
Cobalamin B_{12}	0.0045
Niacin	0.95
Folic Acid	0.055
Panthothenic Acid B_3	3.6
Inosit	170
Ascorbic Acid C	18

Table 10. *The Vitamin Content of Cow's Milk*

Heat Treatment	I.U. Vitamin D/Litre Milk
Pasteurisation	80
UHT Treatment	77
Retorting	75

Table 11. *The Effect of Heat Treatment on Vitamin D*

Vitamin	Pasteurisation % Loss	Short Boiling % Loss	UHT % Loss	Retorting % Loss
B_1	10	10 - 20	5 - 15	30 - 40
B_2	0		4	6
B_3	0	0	0	0
B_6	0 - 5	5 - 8	10	25
B_{12}	10	20	10 - 20	80 - 100

Table 12. *The Effect of Heat Treatment on the Vitamin B Complex*

Vitamin B complex consists of a number of vitamins of varying sensitivity to different heat treatments (table 12) (18, 55, 79, 209, 216).

Vitamin C, ascorbic acid and dehydro-ascorbic acid are heat sensitive. This is particularly true for dehydro-ascorbic acid (55, 79, 209, 216). Some losses are already encountered at pasteurisation (table 13). As the severity of the heat treatment increases, more and more vitamin C is lost.

Process	% Vitamin C Loss
Pasteurisation	5 - 20
Short Boiling	15 - 20
UHT Treatment	10 - 20
Retorting	30 - 50

Table 13. *The Effect of Heat Treatment on Vitamin C*

Process	% Folic Acid Loss
Pasteurisation	3 - 5
Short Boiling	ca. 15
UHT Treatment	10 - 20
Retorting	40 - 50

Table 14. *The Effect of Different Heat Treatments on Folic Acid*

The effects of the different heat treatments of milk (table 14) reported for *folic acid* have to be used with care (79, 155). Methods and procedures of analysis are not always comparable or adequate.

Biotin and *nicotinic acid* are regarded as heat stable and unaffected by commercially applied heat processes (58, 122, 209).

A comparison of the various heat treatments on the vitamin content of milk (table 15) showed the following losses in percent (151). Some of the results differ considerably from those obtained in other studies (see above). Different methods of analysis have been used by different researchers. The oxygen content of milk can and does affect some of the vitamins to a varying degree. In addition, the vitamin content of milk varies widely over the lactation period.

Vitamin	Past.	Direct UHT	Retort	Spray Drying
Vitamin A	0 - 5.7	1.0 - 2.8		
ß-Carotene	neg.	6.1	neg.	
Vitamin D	neg.	neg.	neg.	
Vitamin E	neg.	5	neg.	
Vitamin C	16.6	10 - 30.1	30 - 100	20
Vitamin B_1	11.9	9.4 - 18.0	20 - 45	10 - 15
Vitamin B_2	1.0	0 - 2.7	15	
Vitamin B_6	0 - 8	3.2 - 7.3	15	
Vitamin B_{12}	5	18	20 - 100	30 - 35
Nicotinic Acid	neg.	4.0	10	
Panthothenic Acid	neg.	3.6	10	
Folic Acid	5 - 12.2	12 - 17	40	
Biotin	0 - 10	0 - 10	0 - 10	10 - 15

neg. = negligible

Table 15. *The Effect of Different Heat Treatments on Different Vitamin losses in %*

11. Product Changes During Storage

Summary

All food products change during storage. Depending on the product in question, these alterations may be favourable improving flavour, texture or digestibility, but they are usually undesirable. Often these changes are caused by chemical reactions. Such reactions are very much affected by temperature: the lower the temperature the slower the changes. Attention must also be paid to biochemical reactions caused by the activity of enzymes, some of which are very temperature-resistant.

As far as long-life products are concerned, milk and milk products have been studied very extensively. During storage, three different kinds of product alterations are essential and of major importance: the effect of storage on milk protein, the flavour and the vitamin content. These and other changes are discussed in some detail.

1. General

In addition to changes inflicted by processing, other changes take place during the storage of milk and milk products. In general, the speed of such changes depends on the storage temperature. Since most of the reactions are chemical by nature, it is fair to assume a Q_{10}-value of around 2 to 3. Each increase in storage temperature by 10°C doubles or triples the speed of the chemical reactions. On the other hand, if the storage temperature is lowered by 10°C, the chemical shelf life is doubled or even tripled. Some reactions require a minimum temperature (threshold value) to start at all. This temperature can be provided by processing the product.

Chemical Changes: Q_{10}-Value = 2 – 3

In milk and milk products, natural and microbiological enzymes are present in varying amounts. The most studied and best known are *proteases* and *lipases*. Some of these enzymes change the product rapidly even at low temperatures. In contrast to chemical reactions, enzymatic activities have optimal temperatures.

2. Water

The water content of milk should not change measurably during storage. This very much depends on the packaging which should provide a barrier against loss of water. Chemical changes do not measurably affect the water content.

3. Lactose

At high storage temperatures (>30-35°C), the Maillard reaction starts. This is a chain reaction in which sulphur containing amino acids and reducing sugars such as lactose are involved leading to a browning of the milk. These reactions proceed faster and at lower temperatures with galactose and glucose (hydrolysed lactose).

Some people suffer from lactase deficiency (lactose intolerance). As a consequence, lactose is not split into glucose and galactose and it reaches the colon. Breakdown of lactose by the intestinal flora results in the formation of gas. By adding lactase to milk, the content of lactose can be reduced. Long-life milk offers a good possibility: after UHT treatment, lactase can be added to the milk. Lactase is heat-sensitive and inactivated by UHT treatment. Consequently, the enzyme has to be added subsequent to UHT treatment. After sterile filtration, small amounts of lactase are injected into the aseptic transfer line or, better, into

Lactase Deficiency (Lactose Intolerance)

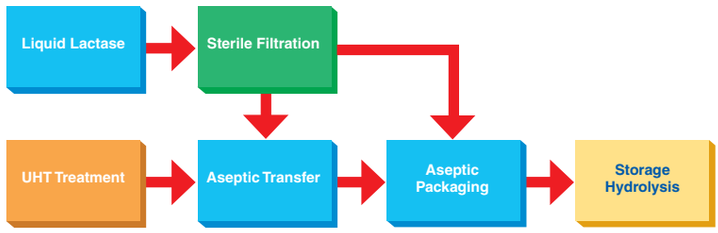


Figure 1. *Hydrolysis of Lactose in Long-Life Milk*

commercially sterile milk during the aseptic filling operation (195) (figure 1). During storage, the lactose is hydrolysed into galactose and glucose. These monosaccharides more easily start the Maillard reaction and, consequently, browning and flavour changes during storage proceed more rapidly and develop at lower temperatures. In addition, the milk develops a slightly sweeter taste.

4. Milk fat

Increase in Free Fatty Acids

During storage, a slight increase in the content of free fatty acids is normal (56). If, however, the untreated milk (intermediate product) contains a relatively high content of thermo-resistant lipases, these will cause a more rapid increase in the free fatty acid content with the development of a rancid flavour as a consequence. This increase is also temperature-dependent (table 1).

The effect after 10 weeks of direct and indirect heating and storage temperature on the content of free fatty acids in long-life milk has been studied (table 2) (218).

Free Fatty Acids: Flavour Threshold

These values are difficult to understand since the higher heat load of indirect systems should lead to a more effective inactivation of the enzyme. One possible answer is that different types of raw milk (intermediate products) have been used. An increase of 0.05% in the free fatty acid content can be recognised by expert graders. Normal consumers are far less sensitive. Different fatty acids have distinct flavour threshold values (table 3) (112).

Unless the flavour changes render the product unacceptable, this lipolytic activity is of no nutritional significance.

Storage Temperature	Free Fatty Acids
4°C	0.32%
20°C	0.41%
38°C	0.59%

Table 1. *Free Fatty Acids in Long-Life Milk: Effect of Storage Temperature*

UHT	4°C	20°C	38°C
Direct	0.28%	0.35%	0.82%
Indirect	0.37%	0.43%	1%

Table 2. *Free Fatty Acids in Long-Life Milk: Effect of Direct and Indirect Heating*

Fatty Acid	in Water	in Oil
Acetic Acid	54 ppm	3 ppm
Butyric Acid	7 ppm	0.6 ppm
Caproic Acid	5 ppm	3 ppm
Caprylic Acid	6 ppm	350 ppm
Capric Acid	4 ppm	200 ppm
Lauric Acid	-	700 ppm

Table 3. *Flavour Threshold Values: Free Fatty Acids*

5. Milk Protein

Sedimentation Gelation

During storage of long-life milk, sedimentation and/or gelation may occur. Sediment formation is frequently observed in long-life milk, though wide variations exist in respect to the amount (56, 24). A slight sediment is normal. At pH values below 6.65, extremely heavy sedimentation consisting mainly of milk protein (casein and albumin) has been encountered (273). Aggregation leading to

sedimentation depends not only on the heat treatment as such but also on a number of other factors such as pH, Ca^{++} concentration, raw milk (intermediate product) quality, etc. Sedimentation can be reduced by adding 0.5 g/l Na-citrate, Na-bicarbonate (224, 225), or Na-diphosphate, while adding $CaCl_2$ increases the amount of sediment (table 4).

During storage, sedimentation can cause a "sandy" flavour defect to develop. A correlation between alcohol stability (protein stability of the untreated milk, see the section on "Quality Control") and the amount of sediment has been demonstrated (figure 2) (250).

During storage, the rennet coagulation time first increases then decreases often below the coagulation time for raw milk. This phenomenon depends very much on storage temperature. In addition, the coagulum obtained is rather soft (56).

When long-life milk is stored at a temperature of 20 - 30°C or higher, a slight flocculation up to complete coagulation may occur. The phenomenon of "sterile coagulation" is described as proceeding in two steps: first flocculation, followed by gelation. The following changes were observed in UHT-sterilised, age-thickened milk:

a) a proteolytic casein decomposition with an increase in the soluble, non-protein nitrogen;

b) a decrease in the relative concentration of β-casein;

c) a change in the α-casein; and

d) an increase in sensitivity of the casein to calcium ions.

Gelation can, to some extent, be overcome by the addition of 0.05% sodium polyphosphate, whereas orthophosphates make the problem worse (272). Gelation is probably caused by the action of heat-stable proteases.

Proteases from *Bacillus cereus* were found to be rather temperature-resistant. In milk, heating to 146°C for 3 seconds at a pH of 6.6 resulted in only a 50% loss of proteolytic activity. Thermo-resistant proteases are also formed by *Pseudomonas fluorescens* (239). More and more microorganisms are shown to be able to produce such enzymes. The half-life of the enzyme in skimmed milk at a temperature of 149°C was 7 seconds (41). Cold storage of untreated milk of good quality for up to 72 hours at 4°C did not lead to more pronounced proteolytic activity, while raw milk intentionally enriched with psychrotrophic microorganisms showed a significant increase in non-protein nitrogen (NPN) compared to the untreated control milk (57).

Inactivation of thermo-resistant proteases at low temperatures (60 min at 55°C) has been proposed, resulting in a 70% inactivation of thermo-resistant proteolytic activity in milk (40).

Factor	Amount of Sediment
No Addition	1.15
$CaCl_2$	1.64
Na-Citrate	0.11

Table 4. *The Effect of Calcium and Citrate on the Amount of Sediment*

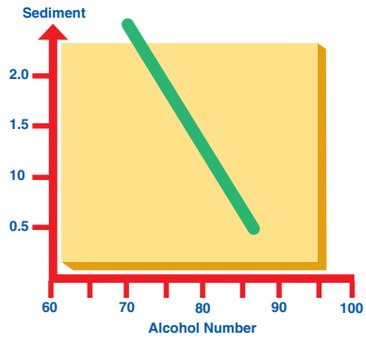


Figure 2. *Relation Between Amount of Sediment and Alcohol Stability*

Gelation = Sterile Coagulation = Age Thickening

Thermo-Resistant Proteases

6. Minerals

No information is available on the effect of storage on the mineral content of long-life milk.

7. Freezing point

Storage of long-life milk at temperatures of 5°C or 25°C did not affect the freezing point. However, a significant density gradient was observed which increased during storage. The gradient was more pronounced at 25°C than at 5°C. This gradient was caused by creaming as well as by sedimentation of casein and casein-fat complexes.

8. Sensory Characteristics

Acceptance of Milk is Subjective!

The subject of flavour is controversial since it is related to consumer acceptance, and is thus very subjective. Consumer groups in different areas and countries react surprisingly differently to certain flavour changes in milk.

Expert Evaluation!

Storing long-life milk with a fat content of 3.5% at 6°C or 20°C respectively for up to 9 weeks, did not result in any difference in flavour recognised by a consumer panel group (125). Expert panel evaluation may, and often does, give an indication of the kind of flavour changes that have taken place in the product (milk) studied. They do not usually provide a suitable means of relating these changes to consumer acceptance.

Cooked Flavour

The cooked flavour of long-life milk arises as a consequence of UHT treatment. Free sulfhydryl groups (-SH), hydrogen sulphide (H_2S) and carbon disulphide (CS_2) are the most important compounds responsible for the cooked flavour. During storage, this cooked flavour diminishes in intensity (114), a reaction which is accelerated by the presence of oxygen (115). An oxygen content of ~2 to ~4 ppm appears to be optimal.

Disapperance of the Cooked Flavour

During the first 2 weeks the effect of storage temperature on the intensity of the cooked flavour is negligible, but after 6 weeks of storage the milk stored at a low temperature (4°C) is preferred (114).

The time required depends on the presence of oxygen. Due to the low dissolved oxygen content resulting from expansion cooling, this period is significantly longer in directly heated milk. Cooked flavour is not always regarded as a defect, particularly by consumer groups used to the consumption of boiled or retorted milk.

As judged by an expert grading panel, in a very general, simplified, and schematic way, the flavour of long-life milk shows the following three development stages (figure 3):

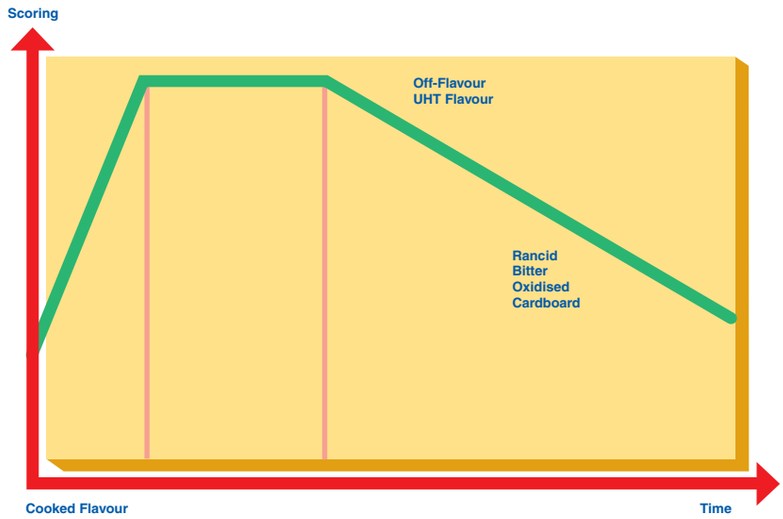


Figure 3. *Schematic Flavour Development in Long-Life Milk*

Oxidative flavour changes occur only during storage (262). Milk protein and lipids are both subject to oxidation. Oxidative flavour changes are dominated by oxidation of the milk lipids. Head space (with air), dissolved oxygen in the milk, and permeation through the package (container) may provide the oxygen necessary for such reactions.

Oxidative Flavour Changes

Two oxidative flavour changes occur in long-life milk (262), both of which are accelerated by light. The "sunlight flavour" is caused by the oxidation of methionine and tryptophan resulting in a "burnt protein" flavour. Light is needed to start the process. "Oxidised" is a flavour defect caused by the oxidation of lipids. The "oxidised" flavour also develops slowly without light. An oily, cardboard-like flavour defect results which is attributed to the oxidation of phospholipids present in the fat membranes. The development of detectable flavour changes may take more than six months if the container provides reasonable protection against the penetration of light and oxygen.

Depending on the quality of the untreated milk (intermediate product), long-life milk may contain greater or lesser residual lipolytic and/or proteolytic activity (presence of thermo-resistant enzymes) (191). Lipolytic activity leads to an increase in free fatty acids resulting in a "rancid" flavour. An increase in the free fatty acid content by 0.05% causes recognisable flavour changes.

Thermo-Resistant Enzymes

Proteolytic enzymes attack the protein. The first change is the development of a "bitter" flavour (267). Some destruction of heat-resistant enzymes has been observed after UHT treatment: inactivation of heat-resistant proteases in normal long-life milk averaged 87-90% (267), a value which appears to be rather high.

If long-life milk is stored for longer periods of time at high temperatures, the Maillard reaction may start. The result is a change in colour (browning) and flavour (poorly studied and identified).

The intensity of heating determines the sensory quality of long-life milk: the more intense the heating, the more unfavourable the taste of freshly processed long-life milk. The sensory quality following storage at ambient temperature is better as the heat intensity is higher (190).

9. Vitamins

The fat-soluble vitamins A, D, and E appear to be stable during the storage of long-life milk.

Fat Soluble Vitamins
Water Soluble Vitamins

The situation is more complicated as far as the water-soluble vitamins are concerned.

Up to ~15% loss of vitamin B_1 was registered during four weeks of storage at ambient temperature. The same storage conditions led to a ~13% loss of vitamin B_2.

Vitamin B_3 was stable during 180 days of storage at room temperature. The vitamin B_6 content is clearly affected by storage. The breakdown of vitamin B_6 appears to be independent of oxygen and storage temperature. Losses of up to 50% after three months, and 60% after six months have been reported.

Vitamins B_1, B_2, B_3, B_6 and B_{12}

Storage losses of vitamin B_{12} seem to be limited to the first four to eight weeks but may reach up to 60% after six months.

During storage, considerable losses of vitamin C are encountered. Decomposition of vitamin C seems to be directly related to the availability of oxygen (219) (dissolved oxygen, head space and permeation of oxygen) and is fairly independent of storage temperature (115). In pasteurised milk, the vitamin C content dropped from ~20 mg/l to ~5 mg/l within 10 days of refrigerated storage, a loss of about 75%. A comparison of pasteurised, directly and indirectly processed long-life milk is shown in figure 4 (219).

Vitamin C

With low levels of oxygen, i.e., ~ 1 ppm or less, the ascorbic acid content remains practically unchanged during 2-3 months at ambient temperature.

Direct heating and expansion cooling result in a very low oxygen content, ~0.1 ppm. Barrier attributes of the packaging material are important to prevent passage of O_2 into the product. In indirectly heated long-life milk (without deaeration), the oxygen content depends on the temperature of the milk fed into the steriliser. It is high (up to 6-9 ppm, the milk is usually saturated with oxygen), resulting in the almost total decomposition of vitamin C within days of storage.

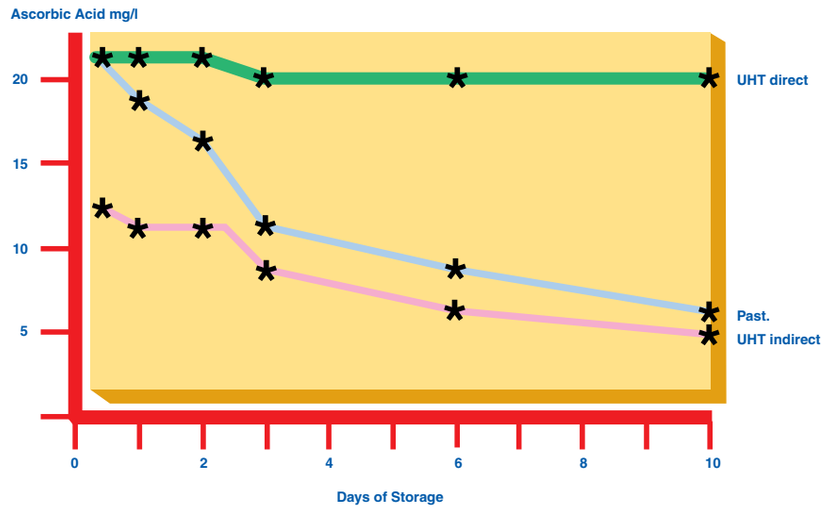


Figure 4. *The Loss of Vitamin C as Effected by Dissolved Oxygen*

The relation between the availability of oxygen and the decomposition of vitamin C has been demonstrated in an experiment (table 5). Sterile milk was filled into bottles with and without head space, the original oxygen content in the milk being <1 ppm (37):

Storage Time Days	No Head space		Head space	
	Vitamin C mg/l	Reducing Compounds mg/l	Vitamin C mg/l	Reducing Compounds mg/l
0	13	20	13	20
1	12	17	11	15
18	13	17	2	3
45	12	16	0	2
90	11	15	0	2
150	14	16	0	2

Table 5. *The Effect of Oxygen (Head Space) on the Decomposition of Vitamin C*

Folic Acid

Losses of folic acid are very similar to those of vitamin C. They depend on the presence of oxygen. At an oxygen concentration below 1 ppm, folic acid is rather stable. In milk saturated with oxygen, i.e., containing about 6 to 8 ppm, an almost complete loss of folic acid occurs within 2-3 weeks. Both biotin and nicotinic acid are stable during storage.

12. Quality Control

Summary

The goal of quality control is to ensure that the materials and products used in the manufacturing process, and those released on to the market, are within given specifications. Meaningful quality control work can only be done if quality specifications are available. In practice, quality control establishes sampling points at which material is extracted for analysis. Some of these sampling points are holding points: passage beyond them requires authorisation. Often the analysis needs time for completion: products have to be put on hold until further passage has been authorised.

The main limitations of quality control procedures are:

- some of the analytical procedures are quite expensive;
- time may be required to obtain analytical results;
- some sampling procedures are not very effective;
- the results are always "after the event", i.e., quality control is not preventive.

1. General

Definition of Quality

The quality of a product has been defined as its ability to fulfil consumer expectations, needs and wants (184) and match the promises made by the producer (185). The ISO 9000 standard defines quality as the "totality of characteristics of an entity that bear on its ability to satisfy stated and implied needs". Quality of foods is the composite of those characteristics that differentiate the individual units of a product, and have significance in determining the degree of acceptability of that unit by the buyer (100). Another definition requires the absence of pathogenic organisms and toxic substances, a sensory factor which is generally acceptable and a nutritional value which is real and meaningful (33). However, it should be borne in mind that consumers find it easier to recognise poor quality than to tell what good quality is (184).

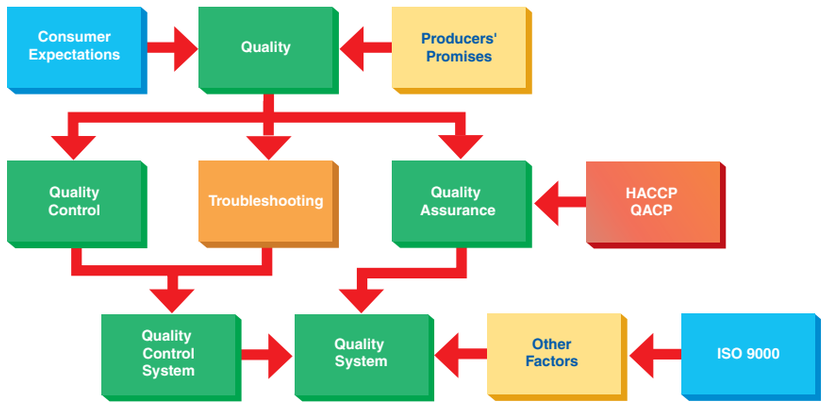


Figure 1. *Factors Having an Impact on the Quality of a Product*

The quality of any product is a complex matter and many factors must be taken into consideration (figure 1).

Quality Control
Quality Assurance

Definitions of quality control and quality assurance are not generally accepted. The purpose of quality control is to ensure that raw materials and intermediate and end products meet their respective specifications, whereas the aim of quality assurance is to prevent the production of sub-standard products. To implement an effective quality control programme, it is essential to specify standards of quality and performance (142). A clear distinction should be made between the definition of a product and its quality specifications. Definitions are outlined in product standards and legislation, and they provide basic requirements. A food product which does not comply with its definition is simply not that product. Quality specifications have to be more stringent and should be established individually by each company. They are part of a company's quality policy!

Product Definition
Quality Specifications

The main intention of a commercial producer is to avoid product spoilage. However, often government and international bodies require, for example, microbiological limits that are more restrictive than product spoilage. Examples are:

- IDF's (International Dairy Federation) microbiological *definition* of long-life milk: after a specified period of incubation, the total count is not to exceed 10 colonies/0.1 ml (140, 147). By definition, long-life milk with higher counts is *not* long-life milk!
- The US FDA (Food and Drug Administration) requires that long-life products are commercially sterile (21 CFR § 113).

Long-Life Products Microbiological AQL

Acceptance quality levels (AQLs) have to be established for all pertinent quality parameters. For long-life products, a microbiological AQL should be stated as a maximum or potential spoilage rate (142). It has been suggested (262) that the specification of long-life milk should be based on a defect rate that does not interfere with marketing. Such a specification would differ according to the company, product and area in which the commodity is marketed.

Quality Control Responsibility

In the present study, the task of quality control is limited to determining whether or not a product (raw materials, intermediate and/or end products) complies with its specifications. This is a passive role! Quality control is performed by a limited number of people who have been specially trained for the task. The quality control manager is responsible for product quality and/or for ensuring that the product released on to the market complies with its (quality) specifications. A quality control system works in a differentiated way. In a production line, sampling points are established (figure 2) at various stages. Material is extracted from the sampling points or containers are sampled for analysis. Some of the sampling points may be "holding points", i.e., further passage of the product requires the authorisation of an appointed person.

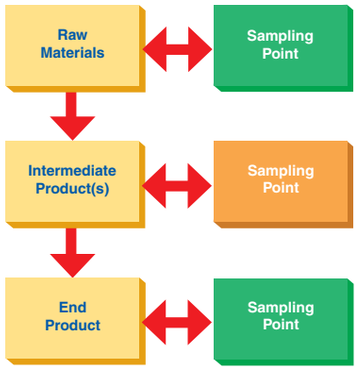


Figure 2. *Quality Control: Sampling Points*

Although intermediate products are also checked and analysed, quality control typically concentrates on raw materials and on end-product control. In a quality control system, "quality is achieved by control", but quality (sterility) cannot be tested "into" a product (200). At most, a sub-standard product can be prevented from being released on to the market.

Whenever possible, quality diagrams or charts should be prepared (figure 3). These charts should contain acceptance and "action" limits. They permit the detection of development trends such as microbiological counts.

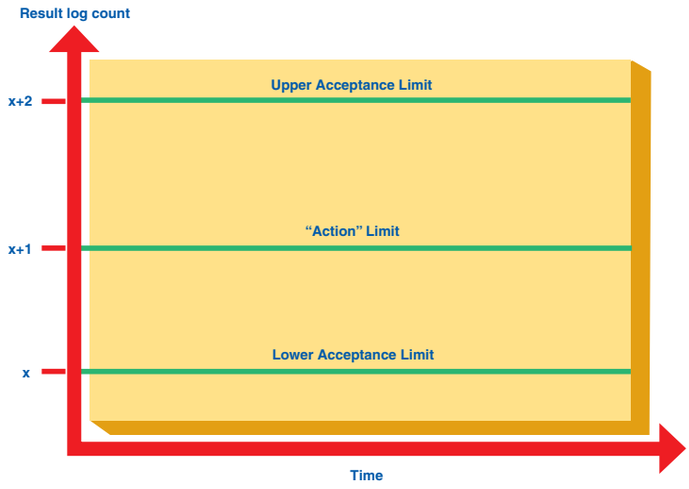


Figure 3. *Quality Diagram*

2. Raw Material Control

Raw Materials

Raw materials are the ingredients used in the formulation of a product, as well as all other materials needed for its production. Of course, a high-quality product cannot be produced from poor raw materials, but poor quality products can be made from high-quality raw materials.

Because of conflicting interests, one of the problems of quality control systems is the lack of co-operation and communication between departments. Raw materials are bought by the purchasing division. They are usually interested in obtaining the least expensive materials available: specifications are often of secondary interest!

The task of quality control is to ensure that the raw materials conform with their respective quality specifications. To this end, specifications are first of all needed! They should cover:

- physical; and
- chemical parameters; as well as
- microbiological aspects.

Quality Specifications

Quality specifications cannot be discussed generally, since they will vary in accordance with the quality policy of the company concerned and will vary from product to product. Though physical and chemical quality specifications are usually available, microbiological specifications are often lacking, especially for long-life products.

For long-life products, a microbiological AQL could be a maximum acceptable defect rate larger than 0. Such an AQL has to be defined by the commercial producer (189).

"Sterilisation" Becomes Reduction in Numbers

The objective of a heat-sterilisation process is to reduce the number of surviving microorganisms to a low, acceptable number. A practical way of expressing this is to give a relative number of non-sterile containers (200). It has been suggested that three different situations be distinguished: the public health aspect, and spoilage by mesophilic and thermophilic microorganisms. For these categories, three different AQLs have been proposed for the canning industry (sterilisation process survivors) (201):

- safe from a public health point of view; preserved from spoilage by *Clostridium botulinum* - the probability of a viable *Cl. botulinum* spore surviving in a container of food should be of the order of less than 10^{-9};
- preserved from spoilage by non-pathogenic microorganisms;

a) preserved from mesophilic, spore-forming organisms - the probability of mesophilic organisms surviving the preservation process should be in the order of less than 10^{-6};

b) preserved from thermophilic, spore-forming organisms - the probability of a thermophilic organism surviving the preservation process in food stored below the growth range of thermophilic organisms should be equal to or less than 10^{-2}. If the canned food is stored at high temperatures, the probability of a non-sterile unit should be less than 10^{-6}.

Defect Rate in Canning

It has been suggested that the end point of a heat preservation process be expressed as a specification, which on a unit basis is the probability of producing a non-sterile unit (142, 201). It has been stated that a failure rate of 1 in 10,000 containers produced is generally regarded as acceptable (177). This is a very questionable statement since a company's quality policy is involved! Maximum acceptable defect rates could be anything from 1 : 100 to 1 : 100,000 or even less.

The defect rate in aseptic packaging should not exceed 0.1% (59). The level of spoilage in canned foods is often quoted as one defect in 10,000. This tends not to take into account flat-souring which may occur if the contaminating

organisms do not produce gas. Taking this into consideration, the true figure is probably nearer to one in 5,000 (142). This standard is not an unrealistic one for aseptic products (142).

Consumer Complaints A Better AQL?

AQL's must be taken seriously. They are part of a company's quality policy. It should be kept in mind that with a linear increase in the quality level of a product, the costs of installation, raw materials, quality control and production increase logarithmically. A suitable compromise must be established. Perhaps AQL's should be based on the reaction of the market rather than on quality control procedures. Consumer complaints could be used.

Goal of Production

AQL's should not be confused with the goal of production. Though a zero defect rate (in an unlimited volume of product) is not achievable, this nevertheless must be the goal of production.

Generally, raw material control can be performed with two different goals in mind: improvement of the general hygienic conditions of raw material production, or the suitability of the raw materials for the manufacture of specific products. Both, but especially factors of processability, depend very much on the product to be produced. For the production of long-life milk, some of these parameters are given in table 1.

Improvement of General Hygiene Conditions for Raw Material Production	Suitability to Produce a Certain Product: Processability
Total Count	Spore Count
Flavour	Protein Stability
Adulteration	pH
Somatic Cell Count	Dirt Particles
Etc.	Thermo-Resistant Enzymes
	Etc.

Table 1. *Quality Parameters for Raw Milk*

Of course, the total quality of the finished product is determined by both the general hygiene conditions of raw material production and their processability.

2.1 Spore Count

UHT Treatment of Milk = ~9 to 10 Decimal Reductions

As pointed out elsewhere, sterilisation processes such as UHT treatment are not absolute. What a sterilisation process achieves is a certain number of decimal (logarithmic) reductions in the spore count of the intermediate product. This number is determined by the time-temperature combination used in UHT treatment, the type and resistance of bacterial spores present, and the kind of product. Since these factors will vary from case to case and time to time, the result achieved by a UHT treatment will also change. Consequently, precise statements as to the number of decimal reductions resulting from such a process cannot be given. However, regarding the situation for milk, it is safe to base calculations on 9 to 10 decimal reductions for the total spore count.

Total Spore Count Thermo-Resistant Spore Count

In determining spore counts, a distinction should be made between the total and the thermo-resistant spore count. Thermo-resistant spores are more difficult to kill and, consequently, a lower sterilising effect is obtained (if all other parameters are kept constant). Spore count limitations are of importance for the raw materials (raw milk, milk powder, cacao powder, sugar, etc.,) but even more so for the intermediate product(s). It has been suggested that payment of untreated milk intended for the production of long-life milk should be based partly on its spore count (220). A bactofuge (170), or other means such as filtration, can be used in order to reduce the bacterial endospore count and thus improve the quality of the raw milk intended for the production of long-life milk.

Bactofuge

2.2 Protein Stability

Protein instability leads mainly to the formation of deposits in heat exchangers, thus reducing the interval between cleaning, i.e., shortening the available production time. A distinction should be made between two different types of deposit:

- *Deposit A* consists predominantly of protein but also contains fat, minerals and lactose. This deposit is formed on heat exchange surfaces in the temperature range of ~80°C to ~110°C with a maximum between ~95 - 100°C;
- *Deposit B* contains mostly minerals but also small amounts of fat, protein and lactose. This deposit starts to form at a temperature of ~90°C and increases roughly linearly with increasing temperature.

Protein Stability Alcohol Test

Protein instability results principally in the formation of deposit A. For cow's milk, the simplest method to determine protein stability is the alcohol stability test. In this method, alcohol stability (alcohol number) is expressed by the highest concentration of ethanol mixed with an equal volume of milk that does not give a precipitate. IDF recommends a minimum alcohol number of 72 whereas Tetra Pak suggests 74.

Protein stability also affects product changes during storage, such as sedimentation, flocculation, etc.

Heat Stability Test

More accurate than the alcohol stability test is testing for heat stability. A rare fault in the salt balance of milk will give an acceptable alcohol number in spite of low protein stability. In a heat stability test, the milk is heated to ~130 - 140°C for some minutes and checked for precipitate formation.

The test requires special flasks and safety measures. A heat stability of more than 9 minutes at 140°C is suggested for milk intended for UHT processing (273).

2.3 pH

The pH relates to sedimentation during storage. Even a small deviation from the normal pH increases precipitate of a protein/fat mixture. A "sandy" flavour defect results. It has been suggested that the drop in pH in the raw milk should not exceed ~0.02 units.

2.4 Dirt Particles.

Dirt particles are difficult to sterilise (heat penetration). Usually they are removed during pre-processing either by filtration or by separation (clarification).

2.5 Thermo-Resistant Enzymes

Thermo-Resistant Proteases and Lipases

In untreated milk and/or the intermediate product, different amounts of thermo-resistant proteases and lipases may be present. A number of bacteria (mainly *Pseudomonas* and *Bacillus* but also others) produce them. Longer periods of cold storage at the farm and dairy increase the risk of unacceptable levels of these enzymes forming. Lipases split fat. The resulting increase in free fatty acids causes rancidity. A bitter flavour and eventually sterile gelation is the consequence of proteolytic activity. Cold storage periods should be kept short and temperatures should be as low as possible. If longer cold storage is unavoidable, and if legislation permits, intermediate thermisation or pasteurisation should be considered. This will not affect the content of thermo-resistant enzymes as such but will reduce the number of bacteria (mainly *Pseudomonas*) capable of producing such enzymes. Total psychrotrophic counts of up to 10^6/ml have been found after only 2 to 3 days of cold storage (183).

3. Intermediate Product Control

Intermediate products are the products fed into the UHT steriliser. It is the intermediate product which is processed and not the raw materials! During pre-processing, such as storage, separation, etc., the quality parameters of importance may be, and often are, negatively affected. Consequently, checking important quality parameters at this stage is even more important than control of the raw materials (ingredients).

Quality limits for raw materials have already been presented above. The same limits apply to the intermediate product(s). One of the most important quality degradations which might take place during pre-processing relates to an increase in the bacterial endospore count.

Bacterial Spore Count

Since the microbiological result obtained from a given UHT treatment is determined by the spore count in the intermediate product, a quality specification should be defined. This quality specification depends on the AQL. Assuming a sterilising effect of 10 and packages containing 1 litre of product, varying maximum acceptable total spore counts result for different AQL's (table 2).

Goal of Production = Zero Defects!

It has been stated that a failure rate of 1 in 10,000 is a generally acceptable level for long-life milk (177). This statement is questionable! A clear distinction should be made between the AQL and the goal of production. "It is better to aim at perfection and miss than to try for something less and succeed" (269).

AQL	Total Spore Count
1:100	100,000/ml
1:1,000	10,000/ml
1:10,000	1,000/ml
1:100,000	100/ml

Table 2. *AQLs and Acceptable Spore Counts*

4. End-Product Control

4.1 General

A quality control system concentrates on controlling the end product. This implies ensuring that a product released on to the market will meet its microbiological quality specifications. As tools of end-product control, three different activities can and should be considered (figure 4).

Limitations of End-Product Control

End-product control has three main limitations and shortcomings:

- it is always "after the event" and, consequently, the procedure cannot be preventive;
- it is expensive, particularly if destructive test methods are applied;
- the results are often not very informative.

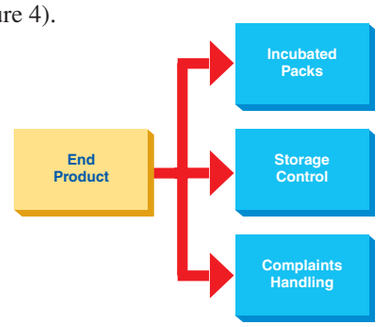


Figure 4. *Tools of End-Product Control*

Unavoidable Defect Rates

From a microbiological point of view, a certain defect rate is unavoidable but should not exceed a specified unsterility level in a released production run. In Germany, the technically unavoidable defect rate has been stated to be at least 0.1% (268)! A defect rate of 0.1% has been regarded as acceptable (201). These defect rates are rather too high! The number of defects in a production run of UHT milk should and can be less than 0.1%, and preferably less then 0.01% (86).

In any event, an AQL must be decided upon which, in turn, has to be checked by quality control. In quality control procedures, samples are taken and analysed. Based on the results obtained, the production run is either released on to the market or retained in part or in total for further analysis. How effective is such a procedure?

Sampling = Statistics

Whenever sampling procedures are used, statistics are also applied. Consequently, the results obtained can only be expressed in terms of probabilities. Obviously, the purpose of end-product sampling is to assess the quality of the production run from which the sample has been drawn and not the quality of the sample as such, which can be stated in absolute terms. Figure 5 illustrates the situation.

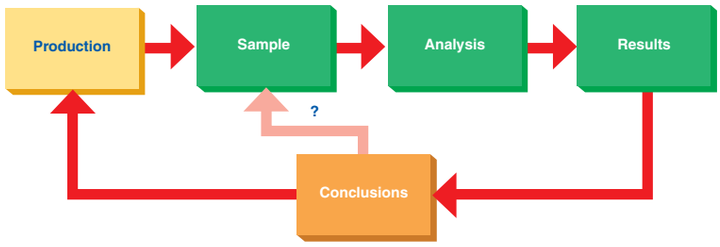


Figure 5. *Sampling Procedures*

4.2 Sampling Statistics

When developing a sampling scheme for microbiological quality control or performance testing, the following questions require an answer:

1) how many samples are to be taken;
2) how to sample;
3) how to incubate; and
4) how to evaluate.

4.2.1 How Many Samples?

When developing a sampling plan, a distinction needs to be made between the producer's risk (a sub-standard production run is released), and the buyer's risk (claims are made wrongly when the product is good) (265). The "Military Standard" which is used by some producers of long-life products deals with the buyer's risk. It is questionable whether this is the correct approach. In the following, sampling plans are discussed on the basis of the producer's risk.

Military Standard = Buyer's Risk!

In order to be able to decide on the number of units to be sampled, the statistical situation must be clarified. A distinction has to be made about whether the characteristic to be tested is a variable or an attribute. Fortunately, this distinction is rather easy: looking at a variable, one always obtains a set of figures which may have a given value within a certain range. An attribute is characterised by a "yes" or "no" reply: the characteristic under study is present or absent. Obviously, a package is either commercially sterile or not: a typical attribute situation.

Variable or Attribute?

Testing for commercial sterility is checking an attribute

In attribute situations, the conclusions which can be drawn from a sample depend upon the sample size only and not on the amount of product from which the sample is drawn. This can be shown by a simple example (figure 6). Consider two containers one of which is filled with 1,000, the other with 100,000 balls. The first contains 900 white and 100 black balls, the other contains 90,000 white and 10,000 black balls. After careful mixing, 50 balls are randomly "sampled" from each of the two containers. The probability of finding a certain number of black balls in each of the two samples is exactly the same. The probability depends on the ratio of black to white balls and not on the number of balls in the container.

Only the Sample Size is of Importance!

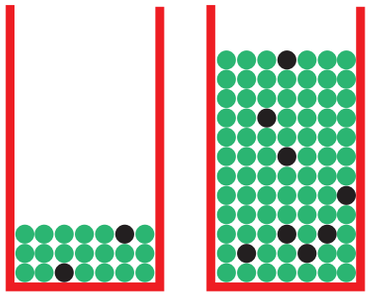


Figure 6. *Sample Size is of Importance*

In addition, unsterility is a rare event, at least as long as the unsterility rate is not above 10%, a level which can, but very rarely, occurs. Even if a defect exists in a production line, unsterility rates are usually low (262). Checking for unsterility is verifying a rare event.

Poisson Distribution

An attribute and a rare event are covered by Poisson distribution

Nevertheless, sampling plans have been recommended that are based on the magnitude of production. This could be justified if a higher degree of safety is desired for larger production runs since these represent a larger value. The low level of defects in a normal production run and/or the low AQL necessitate large numbers of packages being taken in order to achieve statistical significance (86).

Sampling Plan Based on Magnitude of Production

When a new production line is taken into commercial production, the operative staff lack experience. As production proceeds, operative knowledge is accumulated and used to improve the safety of production. Consequently, a higher risk of failure exists immediately after the commencement of commercial production. This is often met by tighter sampling procedures. An example of such a start-up plan is given in table 3 (101). The reduced risk under normal production conditions is accounted for by a reduction in the sample size (table 4) (101).

Start of Commercial Production = High Risk

Size of Production	Number of Samples	Accepted	Rejected
1,301 - 3,200	150	0	1
3,201 - 8,000	225	1	2
8,001 - 22,000	300	1	2
22,001 - 110,000	450	2	3

Table 3. *Recommended Sampling During Start-Up of Production*

Size of Production	Number of Samples	Accepted	Rejected
1,301 - 3,200	30	0	1
3,201 - 8,000	45	0	1
8,001 - 22,000	60	1	2
22,001 - 110,000	90	2	3

Table 4. *Recommended Sampling During Normal Production*

Sample Size	Accepted Defects	Detection Level %
150	0	< 1.5
225	1	1.0
300	1	0.8
450	2	0.9
30	0	< 77
45	0	< 56
60	1	38
90	2	66

Table 5. *Detection Levels (90% Probability) of the Sampling Plans Shown in Tables 3 and 4*

Developing a Sampling Plan

The above example is very debatable! First of all, because of the rather limited number of samples tested the detection level is very low. Based on Poisson distribution and a 90% probability level, table 5 gives the corresponding detection levels.

Secondly, and even more important, whenever a defective (unsterile) unit is found in a sample of rather limited size, it should be regarded as a serious indication of a potential problem. Defective units should never be accepted without further investigation!

In Germany, sampling varied mainly between 0.03% and 0.3% of the production run. Only part of the sampled packages were analysed for defects (261).

In order to be able to develop a sampling plan, two factors must be known: the level of defects to be checked for and the desired probability or risk (risk = 100 - probability in per cent). Probability is an expression of the likelihood that the conclusion drawn is correct, whereas the risk states the opposite (the likelihood that the conclusion is wrong). In statistics, probability levels of 90%, 95%, 99%, and 99.9% (10%, 5%, 1%, and 0.1% risk) are common. Probability levels below 90% (risk above 10%) should not be used: it is not important what you do but it is very important that you know what you are doing! Assuming the following parameters:

It Is Not Important What You Do, But It Is Very Important That You Know What You Are Doing!

- an AQL of 1 : 10,000; and
- a 90% probability level,

23,000 packages are needed (146) as found in the Poisson distribution ($n = 2.3 \cdot 100 / 0.01$) (see page 55 for the Poisson diagram). After proper incubation and evaluation of the sample, the production run is accepted if not more than one defective unit is found. The number of packages needed is prohibitive! Based on the buyer's risk, a different figure results. A defect rate of 0.1% can be checked by sampling 300 units (packages). After incubation, the batch is accepted if not more than one unsterile package is detected (265).

Checking the Producer's Risk is Prohibitive!

A commercial producer samples 100 packages from a production run. After incubation and evaluation, all the packages are judged to be sterile. The production run is released on to the market. What conclusions can really be drawn from this result? Again, the Poisson distribution diagram needs to be consulted. With 90% probability, the defect rate of the production run is less than 2.3% (there is a 10% risk that the defect rate is 2.3% or even higher; defect rate = x • 100/n = 2.3 • 100/100).

Conclusions Which Can Be Drawn from a Sampling Plan

Obviously, within an acceptable economic framework, incubated package control cannot be used to check for compliance with any reasonable AQL, i.e., any AQL above one defect in 100. For there to be any quality control work at all, two specifications are needed:

a) the AQL, which can be anything from 1:1,000 to 1:100,000; and

b) a release specification which is based on the economically acceptable sampling rate and which usually will be around 2% or more.

Quality Specification
Release Specification

These circumstances will become better if one looks at sampling plans on a continuous basis (197). A commercial processor continuously samples a certain number of packages from each successive production run. The aim of such a sampling procedure is to:

a) enable a release decision for a specific batch to be made (this situation has been discussed above);

b) detect changes in the defect rate; or

c) collect data over a longer period of time and, through its accumulation, gain information on average performance.

A producer examines 100 packages every day (table 6). If any defects are found, there will be an alarm. How long will it take to detect a change in the defect rate? How often will there be an alarm and how often will the alarm be a false one? Actually, the results are surprisingly good.

Continuous Sampling

Defect Rate	Signal Frequency
0.01%	100.0 days between alarms on average; varying between 6 and 300 days
0.05%	20.5 days between alarms on average; varying between 2 and 60 days
0.10%	10.5 days between alarms on average; varying between 1 and 23 days
0.20%	5.5 days between alarms on average; varying between 1 and 12 days
0.50%	2.5 days between alarms on average; varying between 1 and 4 days
1.00%	1.6 days between alarms on average; varying between 1 and 3 days

Table 6. *Effectiveness of a Continuous Sampling Procedure*

The following conclusions can be drawn from table 6 (197). Assuming different but constant defect rates in each successive production run, the following results can be noted:

a) moderately poor production runs will take some time to be detected;

b) temporarily poor production runs will often not be detected at all;

c) really bad production runs will be detected after one or a few days; but

d) even in good production runs, an alarm will appear every now and then. This should be taken as a signal: re-sample when a defect is found!

4.2.2 How to Sample?

Sampling can be done in two basically different ways:

1. random sampling; and
2. aimed sampling.

Random Sampling or Aimed Sampling?

4.2.3 Random Sampling

Random Sampling

In random sampling schemes, every package should have the same chance of being included in the sample. The pre-condition for random sampling schemes is uniform conditions of production. However, in practice the production of long-life products is often characterised by an uneven distribution of defective units caused by the large number of possibilities that exist for reinfection (262).

Random sampling plans are usually time-based (86, 261). For practical reasons, a certain number (usually 1 or 2) of packages are drawn at regular intervals. Although this is not a true random sampling procedure, it is close enough.

Accumulation of Results

Even though limited numbers of packages are included in every individual sample from which only restricted conclusions can be drawn, accumulation of the results obtained from random sampling will give the average unsterility rate of a production line and will eventually show if the production line (on average) fulfils the requirements of the AQL (86).

Random Sampling: A Tool for Quality Control

The results gathered from random sampling are used as a basis for product release: a typical tool of quality control. The entire production run has to be kept on hold until the results from incubation are available (86). The results from random sampling can be arranged according to filling machine so as to test relative performance, machine operators, shifts, etc. In this way, useful information for quality improvement can be gathered at low cost.

4.2.4 Aimed Sampling

Aimed sampling focuses on areas of increased microbiological risk. These situations arise whenever production conditions are changed. In a production line for long-life products, areas or functions at risk are:

Aimed Sampling Concentrates on Risk Areas

- starting production (142, 261);
- changing packaging material (reels) (261);
- changing the longitudinal strip;
- changing the intermediate product (raw material batch, new mix of product) (261), etc.;
- if applicable, changing from steriliser to sterile tank;
- changing back from tank to steriliser;
- end of production (261), etc.

Areas of increased risk have to be identified separately for each production line. Since aimed sampling concentrates on functions that have a greater chance of containing defects, the results obtained will show a higher defect rate than the one obtained by random sampling.

Goal of Aimed Sampling: To Quantify the Importance of Risk Functions

The goal of aimed sampling is to quantify the contribution of certain risk areas to the total defect rate. Direct actions for improvement become possible: one of the tools of process control (261) and quality assurance. It is essential that the results obtained from both these procedures (random and aimed sampling) are kept strictly separate. Accumulation of both is recommended. For aimed sampling, a programme should be devised based on the purpose of the procedure.

4.3 How to Incubate?

Prior to microbiological evaluation, the packages included in the sample must be incubated. For this reason, the

Incubation = Time and Temperature

1) incubation temperature; and
2) incubation time,

have to be decided on.

4.3.1 Incubation Temperature

Incubation Temperature: A Guess!

The choice of incubation temperature introduces a factor of selectivity. Ideal would be an "incubation" at ambient temperature during the entire intended shelf life of the product. The best incubation temperature is the optimum temperature (fastest growth) of the spoilage flora. The optimal temperature has to be known. This requires knowing which microorganisms are present in the product. If this is known, incubation is not necessary at all. The choice of an incubation temperature becomes a guess.

Psychrophilic Mesophilic Thermophilic Microorganisms

Microorganisms can be divided according to their growth temperature characteristics. Psychrophilic bacteria grow at low temperatures and have a growth optimum at <20°C, mesophilic organisms grow most rapidly at 30-37°C, while thermophilic microorganisms develop most quickly at temperatures >40°C. Mesophilic psychrotrophic organisms have a growth optimum at 30-37°C, but will also, though more slowly, develop at low temperatures. The temperature range to be considered is very wide: from <20°C to >40°C. A curve visualising the relation between temperature and multiplication of bacteria (figure 7) shows the following:

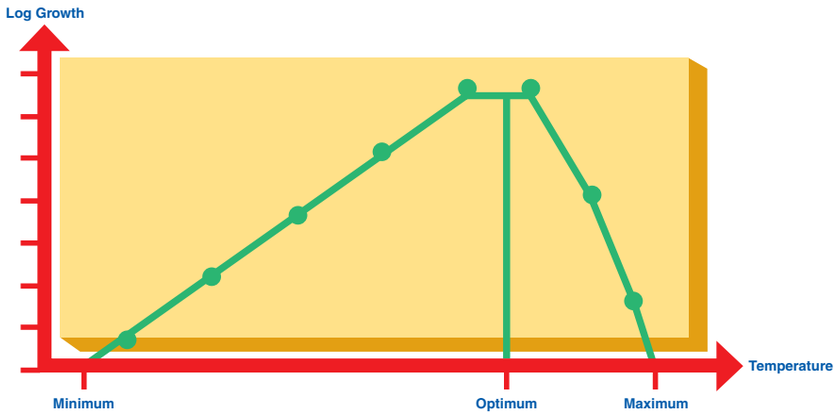


Figure 7. *Typical Growth Curve*

Growth Curve of Microorganisms

At a certain minimum temperature, growth will start. As the temperature rises, the speed of growth increases slowly until the optimum temperature is reached. The maximum temperature of growth is usually relatively close to the optimum: the incubation temperature should not be too high. The most common and frequent spoilage organisms in long-life milk belong to the mesophilic group. Consequently, an incubation temperature in that range should be chosen. Typically, a temperature of 30-35°C is optimal.

Best Incubation Temperature

An incubation temperature of 37°C is often used (261). Because of the rapid decline of growth above the optimum temperature, caution is recommended for higher temperatures of incubation. Attention should be paid to the ambient temperature prevailing. In tropical areas, higher incubation temperatures should be considered.

Since some psychrotrophic organisms will not grow at 30°C, an incubation temperature of 27°C has been recommended (159).

A relatively high percentage of defects was detected after incubation at 30°C (72%). 32% of the spoiled samples showed thermophilic growth only (177). In addition, most of the obligate thermophiles causing spoilage at 55°C will remain dormant under normal storage conditions of long-life milk: the climatic conditions during storage and distribution must be considered. Because of the statistics involved, only one incubation temperature, for instance 35°C, is recommended (177, 261). Thermophilic microorganisms in general and *Bacillus stearothermophilus* spores in particular may survive sterilisation processes and

Thermophilic Microorganisms

cause product spoilage if the packages are stored at high temperatures. An incubation temperature of 50-55°C is suitable and should be considered for the detection of such bacteria (159). Thermophilic spores may be found in the product surviving from the UHT treatment and/or the plant sterilisation process. To ensure that the number of survivors from the UHT process is at an acceptable level, it would be more efficient to check the intermediate product (raw materials) than to perform an end-product control. Plant sterilisation survivors are usually caused by insufficient cleaning. Aimed sampling at the start of the filling operation is often appropriate.

4.3.2 Incubation Time

In a quality control system, the results from the incubated package control is used as one of the parameters on which the release decision is based: the production run has to be kept in storage until the results are available. Consequently, the cost factor needs to be considered.

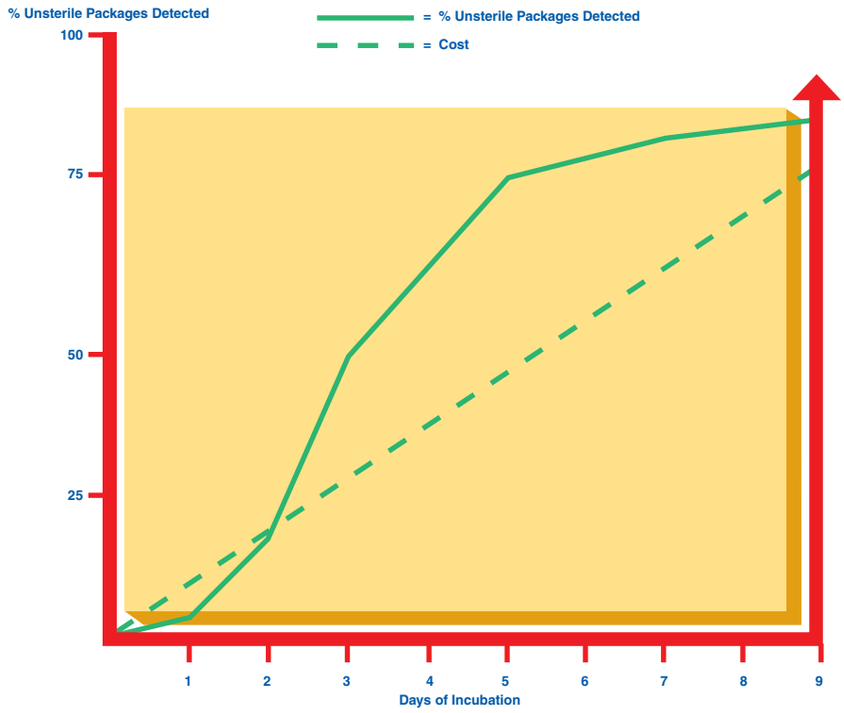


Figure 8. *Relation between Accuracy (Time) and Cost of Evaluation*

Assuming the same product and package volume, the cost of storage increases linearly with time. The detection level of possible defects follows a different pattern. After three days incubation, about 50% of the total number of unsterile packages in the sample can be detected. This value increases to about 75% after five days, and to 80-85% after seven days. Thereafter, further increases in the detection level are very slow and 100% is never reached because the choice of incubation temperature is a factor of selectivity. With this background in hand an incubation time of five to seven days appears to be the best compromise between cost and safety.

Incubation Time A Compromise!

In addition, prolonged incubation times may lead to a delay in the detection of a possible problem with all the consequences that this can mean. With this in mind, an incubation time of three days has been recommended as the shortest acceptable alternative (159). A further possibility is to incubate a limited number of additional samples for two days and evaluate by plating. If heavy reinfection has taken place, the incubation period is regarded as permitting sufficient multiplication to enable detection of spoilage organisms in a volume of 0.1 ml (159).

For low defect rates, particularly if caused by spore formers, an incubation time of five days is regarded as optimal (262). Using test tubes filled with long-life milk, inoculation tests with inactive (dormant?) *Bacillus* spores showed that

an incubation time of seven to nine days at 30°C was needed for an optimal recovery, with the streak method used for evaluation. (164). At longer incubation times, microorganisms may die off again. Checking different incubation times and using organoleptic evaluation, the following results were obtained in South Africa (table 7) (177).

Incubation Period (Days)	at 30°C Number Positive	at 30°C % Positive	at 55°C Number Positive	at 55°C % Positive
0	0	0	0	0
3	6	17.6	2	7.1
6	14	41.2	9	32.1
7	23	67.6	28	100.0
14	34	100.0		

Table 7. *The Effect of Incubation Time and Temperature on the Detection of Defects*

The official sampling plan and examination procedures for long-life milk in France are as follows (figure 8) (86). One per cent of the packages produced in a production run are sampled for incubation at 30°C for 7 days. The remainder of the run is stored and examined daily for blown packages. After the incubation period is over, 1/4 of the sampled units (0.25% of the total production run) are opened and examined for microbial multiplication. If no defects are found, the rest of the incubated sample (0.75% of the production run) is released together with the rest of the run. If defects are detected, the 0.75% incubated packages are opened and checked for sterility. Again, if no further defects are found, the production run is released. Detection of additional unsterile units leads to further trouble-shooting measures.

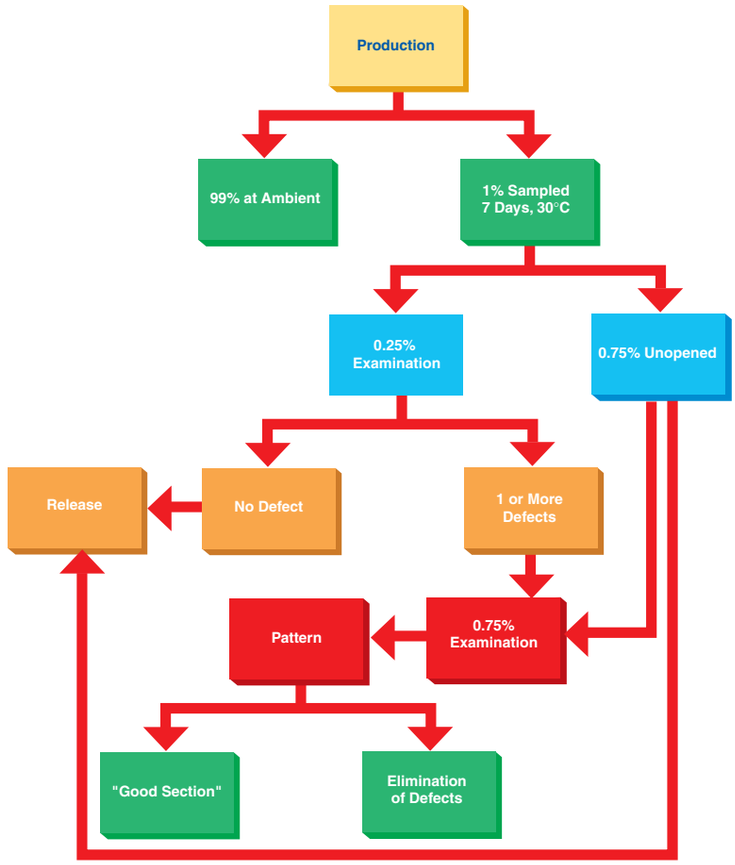


Figure 9. *Sampling Plan Required in France*

4.4 How to Evaluate?

As an indication of the bacteriological quality of long-life milk, the number of samples tested is of greater importance than the test procedure used for evaluation (262). Different methods are available for checking the commercial sterility of incubated packages. Such methods are usually destructive since the packages have to be opened. The most commonly used of these destructive test methods are:

Some Methods of Evaluation

- pH measurement (262);
- determination of oxygen tension (163);
- flavour, smell (sensory) of the product (261);
- titrable acidity (177);
- microbiological testing, etc.

Of all the tests recommended by IDF Standard 48:1969, the titrable acidity test was shown to be the most suitable for detecting spoilage of long-life milk after incubation at 30°C for three or five days respectively (table 8) (177).

The results shown in table 8 should be treated with caution. Generally, microbiological methods of evaluation are the most sensitive ones. They are also the most expensive. Three days of incubation is often too short a period for attaining a 100% detection level.

Test Method	3 Days, Number	3 Days, %	5 Days, Number	5 Days, %
Acidity	11	100	26	100
Alc.Stability (68%)	7	64	15	60
Organoleptic Test	6	54	14	56
Col. Count 100 /ml	7	64	15	60
Col. Count 10/ml	7	64	15	60
Dil.Count 100 /ml	6	54	14	56
Dil.Count 10/ml	6	54	14	56

Alc. = Alcohol, Col. = Colony, Dil. = Dilution

Table 8. *Detection of Unsterility by Different Test Methods and after Different Periods of Incubation*

Using impedance measurement (Bactometer®) and pH determination of inoculated long-life milk incubated for 48 hours, the following results were obtained (table 9) (137).

Test Bacterium	pH Decrease	Impedance Detection Time
Bacillus cereus	1.04	0.70 hours
Bacillus polymyxa	0.61	0.80 hours
Lactobacillus	0.21	6.15 hours
Micrococcus	0.01	-
Escherichia coli	1.01	1.01 hours
Citrobacter	1.02	2.15 hours

Table 9. *Comparison of Evaluation by pH and Impedance*

Impedance Measurement Versus pH Determination

The Bactometer® measures the change in a substrate caused by metabolites due to microbial growth: the impedance of the medium changes. Table 9 shows that development of *Micrococcus* remains undetected by pH measurement as well as by impedance determination. Growth of all other test organisms could be detected by both procedures. Testing a number of different bacterial strains, a count of at least 10^2/ml milk was necessary for detection (245). A count of 10^3/ ml was required for detection of *Pseudomonas putida* and *Streptococcus faecalis* while *Bacillus cereus* was already detected at a count of 10^1/ml. Consequently, since it is unlikely that the original infection is already at such detection levels, samples with low counts (such as long-life milk) have to be incubated before the Bactometer® can be used (245).

Methods of Evaluation ATP, pH, Plating

Determination of bacterial ATP is suggested as a suitable method for the detection of unsterile packages (table 10) (264).
Because of the reagents needed, the method is quite expensive.

Incubation, 30°C	ATP	pH	Bacterial Count
1 day	16/44	5/44	19/44
2 days	31/44	25/44	35/44
3 days	33/44	31/44	37/44
9 days	38/44	37/44	38/44

ATP = Adenosin-Tri-Phosphate

Table 10. *Detection of Reinfection in UHT Milk*

Incubation, 30°C	ATP	pH	Bacterial Count
1 day	1/47	0/47	10/47
2 days	27/47	2/47	33/47
3 days	47/47	2/47	47/47

Table 11. *Detection of Surviving Spore-Formers in UHT Milk*

One non-destructive procedure is used to some extent: the Electester (189). The packages do not need to be opened. Changes in consistency are measured by checking the oscillation curve of a test package and comparing it with the one obtained from sterile units. The equipment is rather expensive but can be used in quality control work (evaluation of incubated packages) as well as for checking and separating non-sterile packages from a bad production run.

Non-Destructive Testing

Which method or combination of methods should be used? Attention should be paid to accuracy and cost. The cost of incubated package evaluation can be expressed by a simple equation:

$$a = nb + nc$$

where *a* is the total cost of analysis, *n* the number of samples tested, *b* the cost of product losses (including packaging material), and *c* the cost of the analytic procedure used. Usually, the more accurate a method of evaluation, the more expensive it is. Microbiological test methods (streak or plating) are the most exact but also the most costly.

Which Method of Evaluation Should Be Used?

Frequently, a combination of pH determination and sensory (appearance, taste and smell) evaluation is used. Sound products should give readings of no more than 0.2 pH units below the normal (142). Evaluation procedures should be simple and inexpensive, such as the determination of pH and sensory assessment. On the other hand, the accuracy of evaluation also needs to be considered, but exactness should be sacrificed in order to be able to increase the sample size and thus the probability of including a non-sterile unit in the sample. It does not make much sense to use very exact methods of evaluation in order to detect a defective container which, in all likelihood, will not be included in the specimen. Any defective unit found should be subjected to microbiological analysis (see the section on "Troubleshooting") (159).

Accuracy of Evaluation Versus Probability of Presence in the Sample

End-product sampling procedures are expensive, particularly if the testing procedures are destructive. Neither are they effective, particularly if the characteristic to be tested is an attribute (133, 262). Since laboratory microbiological procedures involve destructive testing, it is evident that these cannot by their very nature be adequate to give a true statistical assessment of the short-term spoilage pattern (142). Through accumulation, the results can be used to establish average performance values, which may provide indications of long-term trends (142).

5. Storage Control and Handling Complaints

5.1 Storage Control

In storage control procedures, figures obtained from the storage area are used as a measure of product quality. Such systems can be of greater or lesser sophistication.

5.1.1 Storage Waste

The easiest and least expensive way to apply storage control is by using storage waste to determine the difference in numbers of packages entering and leaving the storage area. Such figures are often available but are rarely used for quality control purposes.

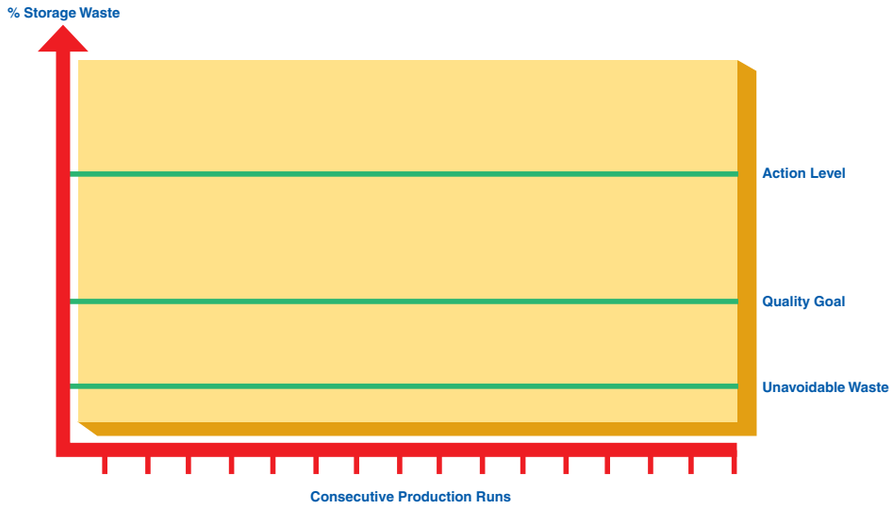


Figure 10. *Storage Waste Diagram*

Depending on the conditions prevailing, a certain loss of packages is unavoidable. When introducing a quality control system, quality charts should be prepared (figure 10).

Storage Waste: A Quality Specification Is Needed!

A quality level should be set up as a goal. In addition, a limit of what is acceptable should be established. Whenever this limit is reached or a clear rising trend in product waste is recognised, action (troubleshooting) must be taken. Of course, waste figures as such do not indicate the reason for product losses. These may be caused by such factors as:

Causes of Storage Waste

a) microbiological spoilage (blown packages);

b) careless driving with fork-lifts, etc.;

c) poor quality of pallets used for storage;

d) poor stacking of packages;

e) theft of product, etc.

If the storage waste exceeds the acceptable level, the reason for the loss has to be identified and proper measures taken in order to restore normal conditions.

5.1.2 Storage Control System

A storage control system is more demanding but also considerably detailed, and it enables valuable information to be gathered. Basically, a storage control system consists of three steps:

Storage Control System: Steps Involved

1) selection of defects;

2) classification of the defects selected; and

3) registration and documentation of the results obtained.

5.1.3 Selection of Defects

Only those defective units can be selected from the storage area which are detectable by observation. Discovery is determined by the:

a) type of spoilage (gas formation, blown packages);

b) outer wrapping used;

c) size of pallets;

d) layout of the storage area; and

e) procedures used during delivery of the product.

The best solution with regard to storage control would be to store each package individually. However, a factory does not produce goods for the purpose of storage control, and some sort of compromise has to be reached. Since factors affecting detection will vary from case to case, an optimal compromise must be worked out individually for each production plant.

Only Gas Formation Can be Detected!

As far as microbiological spoilage is concerned, gas formation is required for detection. Unusually high-acid products show a high percentage of blown packages: storage control systems become very effective. Gas formation is less frequent in low-acid products but nevertheless a sizeable percentage of unsterility is combined with gas formation.

5.1.4 Classification

The classification of defects is the most difficult task of the entire storage control system. Its aim is to determine the most probable reason behind the fault. Classification is teamwork. By inspecting the blown packages, possible defects can be detected. Basically, packages may be classified into different groups. Damage caused by:

a) operation of the aseptic packaging machine;
b) maintenance of the equipment;
c) faulty packaging material;
d) internal transportation;
e) outer-wrapping equipment;
f) handling during storage.

The above points relate to defects caused by the packaging machine and/or the packaging material. However, outer areas may be the cause of unsterility. In such cases, it is not possible to identify a package defect. The event should be recorded by:

g) no package defect detectable.

Depending on experience and knowledge of the classification group, the above areas should be split into sub-groups.

5.1.5 Registration and Documentation

The defect rate is very low during normal production conditions. Results obtained from a single production run are usually insufficient to permit or justify any measures to be taken for improvement. For this reason, accumulation becomes necessary. A suitable recording system has to be established. The purpose of such a registration system is to achieve improvement in production results by:

Quality Improvement Requires Documentation

a) providing feedback for the production department;
b) visualising performance levels and trends in the form of charts; and
c) establishing an information system for senior management by means of suitable summaries (monthly).

The organisation of a storage control system should be as straightforward as possible. The different departments involved must have clearly stated rights and responsibilities.

5.2 Handling Complaints

One of the measures of consumer acceptance and satisfaction with a product is consumer complaints. However, if a producer is to benefit from consumer complaints, he cannot ignore them. Systems for handling consumer complaints must be established to (133):

a) receive, read and respond to individual consumer complaints;
b) resolve complaints quickly and satisfactorily;
c) record, tabulate and evaluate the complaints on a time basis;
d) recognise serious complaints, “red flag” rapid trends, and initiate appropriate actions;
e) feed evaluations, rates and trends back to the QA/QC department, and to the manufacturing, marketing and management departments so that appropriate remedial action can be promptly initiated and carried out.

6. Hygiene Control

6.1 Control of Cleaning Efficiency

Microbiological methods are generally not informative, taking into account that the number of microorganisms on surfaces available for testing is very low.

Analysis of the rinse water after a CIP programme is sometimes recommended, but it is necessary to use a filter method and analyse rather large volumes.

Test Methods for Controlling Cleaning

Unfortunately, the results cannot be used for locating where in a cleaning circuit a problem is present. Consequently, in case of failure, the control and dismantlement of components in the circuit will become necessary.

Generally speaking, it is better to control the important parameters for the results of the cleaning operation. As with all chemical processes, these parameters are:

Control the Process Rather Than The Results!

a) the chemical;

b) the concentration of the chemical;

c) the contact between the chemical and the object being cleaned;

d) the contact time; and

e) the temperature during contact.

In manual cleaning operations, the only recommendation that can be given is to provide good cleaning instructions, adequate tools and competent supervision. Both in CIP and manual cleaning, the best information on the result achieved is obtained by visual inspection. Good torch light is an inexpensive and very helpful tool, especially for the control of cleaning efficiency in tanks. Pumps and valves should be dismantled and inspected at regular intervals. If visually recognisable residues are present, they provide proof that cleaning is less than satisfactory. Even if it is possible to sterilise a soiled surface, it will become more and more difficult as time passes by because of the constantly increasing amounts of dirt. Eventually, unsterility may result as a consequence of insufficient cleaning and plant sterilisation.

Inspection

6.2 Control of Airborne Contamination

Different types of air samplers are available on the market. For screening the microbial load in the air, it is often sufficient to use fallout plates. Petri-dishes using the medium preferred, usually a substrate for obtaining a total count or fungi, are exposed for a set time at points where the sedimentation of dust and aerosol is regarded as a potential risk.

Microbial Load in Air: Methods

Often in food processing areas, an exposure time of 15 minutes is sufficient. After exposure, the plates are closed and incubated at a suitable temperature. The number of colonies is counted. Depending on the AQL, a fallout of 50 CFU/15 minutes might be acceptable. In high-acid food production areas, the fallout count of fungi should not exceed 15 CFU/15 minutes.

6.3 Personal Hygiene

The control of personal hygiene is usually restricted to the hands and fingers. The simplest method is to prepare plates, as described for the control of air. The personnel to be tested are “finger-printed” on the substrate surface of the plates. After incubation, the number of colonies should be low; only a few per finger if the right disinfectant has been used in the correct way. High counts should trigger off an education programme and/or a change in disinfectant.

Quality does not have to be as good as possible,
but as good as is necessary!

13. Quality Assurance (QA) and HACCP

Summary

The primary task of quality assurance is to prevent any deviation from given quality specifications; secondary quality specifications should be tightened. In quality assurance activities, everybody working in and for a plant should be involved. Senior management has to accept overall responsibility for the quality of all the products produced: a precondition for the proper functioning of quality assurance. The workforce needs to be trained. Programmes should be planned well in advance and executed accordingly. Motivation of the staff is an essential part of any quality assurance system.

One of the principles of quality assurance is process control. Good Manufacturing Practices (GMPs) should be put down in writing. The introduction of the Hazard Analysis Critical Control Point (HACCP) concept should be considered since this is the most effective tool for quality assurance available today.

1. General

The production of long-life products involves high risk and potential failure. It demands a highly efficient performance at each stage of "unit operation". The physical and chemical parameters of all the pre-process stages, sterilisation procedures, packaging processes, and possibilities of reinfection must be carefully checked and recorded under the supervision of skilled control staff (86). Operational procedures must be documented and skilfully supervised. Control of production is much more effective and important than end-product control (262). Suitable means of assuring the production of safe and wholesome products of adequate quality should be introduced into every operation.

Process Control Is More Effective Than Product Control!

This is the task of quality assurance which should be designed to prevent the production of sub-standard products (133). Whenever possible, quality specifications should be tightened – an additional task for quality assurance.

Quality Improvement

According to the ISO 9000 standard, quality improvement work should be a continuous process and should consist of three stages that repeat themselves (figure 1):

***Figure 1**. Quality Improvement Loop*

The first step is an analysis of the present situation, followed by a clear and precise definition of a realistic goal, both with regard to its achievement and a time frame for achieving it. The third step is a description of the means necessary to accomplish the goal.

Quality assurance also results in a level of confidence which the producer or manufacturer has in the quality of the product which he markets either directly or indirectly to the consumer. The attainment of these objectives depends on three main essentials (33):

- the planning and installation of the processing plant;
- the management and personnel;
- the control procedures that are adopted and incorporated into a quality system (figure 2).

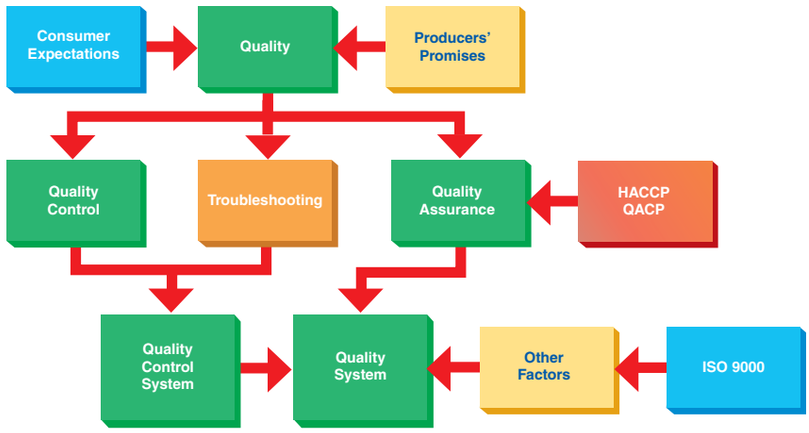


Figure 2. *Components of a Quality System*

A quality system places high demands on management and staff. It is a very complex subject.

Quality: from Production to Consumption

The quality of a processed material or a manufactured product depends on the care which is exercised from the time the raw material is received until the time the product is used by the consumer. Irreparable damage can be caused at any stage in the sequence of steps which take place during processing, manufacture (33), distribution and handling. To prevent this from happening, close teamwork is needed between production, quality control, quality assurance and marketing, in short by everybody working inside and outside the plant. Each person has his or her specific role! The full commitment and support of senior management is a precondition for the introduction of any quality (assurance) system (144, 235). A quality system can only be established if all the departments involved cooperate effectively and the process functions are reasonably understood.

Training and Education = Communication

For this purpose, staff training is essential. Communication experts estimate that people learn by using all their senses, although the outcome very much depends upon which sense is addressed in the learning process (132). As shown in table 1, the most effective means for achieving understanding is by the use of images ("seeing") followed by the spoken word ("hearing"). Consequently, in training, image-based material should be used as much as possible.

Learning and retaining information are not the same thing, as table 2 shows (132). These figures are impressive and certainly make for a strong case. Knowledge is of value only if it can be applied, which requires permanent awareness of the material learned. Practical exercise is the most effective way to ensure that the information learned is also remembered.

Sense	Learning
Touch, Taste, Smell	6%
Hearing	11%
Seeing	83%

Table 1. *Involvement of the Senses in Learning*

Parameter	Retained
Reading	10%
Hearing	20%
Seeing	30%
Seeing and Hearing	50%
Doing	90%

Table 2. *Retention of Learning*

Obtaining a better understanding of the processes involved in the production of long-life products is usually needed, and has the following advantages (156):

a) more effective and more rapid troubleshooting;

b) better system control and maintenance;

c) continuous improvement of processes; and

d) the ability of all employees to control and improve their operations and performance.

Obligations and Tasks of Senior Management

While the responsibility for product quality rests with the quality control manager in a quality control system, the senior management (general or factory manager) has to assume overall responsibility for quality in any quality assurance system. The main obligations of senior management are (33, 172, 235):

a) to formulate the company's quality policy and targets, both with regard to their actual achievement and a realistic time frame;

b) to establish an organisational structure in which rights and responsibilities are clearly defined and enforced;

c) to make available the personnel and funds, as well as the organisational conditions necessary for the successful and cost-effective realisation of the quality policy;

d) to supervise the quality assurance measures and the implementation of any correction which may become necessary;

e) to establish efficient systems of communication between management and members of the departments involved.

Quality Programme: Why?

Quality programmes are developed to ensure that:

a) the finished product complies with the design and concept as agreed on by company management;

b) the processes and products are kept within the tolerances stated in the written (quality) specifications;

c) the processes and products are safe, consistent and wholesome, and that defects are within tolerance;

d) proper corrective measures are formulated and executed if processes and/or products deviate from their respective specifications.

ISO 9000 outlines the requirements for a quality system. Tools for this purpose are GMPs, as well as the QACP (see below) and HACCP concepts (figure 3) which should be incorporated into quality assurance activities.

ISO 9000 describes a quality system in general for all manufacturing enterprises and requires its proper implementation. A certificate can be obtained if the documentation and its execution fulfil the specified requirements. GMPs are more specific and can deal with branches of the food industry and special product groups, etc. QACP and HACCP are very specific: they deal with one product and one production line at a time. The introduction (documentation and implementation) of an acceptable quality assurance system and particularly of the HACCP concept could lead to the reduction of official routine inspections thus considerably reducing inspection costs (133).

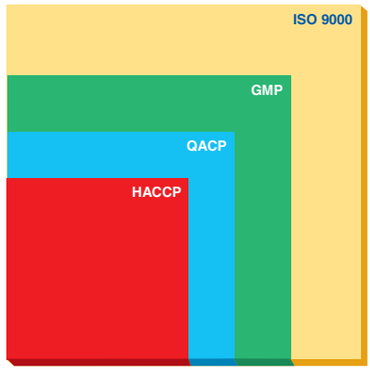


Figure 3. *ISO 9000, GMP, QACP and HACCP*

2. ISO 9000

ISO 9000 outlines the requirements for a quality system in general (with the exception of electrical products and electrical engineering) (246), and offers the possibility of certification. More and more companies obtain ISO 9000 certification and demand the same from their suppliers (246). Basically, the system requires a documented quality system (246) consisting of total verification of

processes, products, procedures, quality specifications and a suitable organisation. The standard includes three different levels: 9001 covers development, production, marketing and services; 9002 deals with production, marketing and services; while 9003 is restricted to services only. ISO 9001 lists 18 areas or points of concern (185):

- the tasks and responsibilities of senior management;
- quality assurance system;
- marketing;
- development;
- supervision of documentation;
- purchasing;
- products received from a third party;
- identification and tracing of products;
- checking of products (quality control);
- supervision of quality control work and procedures;
- handling defective (sub-standard) products;
- corrective action;
- handling of products (storage, packaging and distribution);
- quality records;
- internal quality audits;
- training and education;
- consumer services; and
- statistical methods.

The food industry is working to an increasing extent to get ISO 9000 certification. Unfortunately, the primary goal is sometimes the certificate and not the introduction of a quality system and the economic benefits connected with the process (172). It is not expected that the legislative body will require the mandatory introduction of ISO 9000, though the EU Commission is said to recommend to the extent possible the placing of orders with companies that have obtained ISO 9000 certification (185). "The ISO 9000 certification is not the end of the journey but rather the beginning" (186). It is an indication that a company is on the right track. The standard requires the preparation of three different documents (246):

a) the quality manual, a short outline of 25 to 30 pages which provides a description of the company's quality philosophy. All points in the appropriate ISO 9000 standard should be addressed;

b) quality procedures describing how the information in the quality manual is deployed and implemented. The procedures will answer the questions of who, where and when;

c) work instructions describing in detail how the procedures are to be accomplished.

3. Good Manufacturing Practices

GMPs Should Be in Writing

Good manufacturing practices (GMPs) are guidelines for the production of safe food products. GMPs state the steps that a producer of low-acid or high-acid foods should take in order to ensure that his products are hazard-free and to minimise commercial losses due to non-sterility or any other quality deviations of the product (222). While ISO 9000 is applicable to the manufacturing industries in their entirety, GMPs are more restricted. They may cover the food industry in general, a branch of it or a product group. Larger food-producing companies often have their own GMPs. Good manufacturing practices are often based on

experience. Though passed on by word and deed, they actually should be put down in writing.

Legislation on GMPs

In several countries (the USA, Canada, Taiwan, etc.,) the relevant legislative body has either passed regulations on GMPs for certain food groups (for example, shelf-stable, low-acid foods), or issued recommendations to this effect. At the present time, rules, guidelines and regulations covering GMPs for long-life food products are being formulated in an increasing number of countries, either on a voluntary or legislative basis. Though the legislators often have an understandable desire to regulate aseptic technology, they are usually lacking in the necessary knowledge. Therefore, the industry concerned should observe developments closely and, whenever possible, take an active part in them by co-operating with the legislators.

Co-operation between Legislative Body and Industry

Process Control

GMPs require that scheduled processes be established. (Critical) Control points and parameters need to be identified. For thermal operations such as UHT treatment, this relates to a time-temperature requirement. Instruments usually control such processes. It is important to calibrate such control instruments regularly. The settings for these parameters can be calculated, learned by experience and/or determined under experimental conditions (222).

The aseptic filling operation is a more complex process. The design of the aseptic filling equipment establishes the performance of the packaging material sterilisation process and all other pertinent parameters. As yet, no internationally accepted method for checking performance efficiency exists.

Whenever action is taken (and even by refraining therefrom), a risk is involved. Every operation will face problems of a greater or lesser kind. GMPs aim at minimising the frequency, severity and consequences of such occurrences.

Emergency Programme

Some of the GMPs are concerned with the establishment of an "emergency programme" which should be developed well in advance. An emergency programme deals with action to be taken if a sub-standard product is released on to the market, and it must include a product-recall plan. The point of a recall is to protect the consumer and thus minimise the consequences of failure by:

- locating and retrieving affected stock;
- notifying the trade and consumers of the recall, and providing accurate information to the regional and national health authorities. A recall may be classified according to the severity of the fault, such as mislabelling, product defects or a health hazard. The extent of the product recall should be defined by batch codes and other relevant identification (222).

Crisis Management

In a crisis situation, guidelines should exist as to what to do, who should do what, and how it should be done. Crisis management should be regarded as part of an emergency programme. A crisis-handling team should be established consisting of (203):

a) senior management, as decisions of major importance may have to be made;

b) the production manager;

c) the operational manager;

d) the quality assurance manager; and

e) the public relations manager.

If mismanaged, a crisis can easily develop into a catastrophe! The probability of such an event occurring can be minimised through preparation, as well as by training and educating the workforce in general, and the crisis management team in particular (269).

4. Hazard Analysis Critical Control Point

HACCP and Legislation

Today, the hazard analysis critical control point (HACCP) is the most powerful tool for quality assurance available. At the present time, the European Union (EU) has issued a directive demanding the mandatory introduction of HACCP in the food industry. The US FDA has already published a regulation requiring the fish industry to implement HACCP. It is to be expected that an increasing number of countries will render HACCP compulsory. The respective public health authorities are aware of the limitations implicit in end-product control. This is one of the procedures applied by public health inspectors today. A very limited number of containers (packages) are drawn on for analysis. If, for example, five packages are analysed and no defective package is found, what conclusions can be drawn? Using the Poisson distribution diagram (see the section on "Quality Control") and based on a 90% probability level, the defect rate = 100 • x/n = 230/5 = 46%! With 90% probability, the defect rate is no greater than 46%.

The Legislative Body and End-Product Control

There is a 10% risk that the defect rate will be even higher! Such a procedure is totally inadequate. The most likely and logical development for official food inspection will be to ensure that the food industry follows quality assurance procedures rather than analysing a limited number of end-product units.

With this in mind, some general reflections are necessary. Public health departments are likely to issue regulations for the introduction of HACCP. Health inspectors will check that the HACCP documentation is satisfactory and that the commercial operator implements and follows the outlined procedures. This requires access to the HACCP documentation. However, not all quality parameters are of significance to public health. Some of them may even be trade secrets. There is no need to make these accessible to the public health inspector. Therefore, it is recommended that HACCP documentation be restricted to those quality parameters that have public health significance. All others should be documented under a different heading: quality assurance control point (QACP) has been suggested. In both cases, methods and procedures are exactly the same. Below, the discussion will focus on QACP which provides a wider framework, HACCP being part of QACP. The system covers the entire process, from the production of raw materials to the consumption of the finished product, and it deals separately with one product, one production line and one hazard at a time.

HACCP - QACP

When considering the consequences of agricultural practices, processing technologies and environmental hazards on the safety and wholesomeness of food, decision-makers must assess the risks to public health and the likelihood of food spoilage that may exist or may be introduced by processing and handling food. Nothing is free from risk! Therefore, they must also decide on what is an acceptable level of risk for the consumer (138) and the producer.

Risks in Food Production and Handling Must Be Assessed

The process of developing a QACP (HACCP) programme for a specific food product and processing line requires technical expertise and considerable time. The most efficient use of QACP is to begin applying its principles during the product and process development stages. In this way, safety can be built into the product and process. The introduction and the necessary documentation of QACP require the execution of a number of consecutive steps.

Introduction of QACP

1) The pre-condition for the successful introduction of QACP is the commitment and active support of senior management. Senior management must also assume responsibility for the total quality of the products produced.
2) The introduction of the QACP concept is a matter of teamwork. A group has to be formed which is small, consisting of no more than three to five people. Representatives from quality control, production and marketing etc., should be part of it. It is important to indicate that QACP is not only a subject of production and quality control, but that it also includes many other aspects such as marketing and purchasing. The group needs to appoint a chairman

and a secretary and must have the authority and means (funds) to call on outside expertise if needed.

3) Identification of the product to be studied. The intended use should also be documented. QACP deals with the normal conditions of production and handling. Intentional misuse of the product, tampering and sabotage are not included in the system.

4) Description of the production line to be studied. This is done preferably by preparing a flow chart (157) which should be fairly detailed. It should be borne in mind that QACP (HACCP) deals with operations and functions and not with equipment. This should be observed when flow charts are prepared (see example 2 on page 105).

5) Identify the hazard to be studied. A hazard is a compound, substance, material, etc., the presence of which can lead to harm (HACCP) or product spoilage (QACP). There are three main groups of hazards:

   a) *physical hazards:* pieces of foreign material, the weight or volume of a package, etc.;

   b) *chemical hazards:* disinfectants, cleaning agents, heavy metals, antibiotics, pesticides, microbiological toxins, etc.;

   c) *biological hazards:* in long-life products, microbiological spoilage (QACP) and/or the presence of pathogenic microorganisms (HACCP).

   Only one hazard is studied at a time. In food commodities in general and long-life products in particular, the number of possible hazards is very large. In order to reduce the volume of documentation, hazards should be grouped together as much as possible. For the HACCP concept, this must be done in co-operation with the local public health authorities, whereas QACP is the concern of the producers only.

6) At this stage, a risk assessment should be made covering the entire line, from raw materials to consumption of the product. The purpose is to provide a ranking of the importance of different hazards in order to permit an optimal use of the resources available for quality improvement work. This risk assessment consists of two steps (138):

   a) *probability estimate:* this is an appraisal of the likelihood of the hazard under study becoming a reality, i.e., that the hazard reaches the consumer. The key word is "estimate". Usually there are insufficient data available for a more accurate appraisal. This should not prevent the group from making the best attempt possible. Such a probability estimate must be based on a defined time span. How often does the hazard become a reality within this defined period? It has been suggested that a scale ranging from 0.00 to 1.00 (table 3) be used. The scale used depends on the producer in question but should be the same for all hazards.

   b) *severity estimate:* is an appraisal of the consequences arising if the hazard under study becomes a reality. The appraisal could be based on a monetary scale, with values ranging from 0 to 10. Again, the information available may not be sufficient to arrive at an accurate severity estimate. The team should do the best it can. Since the total cost of failure, including marketing aspects, is to be appraised, an input from marketing is necessary. The severity (cost of failure, consequences) varies from country to country. One example is the insurance costs for road accident deaths calculated for 1995 (table 4) (156).

Time Scale	Probability Rating
every week	1.00
every month	0.90
every three months	0.80
every six months	0.70
every nine months	0.60
every year	0.50
every other year	0.40
every third year	0.30
every fourth year	0.20
every fifth year	0.10
>every fifth year	0.00

Table 3. *Risk Assessment: Probability Ranking*

Country	"Cost of Life" in Thousands of US Dollars
United States	2,600
Sweden	1,236
New Zealand	1,150
UK	1,100
Germany	928
Belgium	400
France	350
Portugal	20

Table 4. *The Cost of a Road Accident Death, 1995*

By multiplying the probability estimate and the severity value, a figure in the range of 0.00 to 10.00 is obtained which represents a relative estimate of the importance of the hazard under study.

Perhaps an easier way of arriving at a risk assessment is to adopt the following procedure:

a) decide upon the time span to be covered for *all* hazards in for example, a 5-year period;

b) determine the *frequency* (probability estimate): how often does the hazard under study become a reality within the total time span decided on, for example, 10 times during the 5-year period;

c) estimate the *average cost* (severity rating) of the incidents when the hazard under study has become a reality. The estimate should not only include direct product losses but should also contain the costs incurred from loss of market share, etc., for example, 10,000.00 monetary units;

d) multiply the frequency of the hazard under study by the average cost rating to obtain a total cost estimate caused by the hazard under study during the 5-year period, for example, 100,000.00 monetary units. In this way, a sum of money is arrived at which is an expression of the importance of the hazard under study and which can serve as a basis for future improvement.

Based on the risk assessment, three groups should be set up:

- *no action:* the basic principle underlying the setting up of tolerances in the food industry is based on the concept of threshold values (138). A certain, albeit low, defect rate is acceptable;
- development and implementation of an *action plan*; or
- *immediate action*.

The development and implementation of an action plan is a very important step: the QACP documentation is not an end in itself but a means to quality improvement. The action plan should clearly indicate the goal of quality improvement, a time frame and the costs involved in its execution.

7) Identification of (critical) control points. (Critical) Control points are stations, stages, functions, operations, processes, etc., where control can be exercised in order to eliminate a hazard or reduce the hazard to an acceptable level. It is recommended that a further risk assessment be attempted at this stage. However, it should relate to failure at the control point only. Often, the magnitude of a hazard is governed by more than one control point. In assessing the risk, the same scale of judgement must be used as above. The sum of the risk assessments of the control points for one particular hazard must be the same as the one arrived at previously (point 6).

8) Depending on the complexity of the technology, it might be necessary to identify (critical) control parameters. In UHT processing and aseptic packaging, thermal and chemical processes are used. These have the following (critical) control parameters:

   a) *thermal processes:*
      - time and
      - temperature;

   b) *chemical processes:*
      - type of chemical;
      - concentration of the chemical;
      - contact between the chemical and the object;
      - contact time; and
      - temperature during contact.

Again, at this stage a risk assessment should be performed. Using the same scale for evaluation, the sum of the risk assessments of the control parameters must be the same as the one obtained from the risk assessment of the (critical) control point. For each control point or parameter, the following values should be stated:

- *critical limit:* a critical limit separates acceptability from unacceptability. Critical limits must be taken very seriously! Often they can be obtained from existing legislation or the equipment supplier. In other cases they have to be determined usually by the quality control manager of the producer and approved by senior management;
- *target values:* target values are the set values of processes, i.e., the intended setting of the parameter;
- *preventive measures:* this section contains a list of all the means that are implemented to prevent the critical limit from being violated. It is important that only those measures that are listed actually be performed. To a large extent, quality improvement will rest on more efficient procedures to prevent process deviations;
- *corrective actions:* a list of procedures used to correct deviating processes as well as those measures to be taken with regard to a product that might have been produced under deviating conditions;

9) *Process monitoring* and *verification* are procedures for ensuring that the processes applied agree with the processes intended for implementation. Such procedures include all kinds of record-keeping, supervision, sampling plans, testing methods, etc. The personnel responsible for monitoring the (critical) control point, their duties, and the persons they are to contact if the operation is out of control should be identified.

10) *Verification* (validation) procedures need to be applied in order to ensure that the proper, intended process conditions have actually been applied. The US FDA has defined validation as follows (156):
"Process validation is establishing documented evidence which provides a high degree of assurance that a specific process will consistently produce a product meeting its predetermined specifications and quality attributes."

11) Once the QACP system has been put into effect, regular internal and external *auditing* is necessary. The purpose is:

   a) to ensure that the procedures outlined in the documentation are really followed;
   b) to include in the documentation any changes and improvements which might have turned up;
   c) to add hazards which might have been overlooked; and
   d) to inform senior management about the progress, shortcomings, etc., of the implementation.

A summary of documentation can be made in a QACP table, (see example 1 on page 104) which is given at the end of this section. For complex technologies such as the production of long-life products, a more detailed description is needed. The actual documentation file should be detailed. Of course, particulars depend on each specific situation (see example 3 on page 117).

5. Quality System

Quality System = *All* Quality-Related Aspects

QACP (HACCP) is a tool for quality assurance only. A quality system comprises *all* product-quality-related parameters and the total organisation of an enterprise. In addition to what has been outlined above, the following aspects need to be considered.

5.1 Top Management (25)

Consumer Needs and Expectations

The first task of top management is to decide what standard of quality is to be implemented. "An effective quality system should be designed to satisfy customer needs and expectations while serving to protect the organisation's interests. A well-structured quality system is a valuable management resource in the organisation and control of quality in relation to benefit, cost and risk considerations.

"The responsibility for and commitment to a quality policy belongs to the highest level of management. Quality management encompasses all activities of the overall management function that determine the quality policy, objectives and responsibilities, and implements them by means such as quality planning, quality control, quality assurance and quality improvement within the quality system" (ISO 9000).

5.1.1 Quality Policy

Senior management "shall define and document its policy and objectives for, and commitment to, quality. The senior management shall ensure that this policy is understood, implemented and maintained at all levels in the organisation."

"The supplier ["the organisation that provides a product to the customer"] shall establish, document and maintain a quality system as a means of ensuring that the product conforms to specified requirements. The supplier shall prepare a quality manual covering the requirements of this International Standard. The quality manual shall include or make reference to the quality system procedures and outline the structure of the documentation used in the quality system" (ISO 9000).

5.1.2 Organisation

An organisation needs to be established which is adequate to fulfilling the requirements of a quality system. To this effect, ISO 9000-1 states that an "organisation should:

Tasks of the Organisation

a) achieve, maintain and seek to improve continuously the quality of its products in relation to the requirements for quality;

b) improve the quality of its own operation, so as to meet continuously all customers' and other stakeholders' stated and implied needs (table 5);

Suppliers, Stakeholders	Expectations or Needs
Customers	Product Quality
Employees	Career/Work Satisfaction
Owners	Investment Performance
Sub-suppliers	Continuous Business Opportunity
Society	Responsible Stewardship

Table 5. *The Parties Involved and Their Different Interests*

c) give confidence to its internal management and other employees that the requirements for quality are being fulfilled and maintained, and that quality improvement is taking place;

d) give confidence to the customers and other stakeholders that the requirements for quality are being, or will be, achieved in the delivered product;

e) give confidence that quality system requirements are fulfilled."

The organisation must contain clearly defined areas of rights and responsibilities for each of the different departments and their respective managers. Co-operation is only possible if borderlines are distinctly established and, if necessary, enforced by senior management.

5.1.3 Responsibility and Authority

"The responsibility, authority and the interrelation of all personnel who manage, perform and verify work affecting quality shall be defined, particularly for personnel who need the organisational freedom and authority to:

a) initiate action to prevent the occurrence of product non-conformity;

b) identify and record any product quality problems;

c) initiate, recommend or provide solutions through designated channels;

d) verify the implementation of solutions;

e) control further processing, delivery or installation of a non-conforming product until the deficiency or unsatisfactory condition has been corrected."

The Conditions of the Job Must Be Defined!

"A management representative shall be appointed who, irrespective of other responsibilities, shall have defined authority and responsibility for ensuring that the requirements of the chosen ISO 9000 standard are implemented and maintained."

5.1.4 Training

"The supplier shall establish and maintain documented procedures for identifying training needs and provide for the training of all personnel performing activities affecting quality. Personnel performing specifically assigned tasks shall be qualified on the basis of appropriate education, training and/or experience, as required. Appropriate records of training shall be maintained."

All Personnel Need To Be Trained

5.1.5 Resources

"The supplier shall identify resource requirements and provide adequate resources, including the assignment of trained personnel, for management, performance work and verification activities including internal audits".

5.1.6 Documentation

Senior management should ensure that a proper documentation system is established and maintained. Such a system should address three types of document:

a) "procedural documents describing the quality system to be applied;

b) planning documents describing the planning and progress of all activities;

c) product documents describing a particular product."

"The supplier shall establish and maintain documented procedures to control all documents and data that relate to the requirements of the ISO 9000 standard chosen including, to the extent applicable, documents of external origin such as standards", etc.

5.1.7 Purchasing

"The supplier shall establish and maintain documented procedures to ensure that the purchased product conforms to specified requirements."

Purchasing According to Specifications and Not To Price!

5.1.8 Control of inspection, Measuring and Test Equipment

"The supplier shall establish and maintain documented procedures to control, calibrate and maintain inspection of measuring and test equipment used by the supplier to demonstrate the conformity of the product to the specified requirements."

Calibration and Maintenance

5.1.9 Handling, Storage, Packaging, Preservation and Delivery

"The supplier shall establish and maintain documented procedures for handling, storage, packaging, preservation and delivery of the product."

5.1.10 Control of a Non-Conforming Product

Handling Sub-Standard Products

"The supplier shall establish and maintain documented procedures to ensure that a product that does not conform to specified requirements is prevented from unintended use. This control shall provide for identification, documentation, evaluation, segregation (when practical), disposition of a non-conforming product, and for notification to the functions concerned."

5.1.11 Management Review

Management Control of the Quality System

"One of the important activities that the executive management of the organisation needs to carry out systematically is an evaluation of the status and adequacy of the quality system, including the quality policy, in relation to the expectations of the stakeholders. The results of internal and external audits are an important source of information. The outcome of the management review should lead to the increased effectiveness of the quality system. Records of such reviews shall be maintained."

5.1.12 Contract Review

"The supplier shall establish and maintain documented procedures for contract review ["systematic activities carried out by the supplier before signing the contract to ensure that requirements for quality are adequately defined, free from ambiguity, documented and can be realised by the supplier"] and for the co-ordination of these activities."

5.1.13 Auditing the Quality System

"Audits should be planned and carried out to determine if the activities and related results of the organisation's quality system comply with planned arrangements, and to determine the effectiveness of the quality system. All elements should be internally audited and evaluated on a regular basis, considering the status of importance of the activity to be audited. For this purpose, an appropriate audit programme should be established by the organisation's management."

5.1.14 Financial Considerations of Quality Systems

Goal of a Company = Profit = Money!

"It is important that the effectiveness of a quality system be measured in financial terms. The impact of an effective quality system upon the organisation's profit and loss statement can be highly significant, particularly by improvement of operations, resulting in reduced losses due to error and by making a contribution to customer satisfaction.

Such measurements and reporting can provide a means for identifying inefficient activities, and initiating internal improvement activities.

By reporting quality system activities and effectiveness in financial terms, management will receive the results in a common business language from all departments."

5.2 Middle Management (33)

In addition to the requirements mentioned above, demands essential to the implementation of any quality system for the production of heat-treated milk are:

a) the efficient maintenance and operation of processing and ancillary plants;

b) the systematic application of plant and process control procedures;

c) the careful monitoring of the composition and hygienic quality of the milk at all stages, from the time of its acceptance at the dairy until the time of consumption.

5.3 Plant Maintenance and Operation

5.3.1 Plant Maintenance

Plant Maintenance = Reduced Frequency of Plant Failure

From an engineering and electrical point of view, the absence of faults and breakdowns which can occur in any dairy plant is the yardstick by which plant maintenance can best be judged. The secret of success lies in the development and implementation of preventive (predictive) maintenance schedules. For preventive maintenance to work satisfactorily, it is of the utmost importance that an up-to-date inventory of the whole plant be kept with full details relating to inspection, repairs, renewals, etc. It is of course necessary to maintain adequate stocks of those materials or spare parts which may be needed.

5.3.2 Operation

The operation of a dairy processing plant requires knowledge and skill. In a modern plant, procedures covering at least the main units will usually be laid down in written form as a manual or guide and will include such matters as starting up, running and shutting down the various units, as well as information on how to detect faults, and so on.

Production Manuals Co-Operation and Communication

For plant performance to be kept at a high standard of efficiency, it is very important that there should be good liaison between supervisory, operational and maintenance staff and laboratory personnel. Good and accurate communication systems have to be established. It is of paramount importance that management should create and enforce clear borderlines between departments, that it act in a coordinating capacity, and establish distinct areas of responsibilities and rights. Full participation of management is necessary in these areas to obtain optimal results.

5.3.3 Plant Control Procedures

Process Control

The efficiency of plant control procedures depends on time-man-machine coordination. System procedures should be laid down by management with full staff co-operation, their application being in the hands of the machine operators.

The system has to be reviewed regularly (auditing), and improvements incorporated. Careful monitoring is required. This will amount to a record of performance, e.g., the amount of milk awaiting processing, the amount processed and the amount packaged, etc. It will also include time/temperature/pressure readings of instruments, recorders, etc., in accordance with the requirements set out in the plant operating manual. Similar routine procedures will apply to the cleaning, sanitation and sterilisation of the processing and packaging equipment, and other relevant parts of the plant.

Record-Keeping

Indications and recommendations to counteract any deviation in standards will also be forthcoming from the laboratory staff as soon as the results of their tests become known and have been interpreted. The elaboration and application of plant control procedures demand knowledge and experience if they are to work effectively and well. Only too often they are ineffective because communication and training are inadequate or there is a lack of knowledge, experience, or sense of responsibility at some point along the chain of command from management to operating and maintenance staff and laboratory personnel.

5.3.4 Monitoring Composition and Hygiene; Milk Acceptance and Storage

Relations between the Milk Producer and Dairy

Close liaison and a good system of communication and information must be established between the milk producer and dairy. Based on a quality payment, a situation should evolve where the quality and quantity of the intake remain more or less the same throughout the year. Aspects of processability should be included in the system.

Handling Raw Milk

At the end of the processing day, any untreated milk surplus should be pasteurised or heat-treated (thermisation) to be carried forward to the next processing day (33).

Raw milk should be stored at the dairy at a low temperature, preferably below 4°C and as a general rule not more than 6°C, for up to 24 hours.

Processability should be assured by proper laboratory testing. Records should be kept for both the raw milk and the intermediate product(s).

Bacterial Spores in Raw Milk

Vegetative bacteria are easily made inactive by UHT treatment, but careful consideration should be given to the spore content (86). The mesophilic spore count in milk produced under tropical conditions is higher than under temperate conditions. Bacterial endospore counts from less than 10/ml to a few thousands/ml have been reported (86).

In more and more countries, regulations require that untreated milk which is used for UHT treatment shall have a low mesophilic count and be produced from disease-free animals (tuberculosis, brucellosis, etc.,).

Increased Time of Cold Storage = Increased Content of Thermo-Resistant Proteases and Lipases

In general, the intention is to improve the standards of hygiene in milk production. On the other hand, the length of time in which untreated milk is kept in cold storage at the farm and in the dairy has increased. Coupled with this development, it is not surprising that there is an increasing number of incidences of heat-resistant enzymes produced by psychrotrophic (psychrophilic) bacteria, mainly *Pseudomonas* sp. The heat resistance of some proteases (table 6) and lipases (table 7) of bacterial origin is very high (86).

In spite of good raw material quality, under unhygienic pre-processing conditions the intermediate product(s) may contain extremely high spore counts: more than 1,000,000 spores/ml have been found (personal experience).

Organism	T (°C)	D (min)	z (°C)	Incomplete Inactivation
Pseudomonas	120	4	20	150°C, 2.4 sec
P.fluorescens	149	1.5	32.5	
Pseudomonas	150	1.7	32	
Pseudomonas	149	0.4		
Pseudomonas	150	0.5	32.5	
P.fluorescens	130	11	34.5	
P.fluorescens				132°C, 7 min
P.fluorescens	150	27	28	
P.fluorescens				140°C, 1.2 sec
B.cereus	150	0.015		

Table 6. *Heat Resistance of Proteases*

Organism	T (°C)	D (min)	z (°C)
P.fluorescens	130	16	
Pseudomonas	150	1.7	25
Pseudomonas	160	1.25	37
Micrococcus	150	1	63

Table 7. *Heat Resistance of Lipases*

In order to reduce the risk of problems connected with such enzymes from occurring, the cold storage periods for untreated milk should be as short as possible. If longer refrigerated storage at the dairy is necessary, an intermediate heat treatment such as pasteurisation or thermisation should be considered, if legally permitted. This will reduce the count of *Pseudomonas* as well as other vegetative bacteria and thus minimise the amount of thermo-resistant enzymes.

6. Comparison of a Quality Control System and a Quality System

Table 8 below attempts a comparison between a typical quality control system and a quality system. Today, most enterprises operate a combination of both.

The limitations of quality control procedures have been discussed above. This is especially true for end-product control. Having established sampling points, quality control works in a differentiated way while a quality system requires integration of all quality related aspects. Of great importance is that quality control activities are often regarded as "police" actions and everybody likes to fool the police! This also contributes to the isolation often encountered. Staff competence and communication between departments counteract isolation.

Quality Control System	Quality System
1. Oriented towards end-product control.	1. Integration of all quality-related aspects into a quality assurance system.
2. Quality is achieved by control.	2. Quality is produced.
3. Only sections of production are covered.	3. The total production process and more besides is included.
4. The quality control system is often isolated.	4. Quality assurance is always integrated.
5. Quality control is often viewed as a "police" function.	5. Communication, training and education result in quality thinking.
6. The control and checking of raw materials and raw material suppliers.	6. Co-ordination and confidence of all parties involved from supplier to consumer.
7. Responsibility for product quality rests with the laboratory.	7. Product quality becomes the responsibility of top management.
8. The laboratory is regarded as a cost factor.	8. Quality assurance prevents production of deviating products and becomes a cost saving.
9. Isolated from an information point of view.	9. Integrated from an information-exchange point of view.

Table 8. *Comparison between a Quality Control System and a Quality System*

Perhaps of greatest importance is the question of responsibility. In a quality control system, the responsibility for the product(s) released on to the market rests with the quality control manager. A quality system requires that senior management accepts the overall responsibility for quality and for all quality-related activities.

Example 1: QACP Chart

Control Point	Control Parameter	Critical Limit	Target Value	Preventive Measures	Corrective Actions
UHT	1. holding time	pressure drop, temp. differential	n.a.	intermediate cleaning, cleaning	*process:* none *product:* tightened inspection
	2. temperature	135°C	140°C	stop steriliser, maintenance of thermosensor, comparison with thermometer	*process:* change of thermosensor *product:* tightened inspection, re-work
Pack.Mat. Sterilisation (TBA-9)	1. chemical	H_2O_2	n.a.	certificate	replace
	2. concentration of chemical	30%	35%	regular control by machine operator	*process:* adjustment *product:* tightened inspection
	3. contact	passage through H_2O_2 bath	constant	n.a.	n.a.
	4. contact time	6.4 seconds	7.2 seconds	level sensor: stop filling process	none
	5. temp. during contact	62°C	67°C	thermosensor: stop filling	none

Example 2: Flow Charts

The purpose of preparing flow charts is to achieve a better understanding of the different process steps involved in the manufacture of a product. This knowledge should then be used to improve operational safety and the quality level of the product. In flow charts, the functions should be listed to identify the (critical) control points, but not necessarily the equipment. Flow charts have to be produced separately for each product.

From a microbiological point of view, the following should be kept in mind:

Unsterility Rate = Process Survivors + Reinfection;
Process Survivors = Process Parameters + Microbial Load.

Flow charts deal with processes and process parameters, not with the quality of raw materials or intermediate products. However, all process stages involved in the manufacture of a product should be included.

Shelf-Stable, Reconstituted Milk

A. Intermediate Product

In the flow chart presented in figure 1, the pre-process treatment, which results in the intermediate product is shown (i.e., the product which is fed into the product sterilisation process):

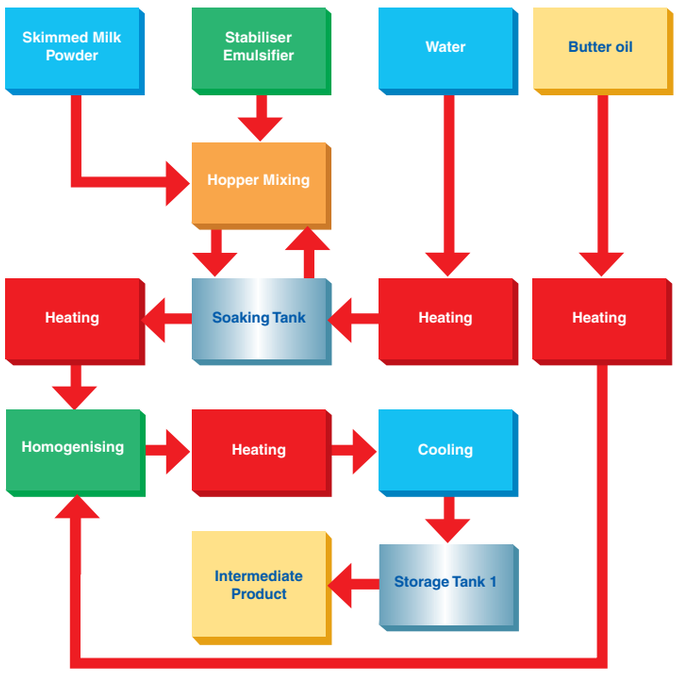


Figure 1. *Flow Chart: Manufacture of the Intermediate Product*

Consideration should be given to the pre-process treatment, i.e., the production of an intermediate product, the sterilisation of the product, the aseptic transfer (if applicable) and the aseptic packaging operation. Distribution can also be included. The intermediate product is used to produce shelf-stable, reconstituted milk. In order to achieve commercial sterility of the intermediate product, either a retort process or UHT treatment can be used. In both processes, the following equation applies:

Process Survivors = Process Parameters + Microbial Load.

B. In-Container Sterilised Product

In the retort process, the intermediate product is filled into a suitable container which is subsequently sealed hermetically. Both the product and the container are sterilised together in an autoclave procedure (figure 2).

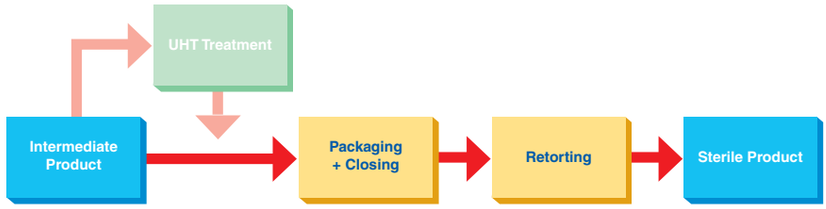


Figure 2. *The Retort Process ("In-Container Sterilisation")*

The intermediate product may either be packed and autoclaved directly or subjected to UHT treatment in order to reduce the load of heat necessary for sterilisation.

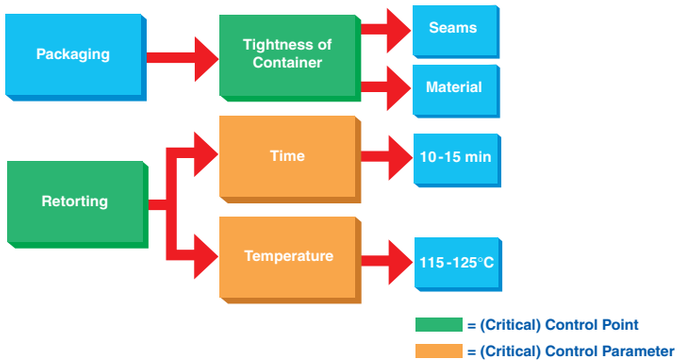


Figure 3. *(Critical) Control Points and Parameters*

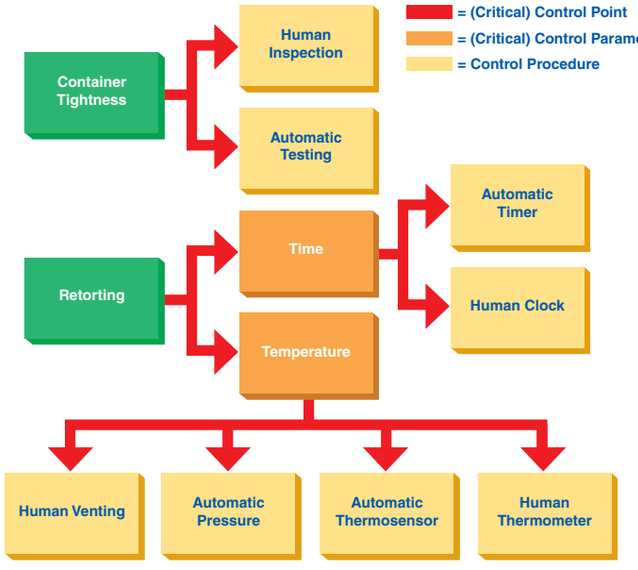


Figure 4. *Process Control*

1. Retorting: (Critical) Control Points and (Critical) Control Parameters

In order to achieve commercial sterility, the necessary combination of time and temperature is determined by the product itself, the size of the container and the type of retort used.

For each operation, (critical) control points and, where applicable, (critical) control parameters must be identified. These can and should be included in the product flow chart (figure 3).

1.1 Retorting: Process Control

Process control procedures can be included in a flow chart (figure 4). A distinction should be made between (critical) control points which are controlled by instruments (automatic control) and those which are subject to human control, usually machine operators. Control devices depend on the specific autoclave used.

Some equipment is operated totally by hand: all functions are controlled by the operator. Venting, i.e., the removal of air from the retort, is a precondition for correct temperature control, irrespective of whether this is done by an instrument or the operator.

A clear distinction should be made between a control function and a guarding device. A control function should ensure the application of the process intended, while guarding should prevent the production of a product under conditions of process deviation. The control of process parameters is not necessarily combined with a guarding function. In addition, instruments may or may not be connected to an automatic recording device.

C. Long-Life Product

The technology used to produce long-life products involves the separation of the processes of product sterilisation and packaging:

1. UHT Process ("In-Flow Sterilisation")

UHT treatment involves rapid heating, short holding at the sterilisation temperature followed by rapid cooling. When combined with aseptic filling, a long-life product is obtained (figure 5).

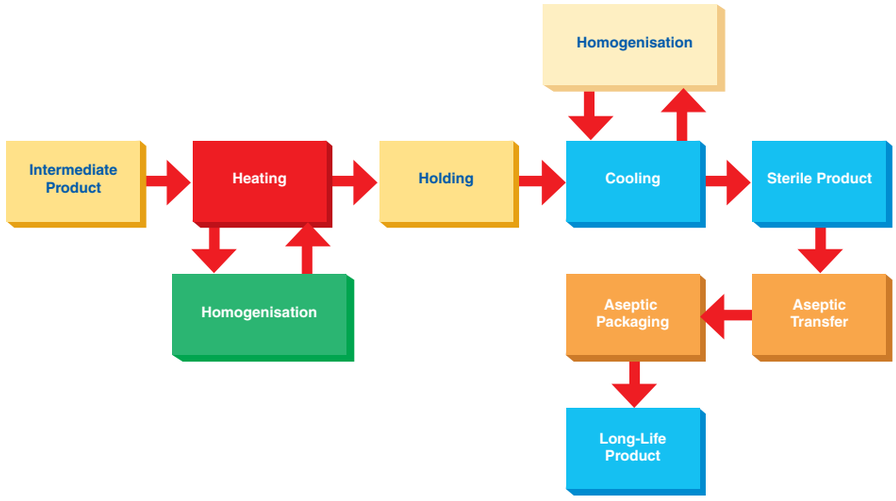


Figure 5. *The UHT Process*

A commercially sterile product is obtained by UHT treatment. Using aseptic conditions, this product is transferred to the packaging operation. In order to maintain the high level of microbiological quality, packaging has to be done under aseptic conditions.

In indirect UHT equipment, homogenisation may either be before (upstream) or after (downstream) the actual product sterilisation. Direct heating always requires downstream homogenisation.

1.1 UHT-Processing: (Critical) Control Points and (Critical) Control Parameters

The UHT process is a (critical) control point in the manufacture of long-life products. The (critical) control parameters should be included in the flow chart. As with any thermal process, two (critical) control parameters can be identified (figure 6): the sterilisation temperature and the holding time at that temperature.

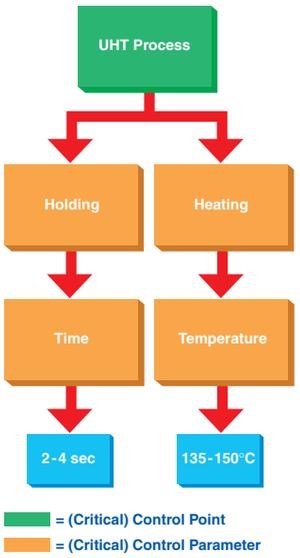


Figure 6. *UHT Treatment: (Critical) Control Points and Parameters*

1.1.1 UHT Treatment: Process Control

Usually, the sterilisation temperature is automatically controlled by a thermosensor which has regulating, recording and guarding functions. The pressure drop and temperature differential, even if measured, are neither registered nor connected to a guarding system (figure 7).

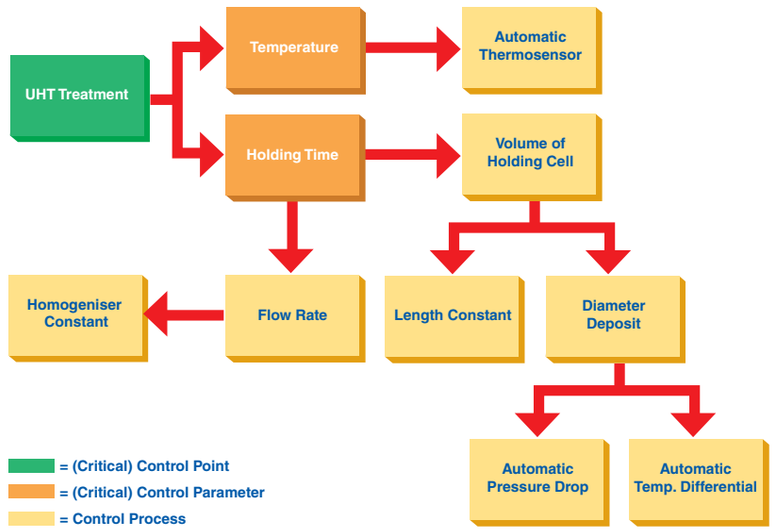


Figure 7. *UHT Treatment: Process Control*

2. Aseptic Transfer

The commercially sterile product obtained by UHT processing has to be transferred to the aseptic filling operation under conditions that exclude microbial infection (figure 8).

Figure 8. *The Aseptic Transfer Line*

Such a transfer line may or may not include one or several aseptic buffer tanks. From a microbiological perspective, reinfection of the product is a (critical) control point. To avoid or minimise recontamination of the product, the process of plant sterilisation and the tightness of the system have to be controlled.

2.1 Aseptic Transfer: Line Sterilisation

The pre-condition for a repeatable and efficient line-sterilisation process is the cleanliness of the system. Usually, cleaning and sterilisation cycles are identical. Depending upon whether or not a sterile tank is included in the transfer line, as shown in figure 9, one or two cleaning and sterilisation cycles are operated. (The filling equipment is cleaned and sterilised separately.)

Tightness of the system is achieved by changing gaskets, membranes, etc., regularly, an action which is not a processing function and, therefore, not included in the flow chart. The control of the cleaning process depends on the degree of automation of the cleaning unit.

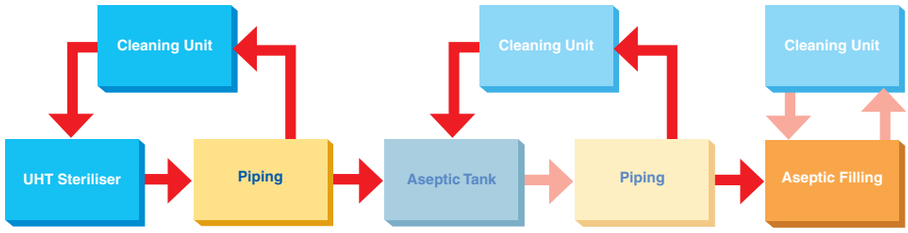


Figure 9. *Cleaning the Aseptic Transfer Line*

2.2 Aseptic Transfer: Control of the Line-Sterilisation Process

The sterilisation process of the aseptic transfer line is usually controlled and guarded by a thermosensor which is placed in the product return line. Usually this operation is also recorded.

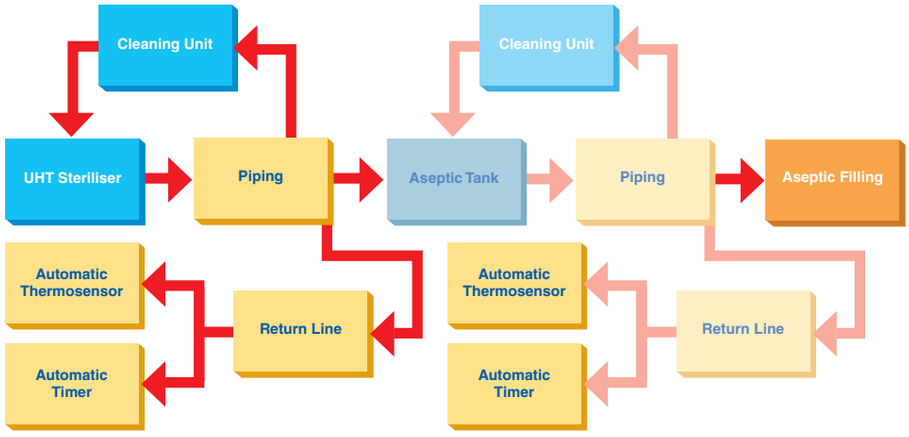


Figure 10. *Sterilisation and Sterilisation Control of the Aseptic Transfer Line*

3. Aseptic Packaging

Flow charts can be prepared for total production lines, for sections of such lines, for individual pieces of equipment, or even for specific functions of such equipment. Examples are given below, using Tetra Pak aseptic packaging systems as a model.

3.1 Tetra Brik Aseptic 3 (TBA/3)

In the aseptic packaging operation, three (critical) control points are identified (figure 11): packaging material sterilisation, a sterile environment in the area where the packages are formed, filled and sealed, and the production of tight packages.

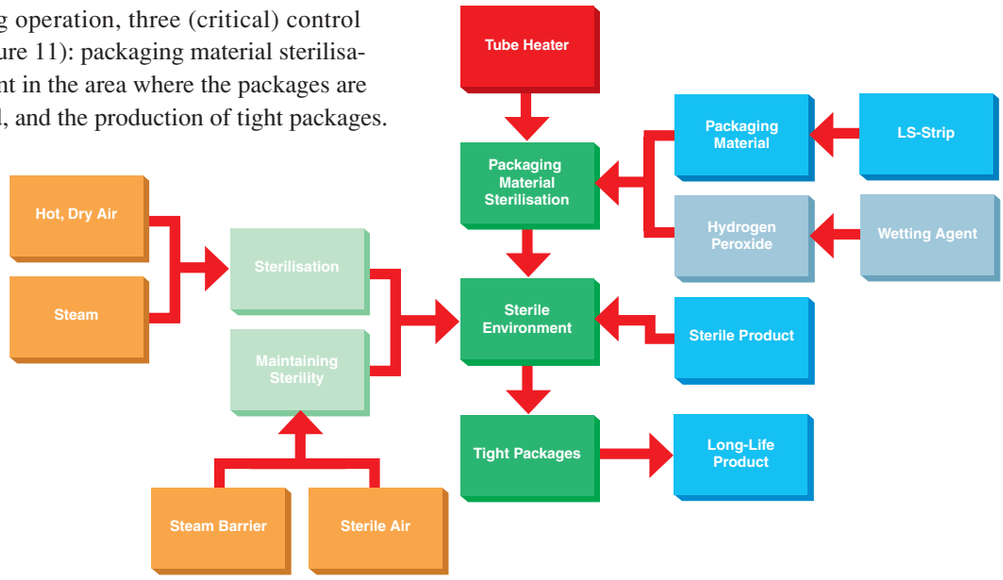


Figure 11. *Flow Chart for the Aseptic Packaging Process (TBA/3)*

3.1.1 TBA/3: (Critical) Control Point: Packaging Material Sterilisation

Packaging material sterilisation is a chemical process which has five (critical) control parameters (figure 12): the chemical used (hydrogen peroxide), the concentration of the chemical, the contact between the chemical and the packaging material, the contact time and the temperature during contact.

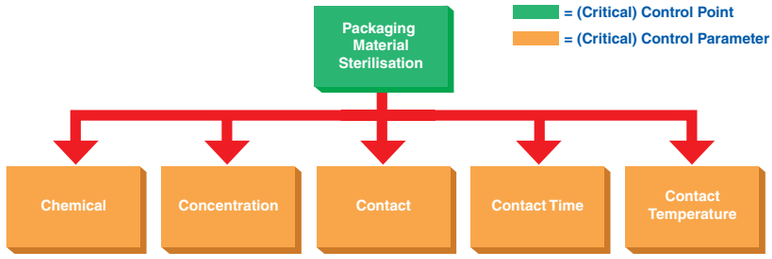


Figure 12. *(Critical) Control Parameters for the Packaging Material Sterilisation Process*

3.1.2 TBA/3: Packaging Material Sterilisation: Process Control

In order to ensure repeatability of the packaging material sterilisation process, suitable control procedures must be applied (figure 13).

The kind of chemical (hydrogen peroxide) is not controlled by the producer who relies on certification, i.e., the reliability of the supplier. The concentration is measured either by the machine operator or by laboratory staff, usually at the beginning of the packaging operation. The consumption of hydrogen peroxide should be determined by the machine operator either at the end of the production run or, better, at regular intervals. The contact time is a function of the packaging material and the machine construction and, consequently, constant: no control is needed. Temperature during contact is achieved by the tube heater element which in turn is controlled by a thermosensor which has a regulating and guarding function.

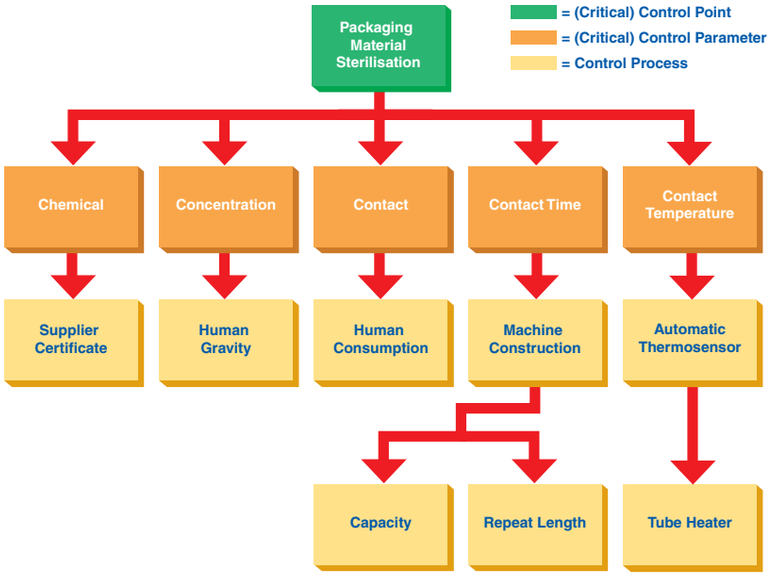


Figure 13. *Control of the Packaging Material Sterilisation Process*

3.1.3 TBA/3: (Critical) Control Points: Sterile Environment

The area in which the packages are formed, filled, and sealed must be sterilised before production commences, this is accomplished by steam and by dry, hot air. In addition, sterility must be maintained in this zone during the entire length of the production run. This function is achieved by an overpressure of sterile air.

Two different sterilisation cycles are needed, both of which are heat processes. The product valve is sterilised by steam while the filler is sterilised by dry, hot air. During production, sterility of the air needed is attained by an incineration process.

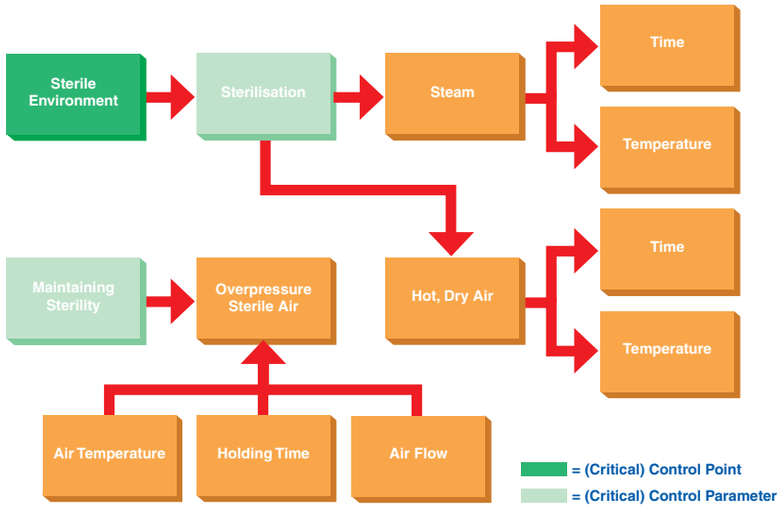


Figure 14. *Aseptic Packaging: Sterile Environment (Critical) Control Points and Parameters*

3.1.4 TBA/3: Sterile Environment: Process Control: Sterilisation

The precondition for repeatable and satisfactory equipment sterilisation is, generally speaking, its cleanliness. In addition, and in order to assure repeatability of the procedure, the process of sterilising the aseptic filler must be controlled and, to the extent possible, guarded and recorded. The process control procedures necessary are outlined in figure 15. The sterilisation process is controlled and guarded by thermosensors and by a timer.

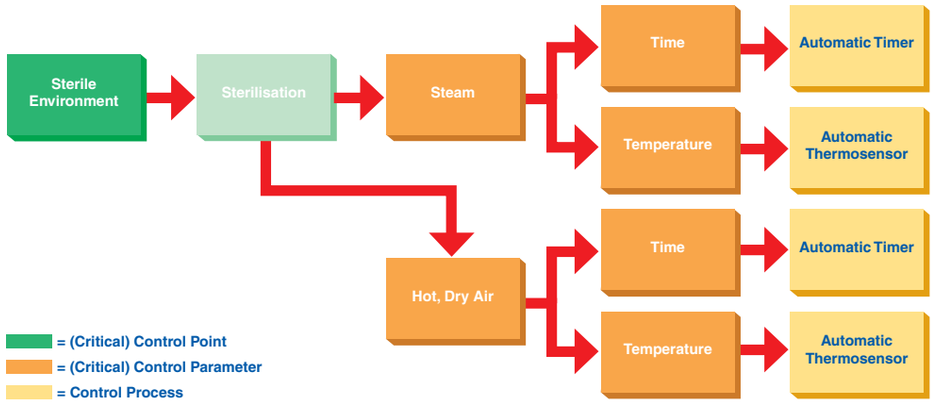


Figure 15. *Process Control: Sterilisation of the Aseptic Filler*

3.1.5 TBA/3: Maintaining Sterility: Process Control

As shown in figure 16, the proper control of the function of maintaining sterility during the production run is achieved either by instruments or by the construction of the equipment.

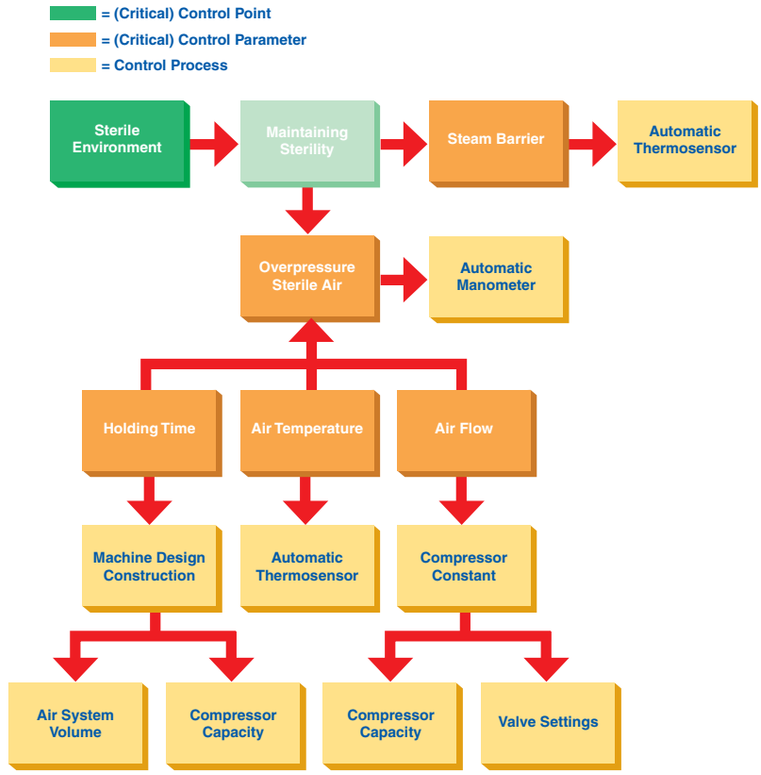


Figure 16. *Process Control: Maintaining Sterility During Production*

3.1.6 TBA/3: (Critical) Control Point: Package Integrity

The tightness of the package is an important factor in determining the microbiological safety of an aseptic packaging operation. At the present time, all methods available depend on the human factor (figure 17).

Consideration must be given to the tightness of the longitudinal and transversal seals as well as to possible damage in the structure of the packaging material itself.

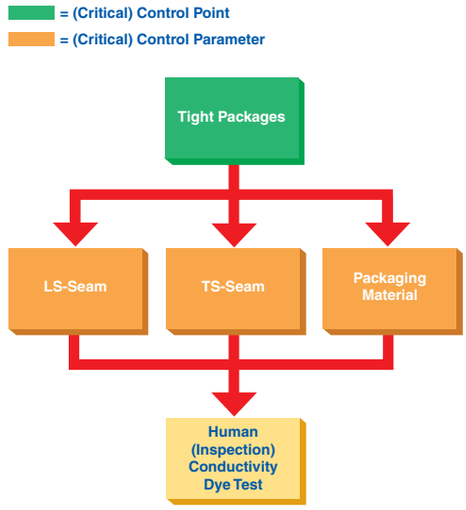


Figure 17. *Package Integrity*

3.2 Tetra Brik Aseptic 8 and 9 (TBA/8 and TBA/9)

Tetra Brik Aseptic 8 and 9 differ in a number of aspects from Tetra Brik Aseptic 3. Consequently, different flow charts result, although, the (critical) control points remain the same (figure 19).

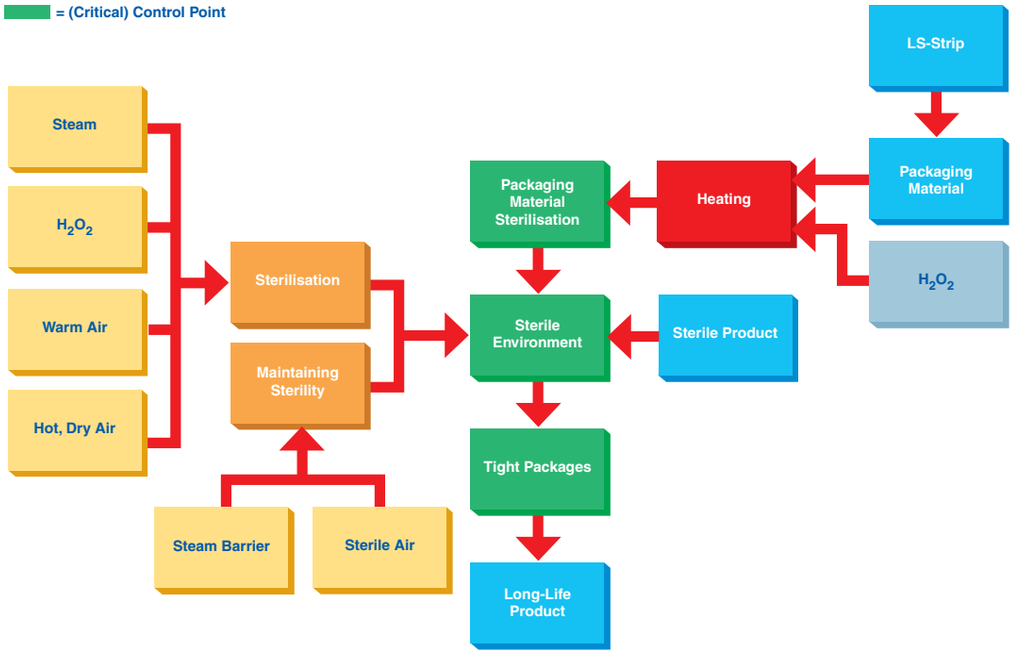


Figure 19. *Flow Chart for the TBA/8 and TBA/9 Aseptic Packaging Systems*

3.2.1 TBA/8 and TBA/9: (Critical) Control Points and (Critical) Control Parameters: Packaging Material Sterilisation

The sterilisation of the packaging material is a chemical process with five (critical) control parameters (figure 20).

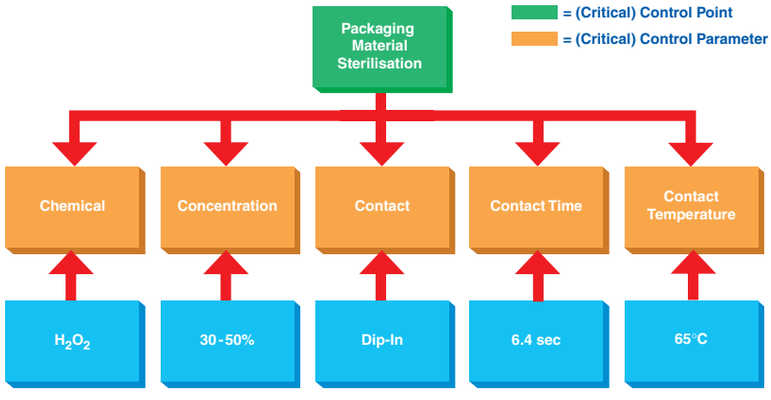


Figure 20. *Packaging Material Sterilisation: (Critical) Control Parameters*

3.2.2 Process Control: TBA/8 and TBA/9: Packaging Material Sterilisation

The control of the packaging material sterilisation process is outlined in figure 21. The (critical) control parameters are controlled and guarded either by instruments, or they are part of the construction of the equipment and thus constant.

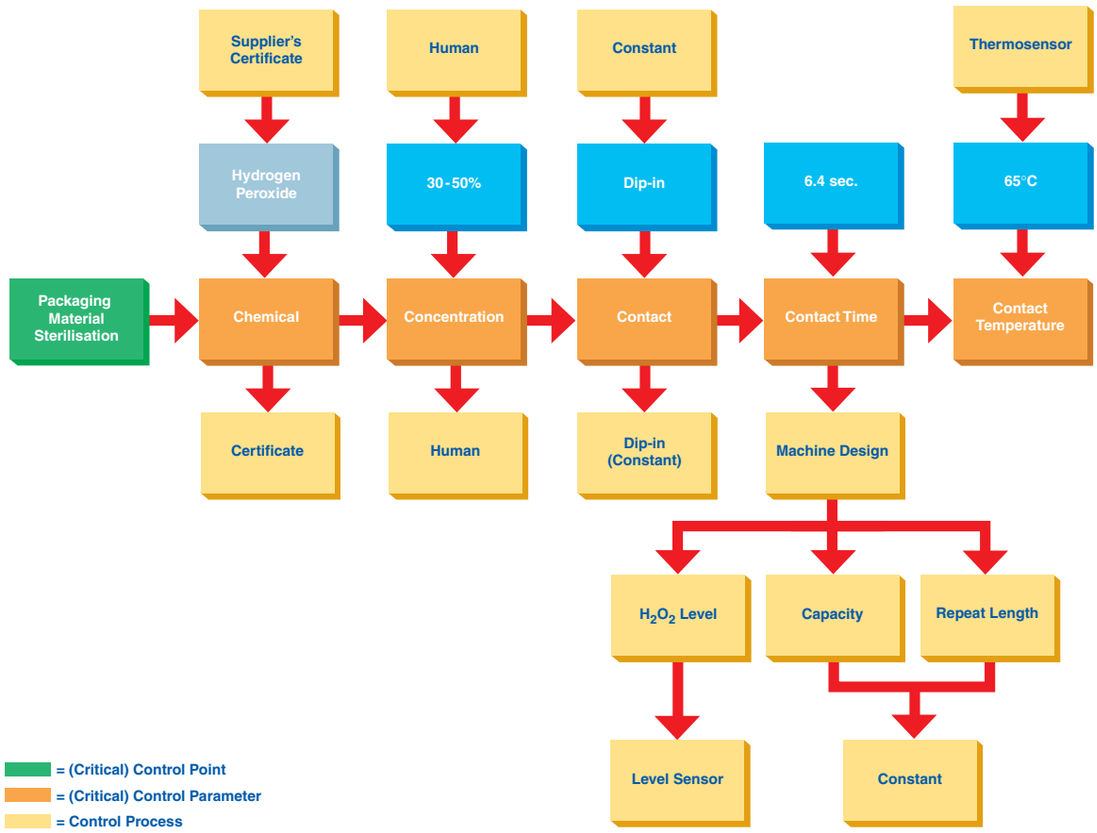


Figure 21. *Packaging Material Sterilisation, TBA/8 and TBA/9: Process Control*

3.2.3 TBA/8 and TBA/9: (Critical) Control Points and (Critical) Control Parameters: Sterile Environment

The area in which packages are formed, filled and sealed must be sterilised prior to production start-up, and sterility must be maintained during the entire production run. These process steps involve a number of (critical) control points and parameters which are shown in figure 22.

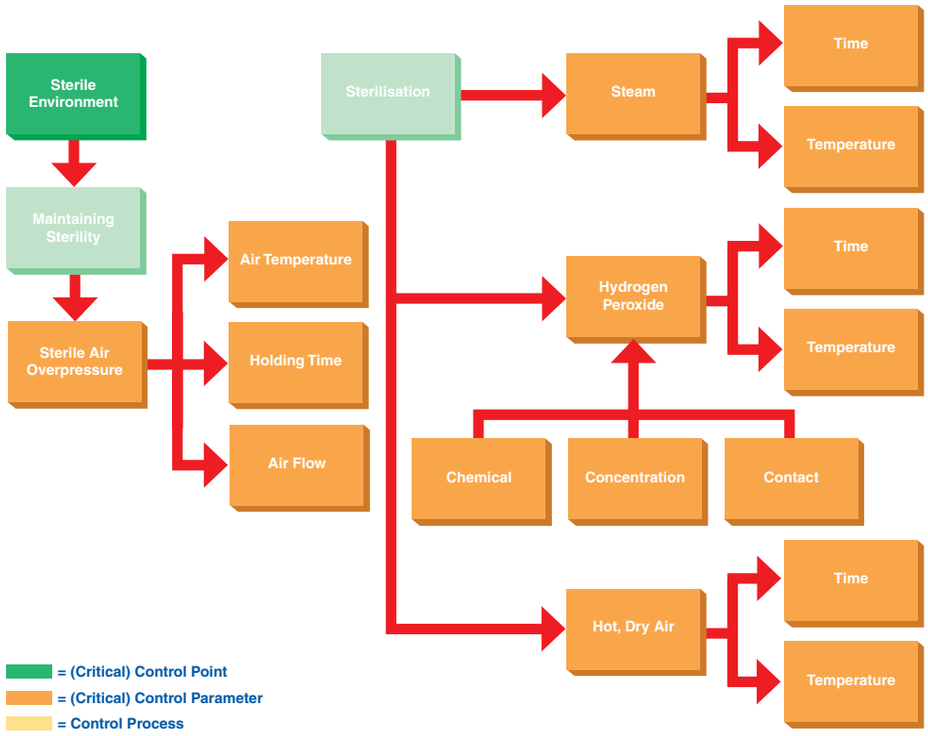


Figure 22. *Sterile Environment TBA/8 and TBA/9: (Critical) Control Points and (Critical) Control Parameters*

3.2.4 Process Control: TBA/8 and TBA/9: Sterile Environment, Sterilisation

The repeatability of the equipment sterilisation operation requires, in general, that all surfaces are free from residue and are clean. This is particularly true if the process of sterilisation is chemical in nature. Sterilisation of the area in which the packages are formed, filled and sealed is done by thermal and chemical processes. At this process stage, a number of (critical) control parameters (figure 23) are involved. Process control is also required in order to ensure that the equipment sterilisation process is repeatable.

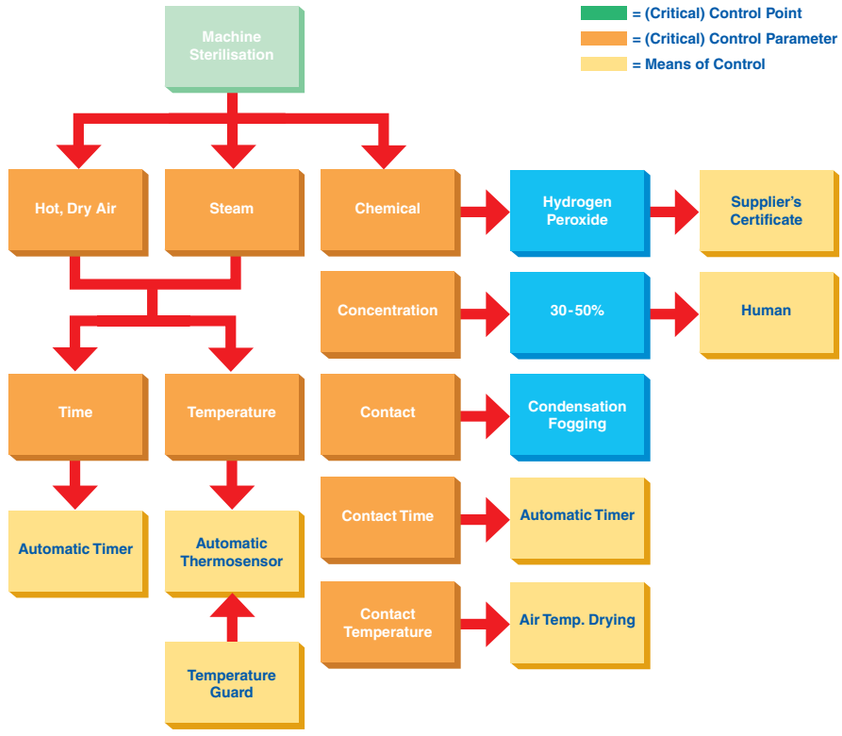


Figure 23. *TBA/8 and TBA/9: Control of Equipment Sterilisation*

3.2.5 Process Control: TBA/8 and TBA/9: Sterile Environment, Maintaining Sterility

During production, sterility has to be maintained in the area where the packages are formed, filled and sealed. The control of this function is essential for the safety and adequacy of the operation. Figure 24 shows the way in which these (critical) control parameters are controlled.

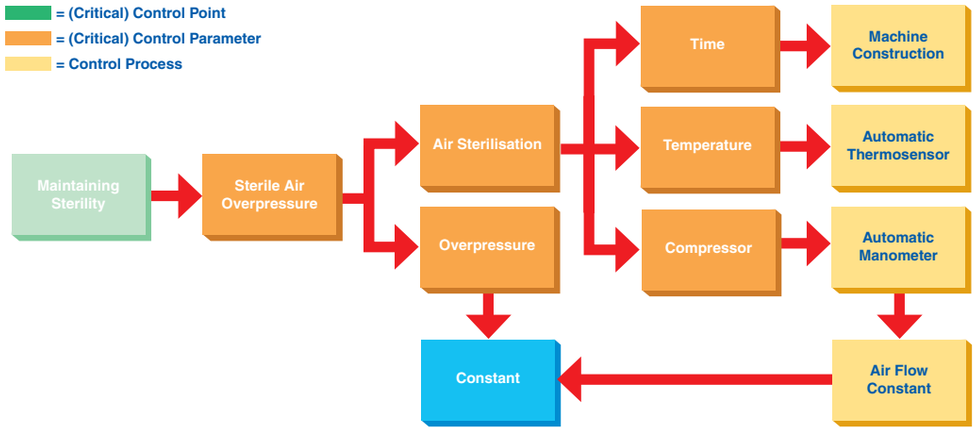


Figure 24. *TBA/8 and TBA/9: Maintaining Sterility During Production: Controlling the Process*

Example 3: A QACP Plan for Long-Life Chocolate Milk

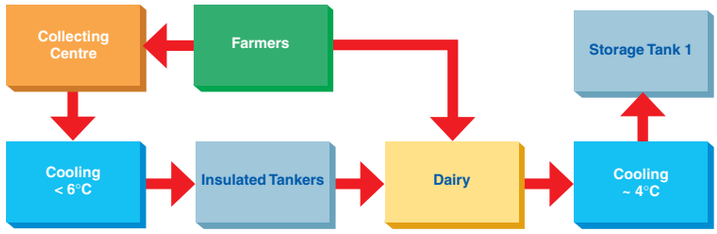
1. Raw Materials

The raw materials (ingredients) used in the manufacturing process for UHT chocolate milk are: reconstituted skimmed milk, fresh milk, cocoa powder, flavouring, sugar, stabiliser, emulsifier and water.

1.1 Quality Specifications

a) *Skimmed Milk Powder:* purchased from Australia according to ADMI specifications. Medium/low-heat powder.

b) *Fresh Milk:* total bacterial endospore count: <1000/ml; alcohol stability: >72.



The flow chart below illustrates the collecting system for untreated milk (figure 1).

Figure 1. *Flow Chart: Collecting System for Untreated Milk*

c) *Cocoa Powder:* special powder quality from Holland, specifically for UHT processing (187). No other quality specifications.

d) *Flavouring:* no specifications.

e) *Sugar:* no specifications.

f) *Stabiliser and Emulsifier:* no specifications.

g) *Water:* potable: 44°C, *Escherichia coli* <1 / 100 ml, total count: <100/ml.

2. Intermediate Product

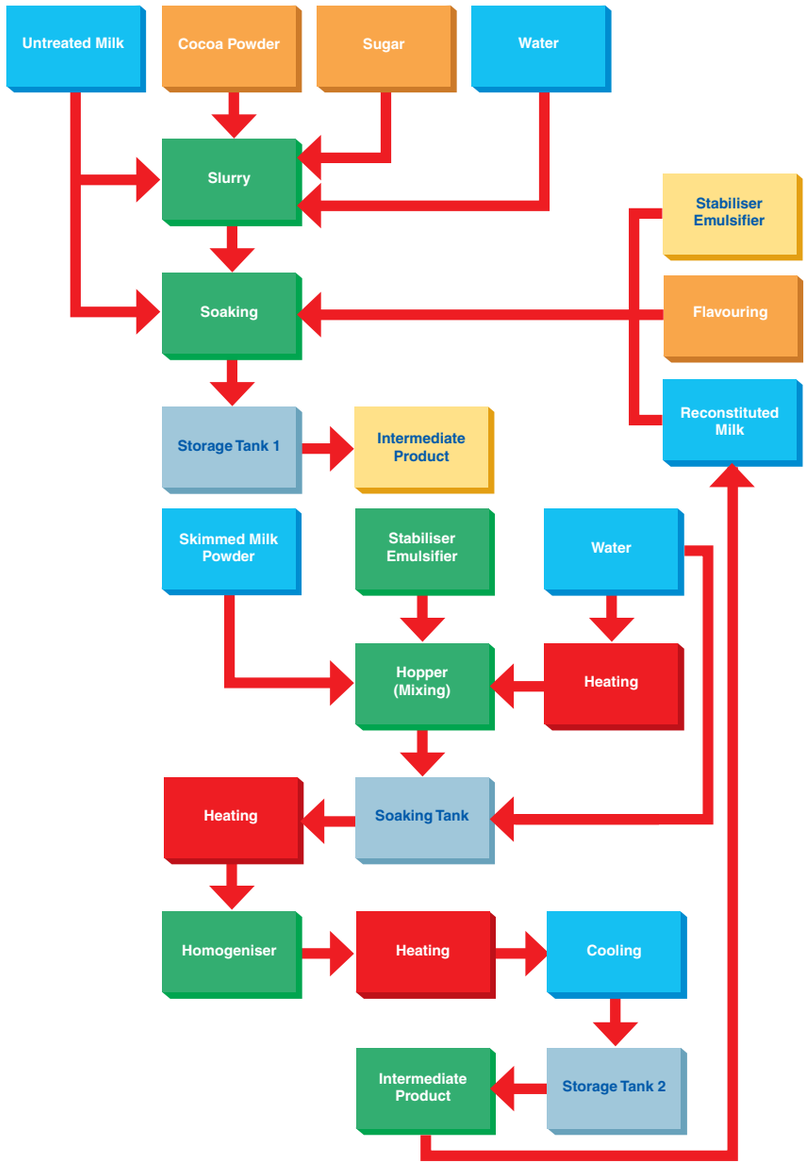


Figure 2. *Flow Chart: Chocolate Milk: Production of Intermediate Product*

The flow chart (figure 2) shows the preparation of the intermediate product. No quality specifications specific to long-life products exist for the intermediate product. Essential for the rapid heat penetration required for efficient UHT treatment is soaking in general and of cocoa powder in particular, the latter being a critical control point.

3. Final Product: Long-Life Chocolate Milk

3.1 Equipment

A Tetra Laval plate heat exchanger (3,500 litres/hour) directly connected to two TBA/9 250. Figure 3 is a flow chart showing the production of long-life chocolate milk.

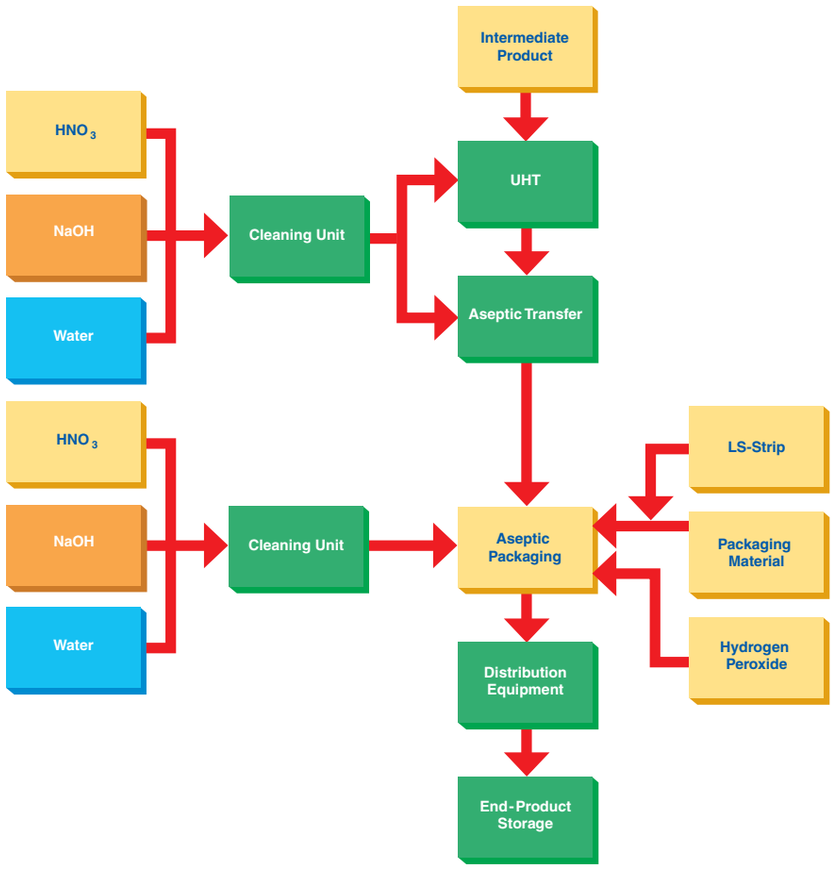


Figure 3. *Flow Chart: Final Processing and Packaging*

The packages are palletised manually and separately for each of the two TBA/9 fillers. Subsequently, the product is stored until released by the quality control department. The decision as to when to release is based on the results obtained from the incubated package control and other parameters.

4. The Hazard To Be Studied

4.1 Spoilage by Growth of Bacterial (Bacillus) Spore-Formers

4.1.1 Risk Assessment

This risk assessment covers the entire line from raw materials (ingredients, packaging materials, etc.,) to consumption. A relative rating of the importance of the hazard under study is obtained by multiplying the probability and the severity rating:

0.75 x 6 = 4.50
(Cost Estimate: 21 x 1,000 = 21,000)

a) *Probability Estimate:* how likely is it that the consumer will get chocolate milk spoiled by the growth of *Bacillus*? Considering the quality of the raw material, the processing and packaging conditions, and the circumstances prevailing during distribution, a probability rating of 0.75 (~ 21 times in five years) is a reasonable estimate.

b) *Severity Estimate:* microbiological spoilage is a comparatively severe event. However, spoilage by *Bacillus* does not imply a public health risk. A severity rating of 6 (average cost per incident: ~ 1,000 monetary units) appears reasonable.

4.1.2 Identification of Control Points

Two control points can be identified: A) process survivors and B) reinfection.

A) *Process survivors:* UHT treatment and sterilisation of the packaging material.

Risk Assessment: the risk of UHT process survivors is relatively low:

probability estimate: 0.10 (~ once every 5 years);

severity estimate: 6 (~ 1,000 monetary units);

Risk Assessment: 0.10 x 6 = 0.60
(Cost Estimate = 1 x 3,000 = 3,000).

Packaging material sterilisation:

probability estimate: 0.15 (~ 1.5 times every 5 years);

severity estimate: 6 (~ 1,000 monetary units);

Risk Assessment: 0.15 x 6 = 0.90
(Cost Estimate: 1.5 x 3,000 = 4,500).

B) *Reinfection:* leakage in the equipment at temperatures above 70°C, and insufficient sterilisation of the plant (UHT equipment, piping and the aseptic filling machine).

Risk Assessment: the risk of reinfection is rather large:

probability estimate: 0.50 (~ 5 times every 5 years);

severity estimate: 6 (~ 1,000 monetary units);

Risk Assessment: 0.50 x 6 = 3.00
(Cost Estimate: 4.5 x 3,000 = 13,500).

A.1 Process Survivors: UHT Treatment

1. UHT Treatment: The following control parameters are valid for UHT treatment: temperature and time.

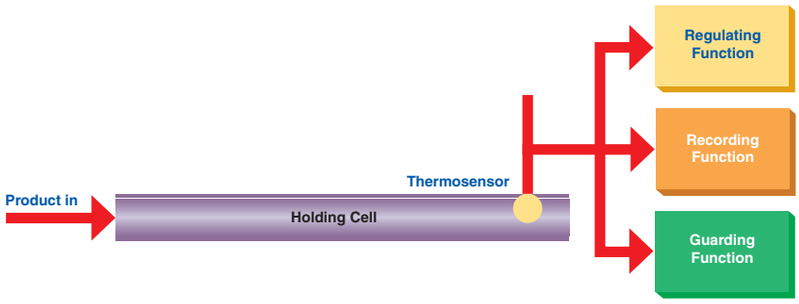


Figure 4. *Sterilisation Temperature Control, Recording and Guarding System*

From a safety point of view, the guarding function is essential.

a.) ***Temperature:*** the sterilisation temperature.

- **Critical Limit**: 135°C.
- **Target Value**: 140°C.
- **Preventive Measures**: calibration and challenging of the thermosensor every three months; daily comparison with a thermometer reading by the machine operator, a guarding function which shuts down the steriliser if the temperature drops below 135°C.
- **Corrective Action**:

  Process: replace the thermosensor.

  Product: requires the simultaneous breakdown of the thermosensor and guard function which is very unlikely. Depending on the temperature drop: tightened inspection (sampling) and/or re-processing (for a different product?).

b.) ***Time:*** refers to the holding time at the sterilisation temperature. The holding time is determined by the capacity of the steriliser and the volume of the holding cell.

b.1) Capacity of the steriliser: the throughput of the steriliser is the number of litres per hour passing through the homogeniser, a positive piston pump. The volume is constant.

- **Critical Limit**: not applicable.
- **Target Value**: not applicable.
- **Preventive Measures**: not applicable.
- **Corrective Action**: not applicable.

b.2) Volume of the holding cell: the volume of the holding cell is determined by its length which is constant, and the diameter. Deposit formation causes the diameter to become successively smaller, thus reducing the holding time (capacity constant) and giving a lower sterilising effect (86). The amount of deposit formed can be measured indirectly by either the pressure drop and/or the temperature differential.

b.2.1) Length of the holding cell:

- **Critical Limit**: not applicable.
- **Target Value**: not applicable.
- **Preventive Measures**: not applicable.
- **Corrective Action**: not applicable.

  Process: not applicable.

  Product: not applicable.

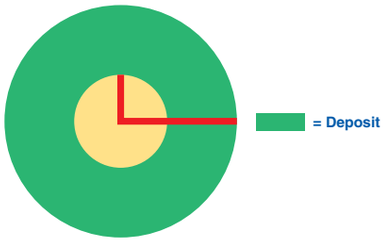


Figure 5. *Deposit Formation: Holding Cell*

b2.2) Diameter of the holding cell: as deposits form (figure 5), the pressure drop over the equipment and the temperature differential between the heating medium and product increase.

- **Critical Limit**: x kg/cm^2; y°C (pressure drop; temperature differential).
- **Target Value**: not applicable.
- **Preventive Measures**: cleaning of the equipment, "sterile" clean.
- **Corrective Action**:

  Process: manual shut-down and full cleaning of the steriliser.

  Product: inspections.

The precondition for proper sterilisation of the product is sufficient soaking, especially of the cocoa powder.

A.2 Process Survivors: Sterilisation of the Packaging Material

Sterilisation of the packaging material is a chemical process. Hydrogen peroxide is used as a sterilant and the packaging material is sterilised by passage through a dip-in bath (figure 6).

The chemical process has five control parameters: the chemical, the concentration of the chemical, contact between the chemical and the object (packaging material), contact time, and temperature during contact.

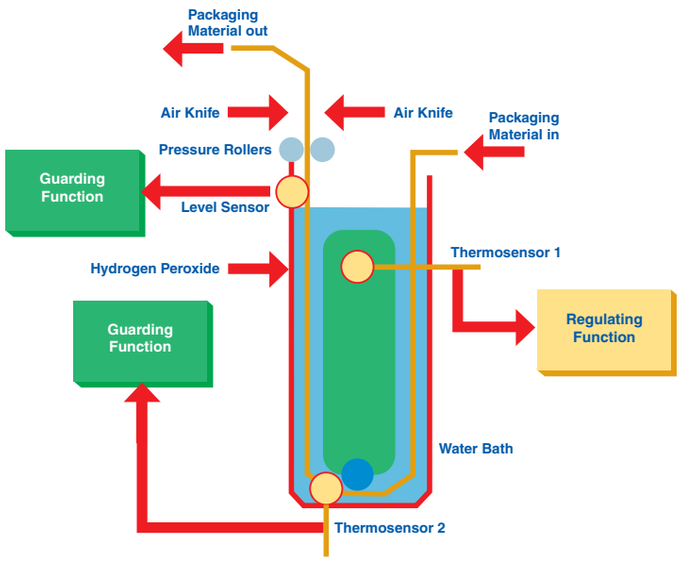


Figure 6. *Sterilisation of the Packaging Material*

a) ***The chemical:*** hydrogen peroxide, H_2O_2.

- **Critical Limit**: not applicable.
- **Target Value**: not applicable.
- **Preventive Measures**: purchase from a reliable source. Food-grade quality.
- **Corrective Action**:

  Process: not applicable.

  Product: not applicable.

b) The ***concentration*** of the chemical.

- **Critical Limit**: 30% (50%).
- **Target Value**: 35% (< 50%).
- **Preventive Measures**: regular (every 6 hours) determination of the concentration by the machine operator.
- **Corrective Action**:

  Process: restore correct concentration.

  Product: depending on the magnitude of deviation, additional packages are tested for sterility or the product is reworked immediately. The decision on which procedure to use is made by the quality control manager.

c) ***Contact*** between the chemical and the packaging material. It is accomplished by passage through the liquid hydrogen peroxide which is constant.

- **Critical Limit**: not applicable.
- **Target Value**: not applicable.
- **Preventive Measures**: not applicable.
- **Corrective Action**:

  Process: not applicable.

  Product: not applicable.

d) ***Contact time:*** contact time is determined by the capacity (packages/hour), the repeat length (the length of packaging material needed for the production of one package), both of which are constant, and the level of hydrogen peroxide in the H_2O_2 bath which is guarded by a level sensor.

- **Critical Limit**: 6.4 seconds.
- **Target Value**: 7.2 seconds.
- **Preventive Measures**: challenging the level sensor every three months.
- **Corrective Action**:

  Process: replace the level sensor.

  Product: re-processing or use in another product.

e) ***Temperature during contact:*** the temperature of the hydrogen peroxide is regulated via a water bath by thermosensor 1. Thermosensor 2 has a guarding function only, stopping the filling operation if the temperature drops below the critical limit.

- **Critical Limit**: 65°C.
- **Target Value**: 70°C.
- **Preventive Measures**: guarding function of thermosensor 2: calibration and challenging every three months.
- **Corrective Action**:

  Process: replace thermosensor 2.

  Product: tightened inspection, re-processing or use in another product.

B. Reinfection

Two sources of reinfection are considered: leakage of the system at temperatures >70°C, and insufficient sterilisation of the equipment.

B.1 Leakage at temperatures >70°C

The only area of interest is the first (regenerative) cooling section of the steriliser (figure 7).

- **Critical Limit**: pinholes or cracks in the first cooling section.
- **Target Value**: not applicable.
- **Preventive Measures**: inspection by opening the heat exchanger every month.
- **Corrective Action**:
- *Process:* replace the plate that has pinholes or cracks.
- *Product:* none.

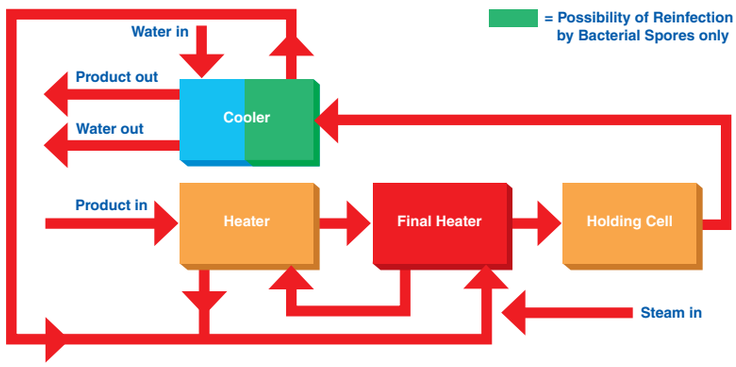


Figure 7. *Reinfection: First (Regenerative) Cooling Section*

B.2 Insufficient Sterilisation

Insufficient sterilisation can be caused by a failure in the heat or chemical sterilisation process. The pre-condition for obtaining a satisfactory result regularly in the plant sterilisation process is to ensure adequate cleanliness of the equipment. This is particularly true for the chemical sterilisation operation (TBA/9).

B.2.1 Failure of the Heat Sterilisation Process

The steriliser and the product line are sterilised by superheated water. Figure 8 shows the control procedure. The critical parameters of the process are temperature and time.

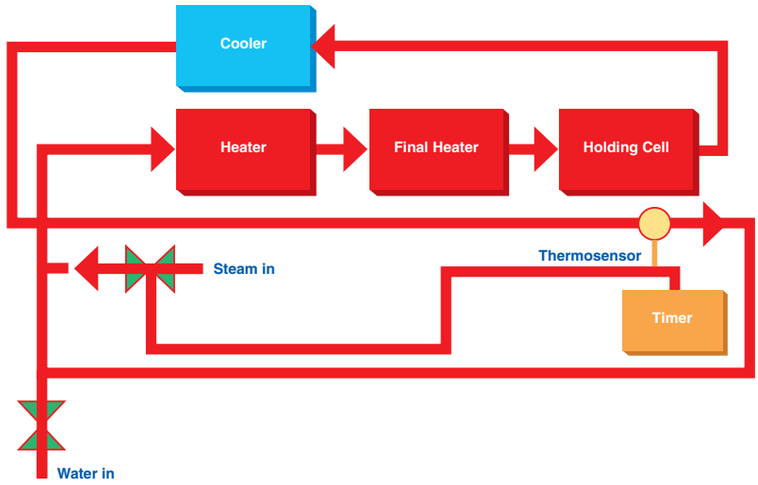


Figure 8. *Control of Plant Sterilisation*

a) ***Temperature:*** the sterilisation temperature is controlled, recorded and guarded by a thermosensor which activates a steam valve and a timer.

- **Critical Limit**: 135°C.
- **Target Value**: 140°C.
- **Preventive Measures**: guarding function of the thermosensor. A drop in temperature below 135°C results in a repetition of the sterilisation cycle. Calibration and challenging of the thermosensor every three months.
- **Corrective Action**:

  Process: replace the thermosensor.

  Product: not applicable.

b) ***Time:*** the plant sterilisation time at a minimum temperature of 135°C is controlled by a timer which, in turn, is activated by the thermosensor (figure 8). The time is recorded on a process chart.

- **Critical Limit**: 25 minutes.
- **Target Value**: 30 minutes.
- **Preventive Measures**: none.
- **Corrective Action**:

  Process: none.

  Product: not applicable.

B.2.2 Failure of the Sterilisation Process in the TBA/9

Part of the TBA/9 filler is sterilised by steam (the product valve area), part by hot air (the piping between the air incinerator and the H_2O_2 evaporator) and part by hydrogen peroxide (the rest of the filler).

B.2.2.1 Steam sterilisation of the product valve

A time-temperature process, the control of which is shown in figure 9.

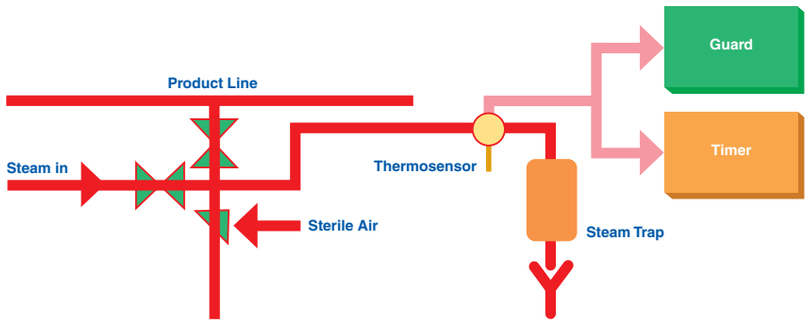


Figure 9. *Steam Sterilisation of the Product Valve*

Temperature:

- **Critical Limit**: 125°C.
- **Target Value**: 130°C.
- **Preventive Measures**: calibration and challenging every three months.
- **Corrective Action**:

  Process: replace the thermosensor.

  Product: not applicable.

Time:

- **Critical Limit**: 25 minutes.
- **Target Value**: 30 minutes.
- **Preventive Measures**: none.
- **Corrective Action**:

  Process: none.

B.2.2.2 Sterilisation by hot, dry air:

The piping between the incinerator and the evaporator (figure 10).

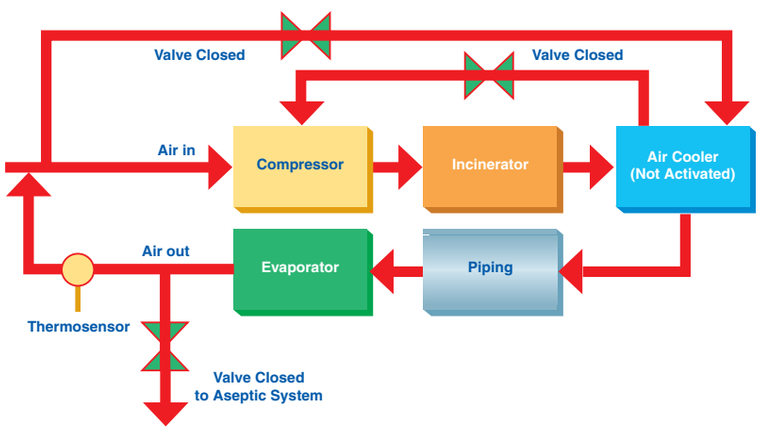


Figure 10. *Sterilisation By Hot, Dry Air*

A time-temperature process which has temperature and time as control parameters.

Temperature:

- **Critical Limit**: 270°C.
- **Target Value**: 280°C.
- **Preventive Measures**: the guarding function of the thermosensor re-starts the sterilisation cycle; calibration and challenging of the thermosensor every 3 months.
- **Corrective Action**:

  Process: replace thermosensor.

  Product: not applicable.

Time: If the critical limit temperature is reached under normal conditions of sterilisation, the temperature guard activates a timer (which is re-set if the temperature drops below the critical limit).

- **Critical Limit**: 20 minutes.
- **Target Value**: 30 minutes.
- **Preventive Measures**: none.
- **Corrective Action**:

  Process: replace the timer.

  Product: not applicable.

B.2.2.3 Sterilisation by hydrogen peroxide

The rest of the TBA/9 is sterilised by H_2O_2, a chemical process. The system of sterilisation is illustrated in figure 11. A certain amount of H_2O_2 is discharged into an evaporator. The hydrogen peroxide is evaporated and distributed by hot, sterile air over all the surfaces to be sterilised. The hydrogen peroxide condenses on the cooler surfaces of the equipment. Subsequently, the hydrogen peroxide is dried off by hot, sterile air. Five (critical) control parameters have to be considered: the chemical, the concentration of the chemical, the contact between the chemical and the object, the contact time, and the temperature during contact.

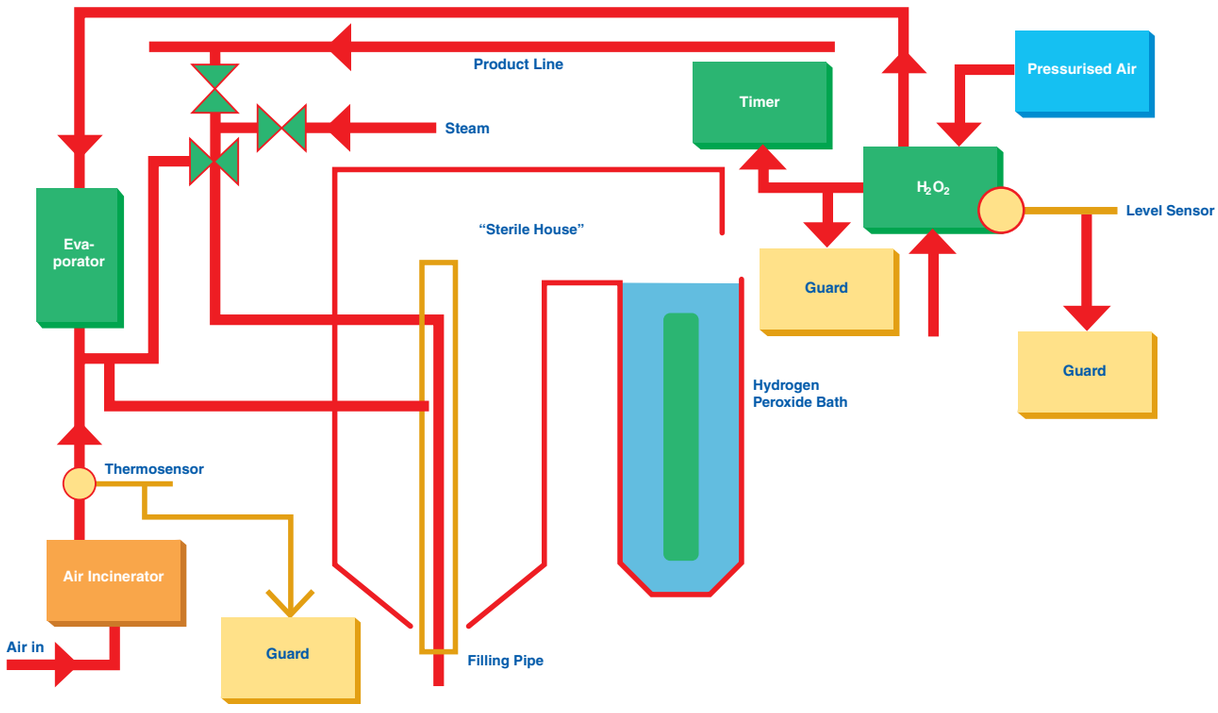


Figure 11. *Hydrogen Peroxide Sterilisation*

I The chemical

- **Critical Limit**: hydrogen peroxide.
- **Target Value**: hydrogen peroxide.
- **Preventive Measures**: purchase from a reliable source, contact with the supplier, supplier's certificate.
- **Corrective Action**:

  Process: replace chemical.

  Product: not applicable.

Contact between the chemical and the object: contact is determined by the volume of hydrogen peroxide discharged into the system, its proper evaporation and the correct functioning of the spray nozzle.

II Volume

- **Critical Limit:** 125 ml.
- **Target Value:** 125 ml.
- **Preventive Measures:** a level sensor connected to a guard automatically repeats the hydrogen peroxide filling process in the spray container. If, on repetition, the required level is not reached, the filler shuts down. The guarding function is challenged every 3 months.
- **Corrective Action:**

  Process: replace the level sensor, clean the piping supplying the hydrogen peroxide to the spray container.

  Product: not applicable.

III Evaporation: evaporation requires a certain minimum temperature that is provided by the flow of hot sterile air.

- **Critical Limit:** 280°C.
- **Target Value:** 360°C.
- **Preventive Measures:** the guarding function of a thermosensor repeats the process if the temperature drops below the critical limit. Regular challenging of the thermosensor every 3 months.
- **Corrective Action:**

  Process: replace the thermosensor.

  Product: not applicable.

IV Spray nozzle function: this function is controlled by a timer which measures the time required for emptying the spray container: if the spray nozzle is clogged, the time increases; if it falls off, the time required is less.

- **Critical Limit:** x seconds, y seconds.
- **Target Value:** not applicable.
- **Preventive Measures:** the guarding function of the timer repeats the spray process and shuts down the filler if it fails recurringly. Regular challenging of the guarding function every 3 months.
- **Corrective Action:**

  Process: repair or replace the spray nozzle.

  Product: not applicable.

V Contact time: the time needed for drying the condensed hydrogen peroxide

- **Critical Limit:** 10 minutes.
- **Target Value:** not applicable.
- **Preventive Measures:** none.
- **Corrective Action:**

  Process: none.

  Product: not applicable.

VI Temperature during contact: the temperature is provided by the air circulating in the system and drying the hydrogen peroxide. Control is achieved by the incineration temperature.

- **Critical Limit:** 330°C.
- **Target Value:** 360°C.
- **Preventive Measures:** the guarding function of the thermosensor repeats the process if the temperature drops below the critical limit. Regular challenging of the thermosensor every 3 months.
- **Corrective Action:**

  Process: replace the thermosensor.

  Product: not applicable.

Example 4: A Guide to the Introduction of Quality Assurance Systems in the Food Industry Based on the ISO 9000 Standard

1. Introduction

ISO 9000

The purpose of this section is to provide a guide for the food industry in general and the dairy sector in particular for the introduction of a quality system as described in the ISO 9000 standard. In so doing, the frame of application has been narrowed considerably. Nevertheless, each food-producing enterprise will have to adjust the quality system to its own specific conditions, products and needs.

The introduction of a quality system can be done in many different ways. An attempt has been made below to outline just one approach.

Generally, two major difficulties are connected with the implementation of the ISO 9000 standards, both of which are implicit in the concept of the standard itself:

Two Problems Involved

1. of necessity, the standard has to be phrased in a very general way since it is intended to be applicable to *all* industries; and
2. as a result of which, the language becomes rather difficult to understand and definitions have to be very general.

Based on the above, an attempt has been made to streamline the definitions of some terms to the requirements of the food industry. ISO 9000 has provision for such procedures *if* these terms are properly defined.

A further problem generally encountered is the change in philosophy which the introduction of a quality assurance system entails. Traditionally, the food industry in general and the dairy sector in particular have been implementing quality control systems for some time. The necessary change in quality assurance requires a change in thinking. It is hoped that this guideline may help make the transition easier.

2. Quality

Definition of Quality

Commodity quality has been defined in many ways. Different views certainly do exist on the subject. ISO 8402 defines quality as the "totality of characteristics of an entity that bear on its ability to satisfy stated and implied needs".

The ISO 9000 standard recommends the use of terms as defined in ISO 8402. However, deviations are permitted, i.e., other definitions may be used. If these are made the definition of each respective term must be given in the documentation. For the food industry in general and the dairy industry in particular, I would suggest using the following definition:

"the quality of a commodity is an expression of that commodity's ability to fulfil consumers' (users') expectations and comply with producers' promises" (Figure 1).

Figure 1: *Definition of Quality*

The above definition is not in contradiction with the one given in ISO 8402. However, it underlines the fact that quality is more than the legal requirements (government regulations and/or norms) that define products, which is part –but only part– of the "producers' promises".

2.1 Comparison between Quality Control (QC) and Quality Assurance (QA)

Quality Control
Quality Assurance

ISO 8402 defines quality control as "operational techniques and activities that are used to fulfil requirements for quality", while quality assurance is defined as "all the planned and systematic activities implemented within the quality system and demonstrated as needed, to provide adequate confidence that an entity will fulfil requirements for quality". For the food industry in general and the dairy sector in particular, the use of the following definitions is suggested:

1) *Quality Control* is those activities that ensure that the materials and ingredients used in the formulation, and/or that are needed in the production of products, as well as all the intermediate and end products, are within their respective (quality) specifications;
2) *Quality Assurance* is those activities that prevent intermediate and/or end products from deviating from their respective (quality) specifications.

Although these definitions are not really identical to those given in ISO 8402, they do not contradict them, are easier to understand and thus easier to implement in the food industry. Emphasis is placed on the necessity of quality specifications. Deviations can only be determined if such specifications exist.

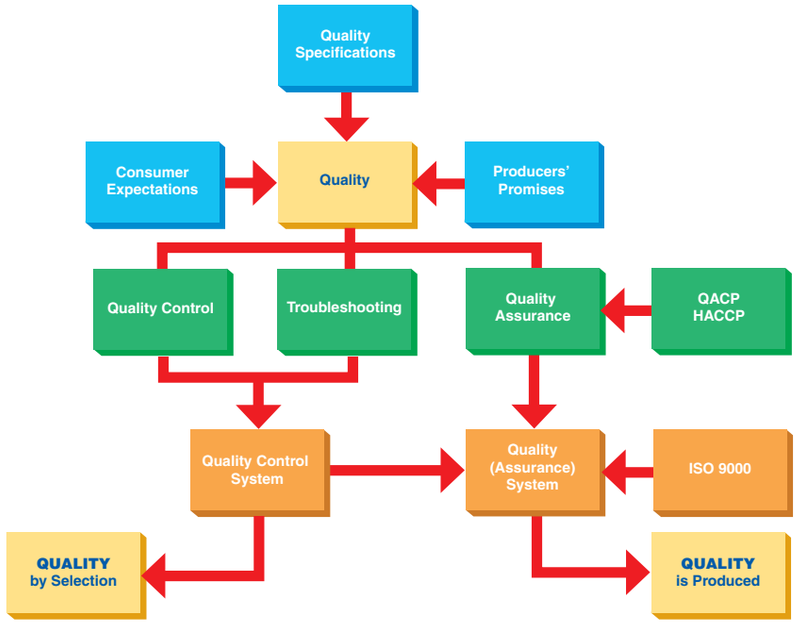


Figure 2: *Interrelationship between Quality, Quality Control and Quality Assurance.*

Consistent Quality

Confidence in company management (internal) and in product reliability (external) results from the consistent production of conforming products. Figure 2 shows the resulting relationship between product quality, quality control and quality assurance.

2.2 Quality (Assurance) System (QAS) and Quality Standards

ISO 9000 is a flexible system to suit varying needs. Each food-producing enterprise will determine its own quality policy and standards.

Quality control and quality assurance activities require specifications. ISO 8402 defines specifications as "documents stating requirements". "A qualifier should be used to indicate the type of specification such as 'product specification', 'test specification', 'quality specification'".

It is also necessary to distinguish clearly between the definition of a product and its quality specifications.

Definitions Specifications

1) *Definition:* the definition of food products is often given in the regulations. Legal requirements (standards, norms) state the *basic* demands which have to be made on a product. If these are disregarded, the product is simply something else.
2) *Quality specification:* a quality specification should go beyond the legal limits defined and contain more than the minimum requirements.

The term "quality" should be looked upon in a very wide sense. Quality improvement relates not only to the sensory, nutritional and biological characteristics of a food product, but also to economic factors and the internal and external environment under which such products are produced.

Quality: More Than Flavour!

2.3 Why Introduce a Quality (Assurance) System?

The European Union (EU) has issued a directive requiring the mandatory introduction of the HACCP concept in the food industry. Some countries outside the EU have already done so for some branches of the food industry.

Regulations on "Good Manufacturing Practices" (GMPs) have been issued in some countries for special food products, among them shelf-stable, low-acid foods.

However, at the present time it appears very unlikely that any country will make *legal* demands making certification and the introduction of ISO 9000 mandatory. Despite this, the food industry, and the dairy industry especially, should be keen to implement the ISO 9000 concept. Competitive strength can only be achieved and maintained if products with a sufficiently high *and* consistent level of quality are produced regularly.

The EU has issued a recommendation that preference be given to suppliers certified according to ISO 9000. More and more companies will require that their national and international suppliers be certified according to ISO 9000. Aside of this, the following advantages can be seen as a consequence of the introduction of a quality system:

- potential benefits for the dairy (marketing advantages, reduced costs in the form of less wastage and lower energy consumption, simplified work routines, continuous quality improvements, etc.,);
- possible risks: quality hazards before and after the dairy, (limited commercial advantages in the absence of maintenance and the lack of development in quality policies, objectives and targets to meet changing market demands);
- conclusions: clear policy, sufficient resources, step-wise introduction of the system and long-term market orientation are required to gain competitive strength from a quality assurance system;
- one of the major reasons for the introduction of any quality system is the inadequacy of quality control procedures. They are often ineffective and always "after the event", i.e., not preventive!

2.4 Major Features of Quality (Assurance) Systems (QAS)

2.4.1 The ISO 9000 System

The IS0 series contains a number of different standards which describe in general terms the basic requirements for establishing and maintaining a quality system:

The ISO 9000 Standard

1) ISO 9000 - 1: Quality management and quality assurance standards - Part 1: Guidelines for selection and use;
2) ISO 9000 - 3: Quality management and quality assurance standards - Part 3: Guidelines for the application of ISO 9001 to the development, supply and maintenance of software;
3) ISO 9001: Quality systems - Model for quality assurance in design, development, production, installation and servicing;
4) ISO 9002: Quality systems - Model for quality assurance in production, installation and servicing;
5) ISO 9003: Quality systems - Model for quality assurance in final inspections and tests;

6) ISO 9004 - 1: Quality management and quality system elements - Part 1: Guidelines;
7) ISO 9004 - 2: Quality management and quality system elements - Part 2: Guidelines for services;
8) ISO 9004 - 4: Quality management and quality system elements - Part 4: Guidelines for quality improvement.

2.4.2 Good Manufacturing Practices (GMPs)

Good Manufacturing Practices

"Good Manufacturing Practices" (GMPs) are guidelines for the production of safe food products. In some countries, GMPs are mandatory for certain food products or categories. Since quality systems have a broader scope, the frame of the ISO 9000 standard is much wider than the requirements of GMPs.

2.4.3 Hazard Analysis Critical Control Point (HACCP)

HACCP, QACP Concepts

The concepts of "Hazard Analysis Critical Control Point" (HACCP) is very specific. One product, one production line and one hazard are addressed at a time. The documentation has to be prepared accordingly. The concept was introduced to ensure safe food products for astronauts beyond the level attainable at that time with traditional control procedures. Since the main objective of HACCP is food safety, it is now mandatory in a number of countries.

HACCP is the most powerful internationally recognised *tool* of quality assurance available today. Implementation of the concept requires full commitment of the senior management and the implementation of each of the following steps:

1) identification of the production line (flow chart);
2) identification of the product and its *intended* use;
3) identification of the hazard to be studied;
4) risk assessment: probability and severity estimate;
5) identification of critical control points; a new risk assessment is recommended;
6) identification of critical control parameters; a new risk assessment could be performed;
7) for each critical control point or parameter establish:
   - the critical limit;
   - the target value;
   - preventive measures;
   - corrective actions;
8) verification procedures;
9) validation procedures; and
10) auditing procedures.

Since HACCP is a powerful tool of quality assurance, a company applying for ISO 9000 certification should show a clear, written commitment to its introduction and incorporation into its quality system. However, the documentation is quite different from the one required by ISO 9000 and in the beginning subject to very frequent changes. Therefore, the documentation for HACCP should be kept separate from the one for ISO 9000 (Figure 3).

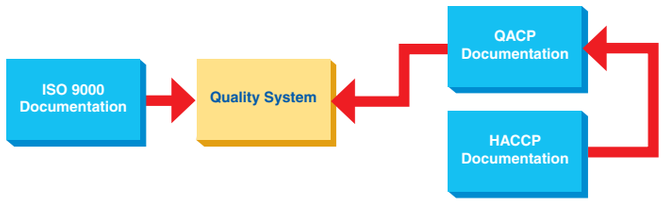


Figure 3: *ISO 9000, QACP and HACCP Documentation*

2.4.4 Quality Assurance Control Point (QACP)

QACP

Quality Assurance Control Point (QACP) is *not* an internationally accepted and recognised term. Although methods and procedures are exactly the same, it is suggested that HACCP and QACP be distinguished because:

1) the area of concern for HACCP is public health aspects and food safety;
2) many product characteristics are important for food quality but they are not of significance for public health; their handling and documentation should be kept separate;
3) the concept is a very powerful tool of quality assurance.

3. The ISO 9000 Standard

Quality Manual
Manual of Procedures
All Other Quality Documents

Basically, and according to the "quality system hierarchy" (ISO 10 013: Guidelines for developing quality manuals), ISO 9000 requires three types of documentation (Figure 4): a quality manual needs to be prepared; the "Quality System Procedures" must be documented; and all other "Quality Documents" must be addressed. The documentation structure shown in figure 4 is a recommendation. All the issues addressed in the figure must be documented, but the arrangement is not optimal for all the various branches of the industry.

Department Manuals?

A quality manual must be prepared for all operations. Factories are organised and operate according to departments. As indicated in figures 4 and 5, a separate manual for procedures ("Manual of Procedures") can be prepared. The documentation of procedures together with instructions and all other relevant information can be placed in "Department Manuals" as illustrated in figure 5. In any event, it is important that the documentation is arranged in such a way that it facilitates the needs of the dairy *and* provides the basis for as easy an implementation as possible.

ISO 9000
HACCP
QACP

The concepts of HACCP and QACP should be incorporated into the ISO 9000 documentation as much as possible, even if the actual HACCP and QACP documentation is done separately.

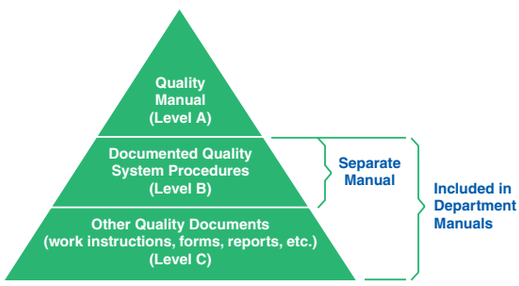


Figure 4: *Typical Quality System Hierarchy*

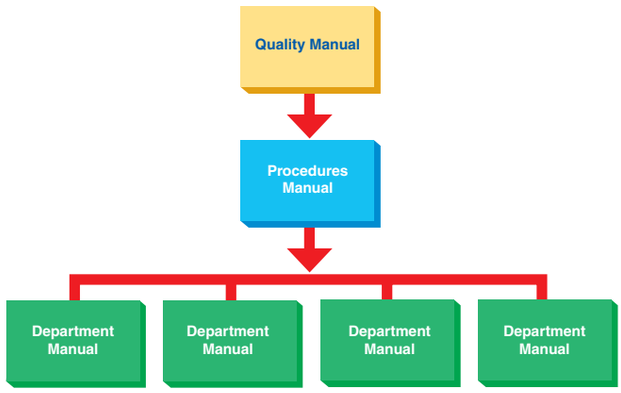


Figure 5: *Documentation Structure*

3.1 Guidelines

Available Guidelines

The ISO 9000 family of standards also provides some guidelines as to how a quality system can be documented. The relevant documents are:

1) ISO 9004 - 1: Quality management and quality system elements - Part 1: Guidelines;
2) ISO 9004 - 2: Quality management and quality system elements - Part 2: Guidelines for services;
3) ISO 10 013: Guidelines for developing quality manuals.

4. Proposed Work Plan for Introducing the ISO 9000 System

4.1 General

Change in Quality Philosophy Necessary?

The implementation of a quality system as described in the ISO 9000 standard is a major decision for any company. From the beginning, it should be very clear that ISO 9000 may require a change in philosophy; it is not *merely* certification. It is a continuous process requiring continuous effort!

Figure 6: *ISO 9000 A Quality System*

With this background and with as great an understanding of the consequences involved as possible, senior management is required to make some basic decisions:

Senior Management Decisions!

a) Do we want to commit our company (dairy) to the implementation of the ISO 9000 standard?
b) Are we, the senior management, prepared to accept our role in the concept?
c) Which of the standards (ISO 9001, 9002 or 9003) is applicable to our operations?

It is important to realise that the introduction of a quality system is only possible if the senior management accepts its role and responsibility within the framework of a quality system.

When starting the actual work on the documentation necessary for ISO 9000 certification, two major problems may, and usually are, encountered:

1) the amount of work is extremely large and frightening; the documentation is started but never finished: "And thus the native hue of resolution / is sicklied o'er with the pale cast of thought" (Hamlet, 3.1);
2) the staff working on the documentation do not sufficiently understand the benefits in it for the dairy; it is believed that the documentation is being done for the sake of the certifying authority.

The Start is Frightening!

4.2 Preparation for Certification

The certifying authority that executes ISO 9000 certification must be identified and contacted. The authority does not have to be located in the same country; an enterprise can require certification from any local or foreign institution which has been authorised to do so.

Certifying Authority

4.2.1 The Amount of Work

The documentation required is voluminous! In order to avoid the "frightening" effect, the work should be started on a very narrow basis and *not* on all aspects at the same time. It has been suggested that work commence with the quality manual and one department. In the dairy industry, either the production department or the quality control department is usually the most suitable one to start with.

Start with the Quality Manual and One Department Manual

4.2.2 Understanding the Purpose

It is the task of senior management to ensure that the staff fully understand why it is necessary to prepare the documentation. Two main reasons should be stressed:

a) the necessity of producing products of uniform quality; and
b) the need for quality improvement.

Quality: Improvement and Uniformity

Quality improvement can be looked upon as a three-stage process (figure 7):

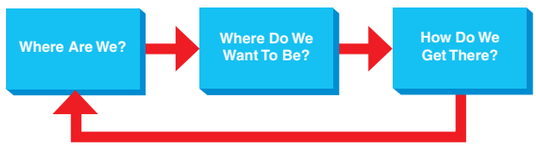


Figure 7: *Quality Improvement Loop*

A quality goal can only be clearly defined if the present level of quality is known! For this reason the question "Where are we?" needs to be answered. Documentation is needed to establish the basis for both the specification of the quality goal *and* the way to achieve it. The better the documentation, the more clearly and precisely the goals and means can be defined.

Definition of the Quality Goal

Figure 7: *Quality: Uniformity and Level*

4.3 Establishment of a Quality Team

Having reached the decision to proceed, a team needs to be formed. The task of the team is to:

ISO 9000 is Teamwork!

1) learn as much as possible about the ISO 9000 standards; to this end participation in a training course can therefore be very helpful;
2) start the documentation work necessary for certification;
3) transfer the accumulated knowledge of the team to *all* other departments in the organisation, and, since a quality system requires the active participation of every employee, to every member of staff;
4) follow the definitions laid down in ISO 8402. Any deviation from these must be listed in the documentation. This necessitates studying the definitions carefully and checking that they are being complied with constantly. Unfortunately, ISO 8402 is not very "reader-friendly". Therefore, at an early stage of the work, it is strongly recommended that the definitions given in ISO 8402 be arranged in alphabetical order!
5) function as teachers and mediators for the staff of the dairy.

Senior management has to provide the means and support needed by the team in order for it to fulfil its obligations.

4.4 Quality Manual

Quality Manual

The term quality manual is defined in ISO 8402 as follows: the quality manual is a "document stating the quality policy and describing the quality system of an organisation". It is also stated that: "a quality manual can vary in depth and format to suit the needs of an organisation". ISO 10 013 provides "Guidelines for developing quality manuals".

ISO 9000	ISO 10 013
1. Scope	a) Title, Scope and Field of Application
2. Normative Reference	b) Table of Contents
3. Definitions	c) Introductory Pages on the Organisation and the Manual
4. Quality System Requirements	d) The Quality Policy and Objectives of the Organisation
	e) The Organisational Structure
	f) The Elements of the Quality System

Table 1: *Layout of the Quality Manual: ISO 9000 or ISO 10 013?*

In the layout of the quality manual, the recommendation of ISO 10 013 should be followed.

The quality manual should be relatively short, no more than 30 to 50 pages. It should always be kept in mind that the quality manual is a public document: it cannot be handled on a confidential basis. Consequently, all confidential material should be excluded and documented elsewhere. Reference often has to be made to such information. In order to facilitate cross-referencing, a coding system should be developed at an early stage in the preparation of the documentation, a description of which needs to be included in the manual.

Necessity of a Coding System

4.4.1 Title, Scope and Field of Application

"The title and scope of the quality manual should clearly define the organisation to which the manual applies". ISO 8402 defines an organisation as a "company, corporation, firm, enterprise or institution or part thereof, whether incorporated or not, public or private, that has its own functions and administration".

Identify the Company

In the dairy industry, milk is often delivered by way of collecting stations. These may cause a problem in ISO 9000 certification, not because of the principle but because of the number of stations that might be involved. Milk collection stations and/or centres are often owned by the dairy. They are also the first point in the chain of milk-handling where a selection can be made. Whenever their number is within the limits of inclusion, then they should be included. If, however, the number is too large, an agreement should be reached with the certifying authority at a very early stage.

Collecting Stations

How should the documentation be handled in order to facilitate a volume which can be handled easily? Sometimes it is better to start the documentation procedure with the reception area of the dairy, excluding the collecting stations. Otherwise, the volume of documentation may become too extensive.

4.4.2 Table of Contents

A list of the contents of the quality manual.

4.4.3 Introductory Pages on the Organisation and the Manual

The ISO 8402 definition of "organisation" should be kept in mind (see above, 4.4.1). This section should contain a short description of the company, its structure, premises and location.

The Company, its Location and the Layout of the Manual

A general description of the layout of the manual should be included.

Information about the manual should contain any general information relating to the handling of the product, and the administration and authorisation of personnel. The coding system should also be described at this stage.

4.4.4 The Quality Policy and Objectives of the Organisation

The central part and basis of any quality system is the company's quality policy. It is the responsibility of senior management to formulate the company's quality policy. ISO 8402 defines quality policy as the "overall intentions and directions of an organisation with regard to quality, as formally expressed by top management". The language used should be clear and precise but in general terms. It is important that the company's commitment to quality is clearly stated.

Quality Policy

This section should also contain a description of the system and procedures used to make the quality policy known to *all* employees.

4.4.5 The Organisational Structure

Management Organisation: Responsibilities and Rights

This section of the quality manual should provide a description of the high-level structure of the company. An organisational chart should be prepared showing the functions that report direct to the president and/or vice-president(s) of the dairy. Together with the organisational chart, a job description of each of the managerial functions should be prepared. These job descriptions should clearly outline responsibilities, authorities *and* rights.

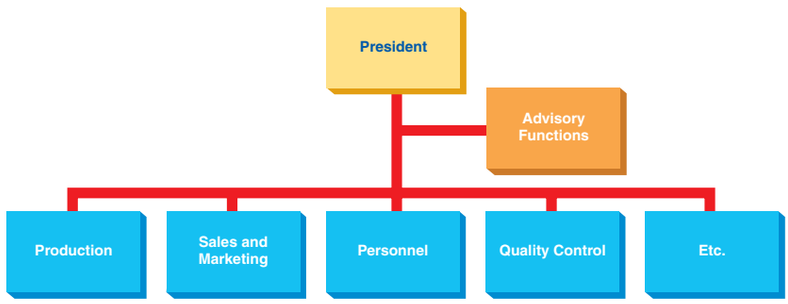


Figure 9: *Organisational Chart*

Simplify the Organisation!

A problem for many companies is that they have a rather complex organisational structure at management level: too many functions report directly to the president. Whenever possible, organisational structures should be streamlined for optimal function.

4.4.6 The Elements of the Quality System

This section is the largest one in the quality manual. It contains a general description of the quality system as implemented by the dairy. At this stage, it is necessary to have a coding system ready because frequent cross-references are unavoidable.

All the "elements of the quality system" should be addressed in the quality manual. For this reason, it is recommended that *all* the 20 sub-headings under section 4 of the ISO 9000 standard selected (9001, 9002 or 9003) be compiled in a list. For the dairy industry, ISO 9002 is usually applicable.

Sub-Section	Points to be Addressed
4.1 Management Responsibility	4.1.1 Quality Policy 4.1.2 Organisation 4.1.3 Management Review
4.2 Quality System	4.2.2 Quality System Procedures 4.2.3 Quality Planning
4.3 Contract Review	4.3.2 Review 4.3.3 Amendment to a Contract 4.3.4 Records
4.4 Design Control	4.4 Only applicable to ISO 9001
4.5 Document and Data Control	4.5.2 Document and Data Approval and Issue 4.5.3 Document and Data Changes
4.6 Purchasing	4.6.2 Evaluation of Sub-contractors 4.6.3 Purchasing Data 4.6.4 Verification of Purchased Product
4.7 Control of Customer-Supplied Products	
4.8 Product Identification and Traceability	
4.9 Process Control	
4.10 Inspection and Testing	4.10.2 Receiving Inspection and Testing 4.10.3 In-Process Inspection and Testing 4.10.4 Final Inspection and Testing
4.11 Control of Inspection, Measuring and Test Equipment	4.11.2 Control Procedures
4.12 Inspection and Test Status	
4.13 Control of Non-Conforming Product	4.13.2 Review and Disposition of Non-Conforming Product
4.14 Corrective and Preventive Action	4.14.2 Corrective Action 4.14.3 Preventive Action
4.15 Handling, Storage, Packaging, Preservation and Delivery	4.15.2 Handling 4.15.3 Storage 4.15.4 Packaging 4.14.5 Preservation 4.15.6 Delivery
4.16 Control of Quality Records	
4.17 Internal Quality Audits	
4.18 Training	
4.19 Servicing	
4.20 Statistical Techniques	4.20.1 Identification of Needs 4.20.2 Procedures

Table 2: *Elements of a Quality System (ISO 9000)*

ISO 9000
ISO 10 013

A problem may be encountered at this point in the documentation. The layout as recommended in ISO 10 013 does not exactly mirror the layout of the ISO 9000 (see table 1). If ISO 10 013 is followed (which we recommend), reference has to be made to each chapter referring to the respective ISO 9000 numbering.

Where Are We?

A short description should be given of the activities, procedures, etc., that have been implemented *at the present time*. In so doing, some shortcomings and faults may be recognised already at this stage. Of course, these should be

rectified and the implemented changes documented. It is important that the documentation mirrors what has actually been done. This provides the answer to the question "Where are we?" (see above).

4.4.6.1 Management Responsibility (ISO 9000 Standard 4.1)

Management Reviews!

The company's quality policy and its organisational structure have already been discussed. However, **management reviews** are of paramount importance. The term 'management review' is defined in ISO 8402 as a "formal evaluation by top management of the status and adequacy of the quality system in relation to quality policy and objectives". The following tasks of the management reviews should be considered and, if found adequate, documented:

1) to ensure that the company's quality policy is not violated;
2) to ensure that the documented methods and procedures are followed in reality;
3) to define clearly the goals of short and long-term quality improvement, both in terms of the actual results to be achieved *and* the time in which they are to be achieved;
4) to provide the means necessary to achieve the goals of quality improvement;
5) to ensure that the goals of quality improvement specified are achieved within the stipulated period.

Management Reviews are the Engine of the Quality System!

Specific procedures for these activities should be documented elsewhere. In the quality manual, reference should be made as to where in the documentation such procedures can be found.

Management reviews should be regarded as the engine that keeps the system going: they are necessary to maintain certification!

4.4.6.2 Quality System (ISO 9000 Standard 4.2)

Quality System

The term "quality system" is defined in ISO 8402 as the "organisational structure, procedures, processes and resources needed to implement quality management".

Quality planning is defined (ISO 8402) as "activities that establish the objectives and requirements for quality and for the application of quality system elements".

4.4.6.3 Contract Review (ISO 9000 Standard 4.3)

Contract Review

The term "contract review" has been defined in ISO 8402 as "systematic activities carried out by the supplier before signing the contract to ensure that requirements for quality are adequately defined, free from ambiguity, documented and can be realised by the supplier". Such contracts determine the conditions prevailing between the supplier (dairy) and recipient of products.

The dairy should prepare and document procedures which are intended to ensure that it is capable of fulfilling the conditions of the contract, not only with regard to quantity and the time of delivery but also with respect to quality.

Procedures should be elaborated and documented with regard to amending a contract. How should changes and additions to the contract, etc., be made?

4.4.6.4 Design Control (ISO 9000 Standard 4.4)

This section is only applicable for dairies which are developing products. It usually doesn't apply.

4.4.6.5 Document and Data Control (ISO 9000 Standard 4.5)

ISO 9000 states that "the supplier shall establish and maintain documented procedures to control all documents and data that relate to the requirements of this International Standard including, to the extent applicable, documents of external origin such as standards and customer drawings".

Procedures should be developed, implemented and documented to ensure that documents are reviewed for correctness. The person or persons authorised to approve documents and data should be identified by name and position. A system should be developed, implemented and documented to identify the issue currently valid. Furthermore, the system should ensure that:

Document and Data Control

a) the valid issue(s) of the document(s) is (are) available at the locations where the activities are performed;

b) invalid or obsolete documents are promptly removed from all points of use and properly disposed of;

c) any obsolete documents retained for legal purposes and/or the preservation of knowledge are suitably identified.

The system should also ensure that any changes in existing documentation have to pass through the same channels of approval.

4.4.6.6 Purchasing (ISO 9000 Standard 4.6)

ISO 9000 requires that "the supplier [dairy] shall establish and maintain documented procedures to ensure that the purchased product conforms to specified requirements".

A separate department for purchasing does not always exist in dairy companies. This may render documentation unnecessarily complicated – every department has to document its procedures separately.

Purchasing

Purchasing procedures for untreated milk are usually well established: the appropriate procedures are developed, documented and implemented. All other items needed are frequently purchased by each respective department.

Quality specifications should be developed and documented for all items which are of paramount importance for the quality of the end product. These should serve as the basis for purchasing.

Quality Specifications

A short description should be given of the purchasing organisation and its procedures in order to ensure compliance with the objectives listed above.

4.4.6.7 Control of Customer-Supplied Products (ISO 9000 Standard 4.7)

This section refers to procedures which are used to ensure that products are handled, stored and delivered according to acceptable and agreed upon conditions, both with regard to quantity and quality.

4.4.6.8 Product Identification and Traceability (ISO 9000 Standard 4.8)

ISO 9000 requires that "where appropriate, the supplier [dairy] shall establish and maintain documented procedures for identifying the product by suitable means from receipt and during all stages of production, delivery and installation".

Traceability!

A system should be developed, implemented and documented which permits the identification of end products back to their raw materials (ingredients). A general description of this system should be included in the quality manual.

4.4.6.9 Process Control (ISO 9000 Standard 4.9)

Process Control

Any dairy applying for ISO 9000 certification must have a system for process control. The main feature of any quality (assurance) system is process control. At this stage, reference should be made to the department manual and/or the manual of procedures depending on how the documentation has been arranged.

A *general* description should be included in the quality manual of the process control procedures applied, including:

a) the manner of production;

b) suitable control equipment;

c) compliance with reference standards if applicable;

d) monitoring and control of process parameters; and

e) suitable maintenance of equipment.

Always keep in mind that the quality manual is a public document. Confidential information should not be included in it, but reference should to be made to where specific facts can be found.

4.4.6.10 Inspection and Testing (ISO 9000 Standard 4.10)

Inspection and Testing

ISO 9000 states that "the supplier [dairy] shall establish and maintain documented procedures for inspection and testing activities in order to verify that the specified requirements for the product are met. The required inspection and testing, and the records to be established, shall be detailed in the quality plan and/or *documented procedures*".

A detailed description of the test procedures applied should be given in the manual of procedures and/or in the department manual. This should apply to:

a) raw materials;

b) intermediate products; and

c) end products.

The quality manual should contain a general description of such procedures *and* provide references as to where more detailed information can be found.

4.4.6.11 Control of Inspection, Measuring and Test Equipment (ISO 9000 Standard 4.11)

Control of Inspection, Measuring and Test Equipment

ISO 9000 requires that "the supplier [dairy] shall establish and maintain documented procedures to control, calibrate and maintain inspection, measuring and test equipment used by the supplier [dairy] to demonstrate the conformity of the product to the specified requirements".

This demands some consideration, since the standard also requires the identification of "all inspection, measuring and test equipment that *can* affect product quality and calibrate and adjust them at prescribed intervals …".

Calibration

The problem is that all instruments and measuring devices used in the dairy *can* affect product quality and, consequently, should be calibrated! In practice, this is neither possible nor necessary. A system should be looked for in order to reduce the number of instruments to be calibrated.

In the steps outlined for the production of dairy products (see "Production Manual, Product Files"), a distinction can be made between:

a) process steps;

b) control points;

c) hold points; and

d) critical control points.

Process steps are stages where no control is exercised at all, either because it is not necessary or because it is not possible.

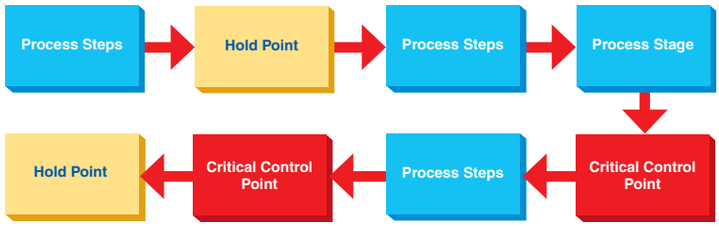


Figure 10: *Schematic Flow Chart: Production*

Control points are stations, functions, processes, operations, etc., where control can be exercised in order to ensure that the product meets its quality specifications.

ISO 8402 defines *a hold point* as a "point, defined in an appropriate document, beyond which an activity must not proceed without the approval of a designated authority". Processing beyond the hold point requires clearance by, for instance the quality control manager or any other authorised person.

Control Points
Hold Points
Critical Control Points

Critical control points are stations, functions, processes, operations, etc., where control can be exercised in order to ensure that the product is safe from a public health point of view (ISO 8402 defines safety as a "state in which the risk of harm [to persons] or damage is limited to an acceptable level)".

Calibration of instruments and measuring devices, etc., can be restricted to those functions which either control hold points or critical control points. In addition, the equipment used to analyse or determine the parameters that serve as a basis for payment (for example, of raw materials) also need to be calibrated.

If a system like the one described above is used, the principles should be outlined in the quality manual.

4.4.6.12 Inspection and Test Status (ISO 9000 Standard 4.12)

A short description should be given of the procedures used to ensure that all the products released on to the market meet their respective specifications or that their release has been granted by an authorised person.

4.4.6.13 Control of a Non-Conforming Product (ISO 9000 Standard 4.13)

ISO 8402 defines non-conformity as the "non-fulfilment of a specified requirement". The ISO 9000 standard requires that "the supplier [dairy] shall establish and maintain documented procedures to ensure that a product that does not conform to specified requirements is prevented from unintended use".

Deviating Products

The quality manual should contain a short description of the procedures used to ensure that the responsibility of deciding on action to be taken with regard to a non-conforming product is clearly defined and that the product is:

a) reworked to meet the specified requirements;

b) accepted with or without repair;

c) considered for alternative applications; or

d) rejected or scrapped.

Reference should be made to sections where such procedures are described in more detail.

4.4.6.14 Corrective and Preventive Action (ISO 9000 Standard 4.14)

Corrective Action

ISO 8402 defines *corrective action* as "action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation in order to prevent recurrence". Corrective action is part of the HACCP and QACP concepts.

Corrective action should be listed for each (critical) control point. For the dairy industry, it is recommended that corrective action for the process and the product be distinguished.

Preventive Action

Preventive action is defined by ISO 8402 as "action taken to eliminate the causes of a potential non-conformity, defect or other undesirable situation in order to prevent its occurrence". Again, preventive action is part of the HACCP (QACP) concept and should definitely be incorporated into a quality system.

As far as the quality manual is concerned, it should be stated that the idea of corrective and preventive action is incorporated into the procedures, and reference should be made to where the documented information can be found.

4.4.6.15 Handling, Storage, Packaging, Preservation and Delivery (ISO 9000 Standard 4.15)

Handling, Storage, Packaging, Preservation and Delivery

This section of the quality manual should contain a short description of the procedures used in the areas indicated in the title. Where appropriate, reference should be made as to where in the documentation more detailed information can be found.

4.4.6.16 Control of Quality Records (ISO 9000 Standard 4.16)

Control of Quality Records

The ISO 9000 standard requires that "the supplier [dairy] shall establish and maintain documented procedures for identification, collection, indexing, access, filing, storage, maintenance and disposition of quality records".

4.4.6.17 Internal Audits (ISO 9000 Standard 4.17)

Internal Audits

The ISO 9000 standard requires that "the supplier [dairy] shall establish and maintain documented procedures for planning and implementing internal quality audits to verify whether quality activities and related results comply with planned arrangements and to determine the effectiveness of the quality system." A quality audit is a "systematic and independent examination to determine whether quality activities and related results comply with planned arrangements and whether those arrangements are implemented effectively and are suitable to achieve objectives" (ISO 8402).

It should be underlined here that internal quality audits are a very important part of any quality system. They are one of the grounds on which the "management review" bases its decisions on quality improvement.

Internal Audits: Schedule

The quality manual should contain a statement indicating the commitment of the company to internal quality audits. A person should be assigned responsibility for the proper execution and recording of the internal quality audits. A recording system should be established and reference should be made as to where in the documentation more detailed information can be found on documented procedures for such audits. This documentation should include a schedule indicating the intervals at which such audits are to be executed in different departments. Before applying for certification, at least one internal audit should be properly executed and recorded.

4.4.6.18 Training (ISO 9000 Standard 4.18)

Motivation: Training and Education

A quality system requires the participation of every employee. Motivation of the working staff is of great importance. Training and education are activities which favour motivation and increase the ability of an employee to execute a task correctly.

A training and education programme should be developed, implemented and documented. The quality manual should contain a declaration of the dairy's commitment to the training and education programme and reference to where in the documentation more detailed information can be found.

4.4.6.19 Servicing (ISO 9000 Standard 4.19)

Servicing

The ISO 9000 standard states: "where servicing is a specified requirement, the supplier [dairy] shall establish and maintain documented procedures for performing, verifying and reporting that the servicing meets the specified requirements".

Dairies usually do not have *specified* service requirements and, consequently, this section is not applicable.

4.4.6.20 Statistical Techniques (ISO 9000 Standard 4.20)

Statistical Procedures

Generally, the ISO 9000 standard requirements for statistical techniques seem to cause problems. The standard demands that:

a) requirements for statistical techniques be identified; and

b) documented procedures be established and maintained.

Obviously, every dairy must already have some sort of reporting system to senior management in which detailed results are summarised. Usually, the system will utilise statistics in one form or another.

Summaries = Statistics

It appears likely that the introduction of functional management review procedures will require specific information that has to be summarised statistically. Graphical illustrations, tables, mean values with their respective deviations, etc., will be necessary.

The existence of such procedures should be indicated in the quality manual together with a statement as to future needs in this area and, principally, how those needs are to be met.

4.5 Manual of Procedures

Manual of Procedures

As shown in figure 5 on documentation structure, a manual of procedures can and should be prepared.

Any manual should, as much as possible, have the same layout as the quality manual. The following can be considered for inclusion in the "Manual of Procedures":

a) title, scope and field of application;

b) table of contents;

c) a description of the manual;

d) the section on the actual procedures included in the manual.

Procedures = A Specified Way to Perform Activities

ISO 8402 defines procedures as a "specified way to perform an activity". And: "A written or documented procedure usually contains the purpose and scope of an activity: what shall be done and by whom; when, where and how it shall be done; what materials, equipment and documents shall be used; and how it shall be controlled and recorded."

The question of whether a "manual of procedures" is actually necessary can be seen in the following alternatives:

Four Alternatives

a) a separate "manual of procedures" has not been prepared. The procedures are documented in the various department manuals which is where they belong since this is the document that each department works from. The problem is that there are some activities that lie outside the frame of department activities, (see b);

b) a separate "manual of procedures" has been prepared containing only those procedures which are specific to the activities described in the quality manual, such as internal audits, management reviews, etc. These activities are *not* executed by any specific department and, consequently, the procedures cannot be documented in a department manual;

c) a separate “manual of procedures” has been prepared containing those procedures which are specific to the activities described in the quality manual and, in addition, those procedures that are referred to in the quality manual;

d) a separate “manual of procedures” has been prepared containing all the documented procedures.

We would recommend alternative b) since this solution requires a minimum of documentation work yet still covers the entire operation.

4.6 Department Manuals

Layout of Manuals

Any manual should, as much as possible, have the same layout as the quality manual. Of course, the optimal layout depends upon the department in question. The most important aspect is the functional one! Organisational structure will differ from dairy to dairy and, consequently, no generally valid recommendations can be given. A common way of looking at different operations is the following: every operation has an input, a process and an output. The documentation can be arranged accordingly.

Figure 11: General Description of Operations

All dairies have a production department which is why it is used as an example below.

4.7 Manual of the Production Department

Manual of the Production Department

A manual of the production department could have a general and a special section. The general section contains information which is common to the department; the special section could be arranged in accordance with the different products produced. Thus the following layout may result:

I. *General section*

General Section

1) title, scope and field of application;
2) table of contents;
3) location of the department;
4) description of the organisational structure, including job descriptions with the rights and responsibilities of the different functions;
5) usually the reception of milk;
6) any procedures which pertain to the production department;
7) any instructions which pertain to the production department;
8) cleaning procedures, depending on the operation in question;
9) any other area, activity or function which pertains to the production department.

II. *Special Section* (arranged separately for each product: product files).

1) pasteurised milk for consumption;
2) butter;
3) others.

Product Files

A flow chart should be prepared for each product. Control points, hold points and critical control points should be clearly identified in it. For each hold point, the parameters should be stated on which further passage down the line is based. In the documentation, the person authorised to release the product for further treatment should be identified (quality control manager or other person responsible). Reference should be made to those sections of the documentation where the parameters are checked (usually the laboratory, i.e., the quality control manual). Where applicable, control or critical control parameters should be identified.

Flow Chart

If possible and/or feasible, the following should be specified for each (critical) control point or parameter:

(Critical) Control Point/Parameter

- *the critical limit;* a critical limit separates acceptability from unacceptability. Such limits do not have ± values. A violation of a critical limit requires corrective action;
- *target value;* the target value is the value at which a process is intended to be executed. Target values may, and often do, have a ± value, a range;
- *preventive action;* preventive action is the implementation of activities which prevent a critical limit from being violated. Guarding functions, maintenance, calibration, challenging, etc., are forms of preventive action;
- *corrective action;* corrective action may be related to the process and/or the product. What is being done to restore the process and ensure its operation within the critical limit? What is being done with regard to a product that has been produced under conditions of process deviation?;
- *verification;* ISO 8402 defines verification as “confirmation by the examination and provision of objective evidence that specified requirements have been fulfilled”. In the production of pasteurised milk, the negative phosphatase or peroxidase test is a verification procedure;
- *validation;* ISO 8402 defines validation as “confirmation by the examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled”.

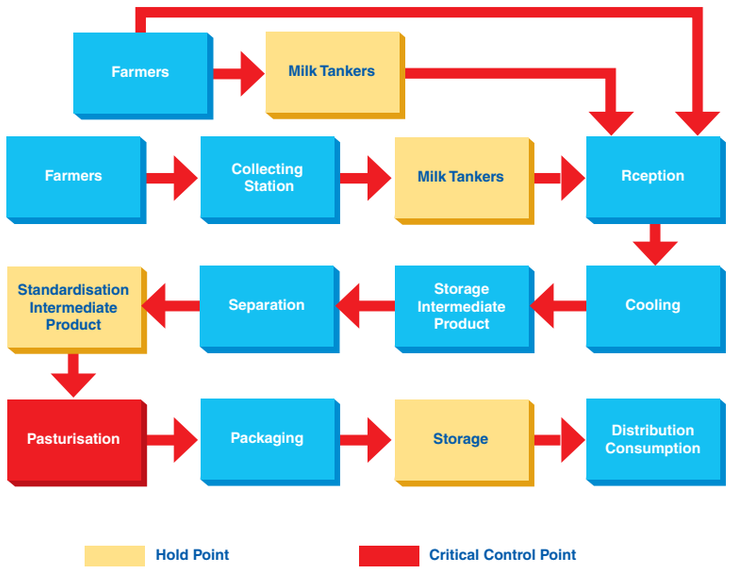


Figure 12: *Flow Chart: Production of Pasteurised Milk*

The Equipment Used

The equipment used in the production line should be identified by stating:

- the name of the manufacturing company;
- the kind, type and model of equipment;
- the year of manufacture and/or installation;
- the capacity of the equipment.

Internal Identification System

It is advisable to identify the equipment by an internal numbering system to which easy reference can be made. Such identification marks should be attached to each piece of equipment.

Maintenance schemes should also be listed or referred to, depending on whether service and maintenance are carried out by the production department or not. Persons responsible for these activities should be identified.

Product Specifications

Specifications of raw materials, intermediate products and end products should be included in the documentation, or reference should be made as to where in the documentation material these specifications can be found.

Any procedures and instructions used specifically for the production of the product under study should be documented here.

The documentation should describe clearly the test methods and procedures which are used, and *not* those which should be used. This documentation will serve as the basis for quality improvement.

Ready for Certification?

As soon as the documentation has been prepared for all departments and at least one internal audit has been executed and recorded, the dairy is ready for certification and can apply accordingly.

5. Certification

5.1 General

How to Apply for Certification?

As mentioned above, the certifying authority has to be identified at an early stage in the process of obtaining ISO 9000 certification. Different institutions follow different procedures. All of them require an official request from the dairy and a certain amount of information. However, already at this stage some differences may exist.

In short, the following steps are usually involved in the certification process:

- obtain information from the certifying authority, on how to proceed;
- contact the authorised institution selected and inform it that the dairy believes it is ready to apply for ISO 9000 certification;
- material is sent by the certifying institution containing among other things a form for making an official application for certification. Usually, other information is also required by the certifying institution;
- the material has to be filled in and submitted to the certifying institution;
- the certifying institution acknowledges receipt of the application and asks that the quality manual be submitted, usually together with the manual of procedures and a request for a down payment.
- having studied the material submitted, the certifying institution will be either satisfied or not. If objections exist, these have to be identified and specified by the certifying institution and straightened out by the dairy. Eventually, the application is accepted;
- an auditing team is dispatched to the dairy in order to study the remaining documentation and the implementation of the documented methods and procedures. It is called an external audit. If the audit is to the satisfaction of the auditors, a recommendation will be given to the board of the certifying institution which, in due course, will issue the ISO 9000 certificate.

5.2 Cost of Certification

Cost of Certification

The process of certification is not free of charge. There are costs involved and different authorised institutions may and probably will have different charges. When considering the costs of certification, attention should be paid not only to the actual certification expenses, but to the costs of maintaining certification as well.

6. Maintaining Certification

6.1 General

Certification Is Not the Goal But the Beginning

ISO 9000 certification is not the goal of the procedure, it is the start of an uninterrupted process: the continuous operation of a quality system.

Certain requirements are connected with maintaining an ISO 9000 certificate.

6.2 Internal Audits

Internal Audits

Internal audits are to be carried out at regular intervals by personnel "independent of those having direct responsibility for the activity being audited". The task of the internal audit is to:

- ensure that the company's quality policy is not violated;
- ensure that documented methods, procedures, etc., are implemented as documented;
- ensure that any corrective and/or improvement action suggested earlier is properly implemented;
- record any deviation from documented procedures;
- list any suggestion for improving methods and procedures, etc.

Record-Keeping

Suitable records have to be prepared from each of the audits. These records should be submitted to the management responsible for the area in which the audit was executed, as well as to the group comprising the management review team.

6.3 External Audits

External audits will be carried out at regular intervals by the certifying institution, often with a frequency of once a year. Usually, after a period of three years, the certificate has to be "renewed", i.e., a complete revision of the ISO 9000 documentation and its implementation has to be executed.

6.4 Management Reviews

Management Reviews

ISO 9000 - 1 states "One of the important activities that the executive management of the supplier [dairy] organisation needs to carry out systematically is an evaluation of the status and adequacy of the quality system, including the quality policy, in relation to the expectations of the stakeholders. Management reviews usually take into account many additional factors beyond the requirements found in ISO 9001, ISO 9002 or ISO 9003. The results of internal audits and external audits are an important source of information. It is important that the outcome of the management review should lead to increased effectiveness and efficiency of the quality system".

Based on information obtained not only from internal and external audits but also from summary reports submitted by the different departments and any other suitable source, the management review should define quality improvement goals. Graphical presentation allows for rapid evaluation and permits the detection of trend developments. The statistical evaluation of certain parameters often provides a more accurate and reliable ground for decisions.

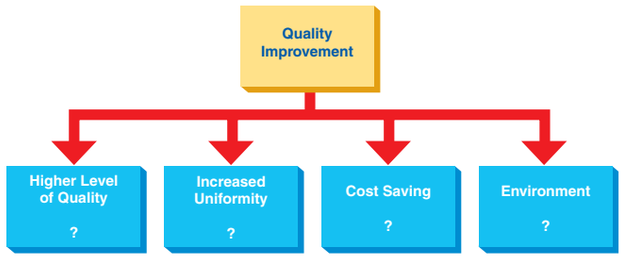


Figure 13: *Quality Improvement*

Quality Improvement

Quality improvement goals must be stated precisely with regard to the actual goal and the time in which it is to be achieved. Follow-up procedures must be developed in order to ensure that the goals are met as planned. For this reason, adequate funds have to be provided.

6.5 Motivating the Workforce

MOTIVATION!

Any quality system requires the active participation of the total workforce of a company. Motivation is essential if this is to be realised.

Training and education of the staff is important. Suitable programmes should be developed by the personnel department. A system for recording these activities should be devised and implemented.

The staff should be informed at an early stage about any planned activity for improvement. Information should be given on a regular basis as to progress. Co-operation within the workforce requires such information. Progress should be shown graphically if possible. Suitable procedures will have to be devised from case to case. The responsibility of ensuring that the information is given in a suitable way rests with the department manager.

14. Troubleshooting

Summary

Troubleshooting refers to those activities which need to be activated if a process or a product deviates from its respective definitions or specifications. Problems can be solved in an intuitive way by immediate action or by first systematically analysing the situation. Troubleshooting activities, particularly systematic troubleshooting, can be rather expensive, especially if the goal is to identify the cause of the problem. Only then can action to prevent repetition of the fault be taken. Intuitive problem-solving cannot be discussed in any detail. The systematic approach requires information and data. In the present chapter, the systematic approach of microbiological troubleshooting is discussed in some detail.

1. General

The pre-condition for effective troubleshooting is the existence of suitable quality specifications. Troubleshooting activities have to be implemented as soon as a product (or where applicable, a process) deviates from the specifications or when a trend towards such a deviation has become evident.

Troubleshooting Requires Quality Specifications!

One of the more expensive faults is the attempt to solve a problem which doesn't exist.

Troubleshooting is often done in an ineffective way. The following mistakes (261) are common in microbiological problem-solving:

a) the fault is not the subject of the investigation: an imagined defect is rectified;

b) quick technical adjustments change the situation to such an extent that an analysis is no longer possible. In addition, new faults are often introduced. Uncontrolled action leads to gaskets being changed, connections being tightened, equipment being adjusted and cleaned, and so on. Parameters are "corrected" without knowing why. Having taken all these actions, the problem often disappears. However, the different measures are neither co-ordinated nor recorded and many activities are executed simultaneously;

c) often more than one reason can cause or contribute to the problem. The most important reason, however, is not identified;

d) shortage of time, lack of personnel, and organisational inefficiency contribute to the confusion;

e) a systematic bacteriological analysis is either not done or the results are not correlated to the technical findings.

Basically, problems can be attacked in two different ways:

1) by the intuitive; or

2) the systematic approach.

Two Ways to Solve Problems: By Intuition or Systematically

There are advantages and disadvantages to both.

2. The Intuitive Approach

Intuitive troubleshooting is the non-systematic approach to problem-solving. The first step is action. Consequently, it can be very quick, which is its main advantage. Time is a very, if not the most, important aspect of troubleshooting (261). In addition, intuitive problem-solving is inexpensive. On the other hand, the cause of the problem is not always identified and, consequently, no action can be taken to prevent its repetition. This is the main disadvantage. Further disadvantages with the approach are that not all problems can be solved and intuitive troubleshooting cannot be taught. It requires experience, intuition and luck.

Intuitive Troubleshooting is Fast but Does Not Identify the Cause!

One of the problems which cannot be solved by the intuitive approach is shown in figure 1. When a production line commences commercial operation, operational experience is obtained and used to improve the "production quality level". However, after a certain period of time, additional experience is applied to "make the job easier". Most people are lazy! As a result, the level of production quality gradually falls until a situation is reached where the "level needed for successful marketing" is no longer maintained. The staff involved in the process are unaware of what is happening. Changes are not made intentionally! Thus, problems result which are very difficult to solve since they are caused by a relatively large number of mainly operational mistakes, each one of which would be of minor significance on its own.

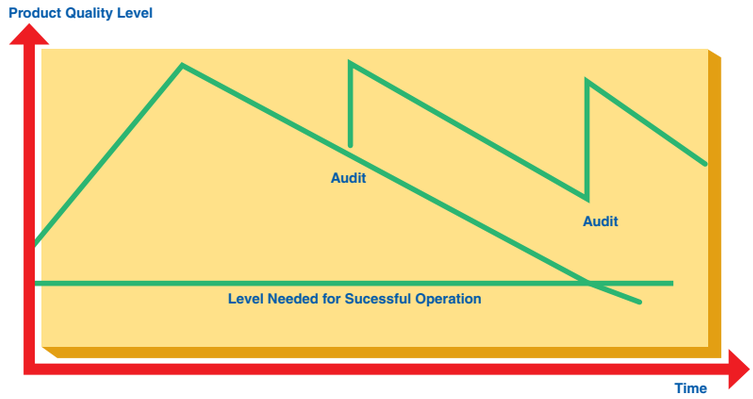


Figure 1. *Development of the "Production Quality Level" over Time*

Such problems can be prevented by regular technical audits executed by an expert outsider, a system, which some larger companies have adopted (144).

3. The Systematic Approach

Systematic Troubleshooting Information is Needed!

In systematic troubleshooting, the first step is thinking: existing data are analysed, a theory is formed. Usually additional information is needed which should be collected in the right way. By gathering and scrutinising information, the area causing the problem is successively narrowed.

This can be done either by

- identification or
- exclusion.

Systematic Troubleshooting = Team Work

Systematic troubleshooting is teamwork. Members of the team should represent quality control, production, and the department where the problem is most likely to be located. Proper training of the permanent members of the troubleshooting team is necessary: thorough knowledge of the products and technology involved is required.

Cause is Identified Repetition Can be Avoided

The main advantage with systematic troubleshooting is that the cause of the problem is identified and, consequently, measures can be taken to prevent its repetition. In addition, all problems can be solved even though it may take considerable time to arrive at their solutions. Systematic troubleshooting can be taught.

Systematic Troubleshooting = Time Consuming!

Because of the time factor involved, pure systematic troubleshooting is rarely used in practice. Consequently, the cause of a problem is not clearly identified, the location of the fault is only traced to certain areas. Fault frequencies can be established and used for preventive action. However, process optimisation based on fault frequencies is impossible (262). Systematic troubleshooting may be very time-consuming and expensive.

In a long-life product production line, the source of microbiological problems can be expressed in a simple equation:

Defect Rate = Process Survivors + Reinfection.

If all microorganisms are killed by the sterilisation of the product and packaging material, and if reinfection of the product is eliminated, the resulting defect rate is zero.

Microorganisms surviving UHT treatment of low-acid products are always Gram positive organisms, often thermophilic bacteria, and usually spore-formers. Though relatively rare, the most common sources are high spore counts in the raw materials and/or intermediate products. If powders are used in the formulation of the product, proper soaking is essential.

Failure of the UHT process as such is less frequent. On the other hand, reinfection often results in a mixed flora dominated by vegetative, non-spore-forming microorganisms (80). However, pure reinfection with spore-forming microorganisms is also possible, particularly as a consequence of inadequate cleaning and equipment sterilisation as well as by leakage in heat exchangers and other equipment operating at a temperature above ~ 80°C.

In practice, a mixture of intuitive and systematic troubleshooting is usually applied. A major danger with systematic troubleshooting is outlined in figure 2. A problem is encountered and some, usually insufficient, data are available relating to the problem. Consciously or unconsciously, a theory is formed as to where the problem is located. Next, an experiment is designed to prove the theory, but not to disprove it! In this procedure, only data is collected which on the surface support the theory. After some time, the people involved are honestly convinced that their theory is correct. The results and findings are reported to management and from that stage on prestige is part of the process. Once prestige has come into the picture, the issue is no longer that of identifying the cause of the problem and solving it, but of proving that **I am right.**

A Common Fault in Troubleshooting

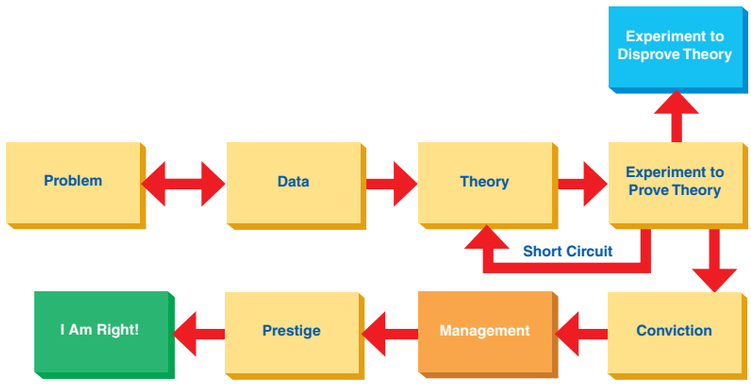


Figure 2. *Danger with Troubleshooting*

A typical and common situation is the following (figure 3) (261). In an operation, fifty packages are routinely taken for incubation from each of two filling machines (A and B) in a production line. On one occasion, one defective package is found in the sample taken from filler B and none in that taken from filler A. Knowingly or unknowingly, a theory is formed: the problem is located in filler B. To prove the theory, additional samples are taken from filler B but *not* from filler A which would disprove the theory. Obviously, in so doing, only evidence that points to filler B is accumulated, thus giving further support to the theory.

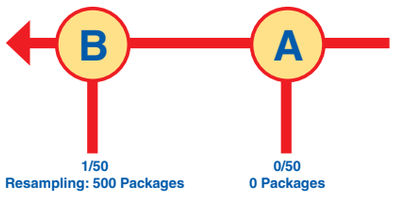


Figure 3. *Faulty Re-sampling in Troubleshooting*

3.1 Microbiological Troubleshooting

Consumer Expectations

What is a microbiological problem? The question really is: what does the consumer expect from a food product! Does the consumer demand perfection or not? Here a distinction should be made between the consumer as an individual and consumers as a group. The individual consumer does not anticipate that the package he or she picks up from a shelf is unsterile: the individual customer expects perfection. The consumer group reacts differently.

A certain, albeit limited, defect rate is acceptable, i.e., it does not interfere with marketing. The acceptance level differs for different products and consumer groups but is larger than zero. It is very important to keep this in mind.

What is a Problem?

After proper incubation, is the detection of one defective unit in a sample a problem? Opinions differ on the subject. For the reason outlined above, sterilising processes do *not* kill all bacterial spores. They achieve a certain number of decimal reductions in spore counts. Consequently, there have to be "process survivors" even if their number is very low. Other sources of failure such as reinfection must also be considered.

Assuming a production of 100,000 packages, one of which contains a surviving bacterial spore capable of growth and multiplication, i.e., one potentially unsterile package. If a sample is drawn from this batch (production run), there is a certain, albeit very limited, probability that the unsterile package will be included in the sample. Although the detection of one unsterile package is not necessarily a problem, every defect unit found in a sample of limited size should be regarded as an indication of a possible problem: re-sampling procedures should be implemented.

The Time Factor Involved: Proper Record-Keeping is a Necessity!

Microbiological problems are usually detected first after the elapse of some time. Proper incubation and the evaluation of samples are needed before recognisable defects can be detected. Consumer complaints take even more time. Retrospective searches to trace the cause(s) of end-product failure are easier to carry out if all relevant records have been completed conscientiously and correctly (86).

Spoilage Caused by Microbial Growth or Not?

If a spoiled product is encountered, it is important to decide whether the spoilage is caused by microbial development or not (84). For this reason and for purposes of "rough identification", a streak should be prepared using normal "plate count agar". The total count as such is of no interest (158). To determine such counts is an unnecessary and expensive exercise.

As outlined above, the first step in systematic troubleshooting is to form a theory. The accuracy of the theory is determined by the exactness and amount of information available. Usually, the information at hand is insufficient and inconclusive and, consequently, additional information needs to be gathered. What kind of basic data is required and/or may be helpful?

What Kind of Information is Helpful?

The factory surroundings are of relevance. In addition, it is necessary to gather information on:

1) the equipment involved;
2) the installation;
3) the type of product produced;
4) the tightness of the packages;
5) the size of the problem;
6) the type of product spoilage;
7) the type of spoilage flora (86);
8) the distribution of spoiled packages over the production run;
9) the history of the problem.

Systematic troubleshooting is like solving a puzzle. Information has to be gathered, put together, analysed and, most important, the different items of information have to fit together. Then and only then will a clear picture emerge!

Machine operator records on processing and packaging are naturally examined for the detection of operational faults and events. When evaluating data of any kind, the reliability of the source should also be considered.

Basically, a distinction should be made between three "classes" of information:

a) information which is gathered by the "troubleshooter(s)": facts, procedures and methods are known – the most reliable source of information;

b) information which is gathered by someone known to the "troubleshooter(s)": the source of information with his strengths and limitations is known; this information is of less reliability;

c) information which is obtained from an unknown person; nothing is known about his reliability. Everybody reports what he or she thinks is right! The information should be treated with caution.

It should be kept in mind that the reporting of facts and results is subjective. They are not always "knowingly" falsified, but are presented in the way as seen and perceived by the person concerned. Lack of data is one dilemma, too much and often irrelevant and incorrect information may be another! As far as possible, relevant information should be summarised and critically scrutinised.

Based on the "theory" formed from the information available, additional data are usually required. Do keep in mind that data collection should be aimed at both proving and disproving the theory.

3.1.1 The Equipment

At home base, this is not, or should not be, a problem: the equipment should be known. However, the lack of knowledge about equipment among the quality control staff is a shortcoming. By the same token, production staff often have a very limited knowledge of laboratory work.

Frequently, outsiders lacking a basic knowledge of the equipment used are involved in problem-solving. They have to know what kind and make of equipment is involved, and how old and well maintained it is.

3.1.2 The Installation

The way equipment is installed may or may not have an impact on the results obtained from a production line. Again, at home base this should not be a problem, but outsiders are not familiar with the installation. In any event, a flow chart should be prepared outlining the basic functions of the production line(s) affected.

3.1.3 The Type of Product

Depending on their composition, different products permit the growth of different microorganisms. The kind of product changes inflicted by microbial growth depend on the kind of product in which they develop. It is important to know the basic composition of the product, particularly if sugars have been added – how much, and of what kind.

Powders used in the formulation may be insufficiently soaked. Soaking procedures are critical and should be described in a flow chart.

3.1.4 The Size of the Problem

The level of unsterility is often expressed in per cent. Even though a percentage figure is informative in itself, it is far better to state the number of containers tested and the number of defective packages found, as this will permit the use of the Poisson distribution graph. In this respect, information on the conditions of incubation (time and temperature) and methods of evaluation is necessary.

3.1.5 The Type of Product Spoilage

Product changes must be described (84). They should include pH, gas formation ("blown" packages), coagulation and the type of coagulum (soft, hard, "broken", etc.,), whey separation, smell and/or taste (questionable because of a possible risk of pathogenic microorganisms being present), and any other pertinent change. From a microbiological point of view, typical product changes can be expected only if the unsterility is caused by a pure infection, i.e., a single kind of microorganism only.

3.1.6 The Types of Spoilage Flora

One of the first questions should be (80): what type(s) of microorganism is (are) causing the unsterility? This information is usually very helpful, but often not available.

For this reason, a "rough identification scheme" has been developed which is easy to execute, inexpensive, quick, and in almost all cases sufficient for the purpose at hand. It is, however, not scientific!

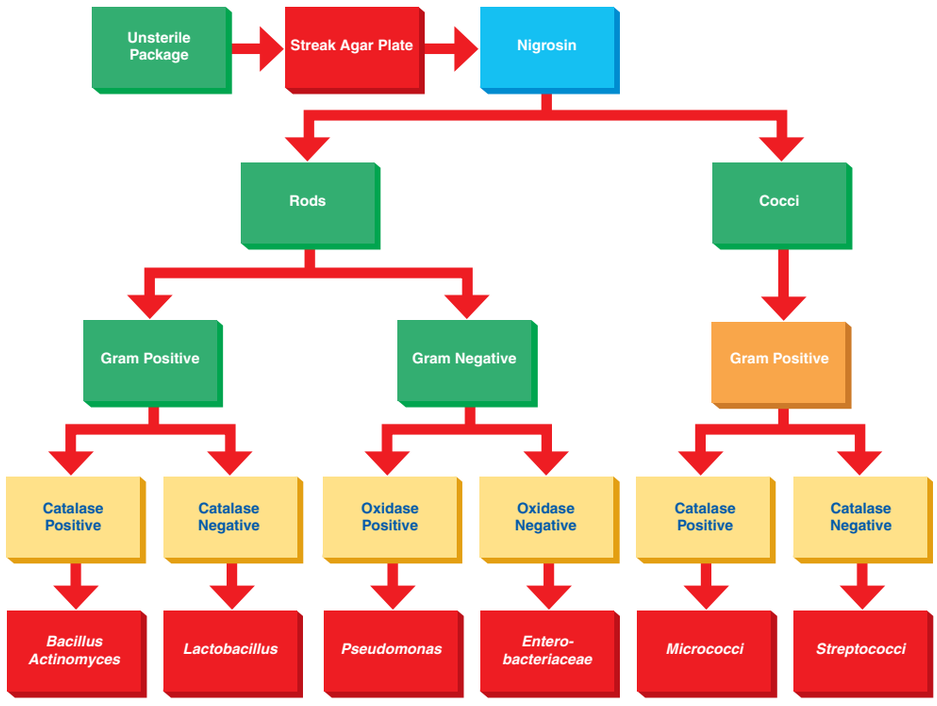


Figure 4. *Rough Identification Scheme*

The Types of Spoilage Organism are Important Information

The "rough identification scheme" divides the spoilage organisms into six rather large groups of microorganism (figure 4). Each of these indicates a certain, different source of infection. *Bacillus* is the most heat-resistant group and *Pseudomonas* the most sensitive. The temperature limits for effective killing are

presented in table 1 below. Because the groups comprise a large number of different bacteria, the temperature limits have to be treated with caution. The importance of conducting such an analysis has been stressed in the literature (159).

When opening packages for microbiological analysis, aseptic conditions are desirable but not necessary. The number of spoilage organisms in the spoiled product is so high that a certain amount of contamination during handling and opening can be ignored (159). A distinction should be made between mixed and pure infections. Only one kind of microorganism causes a pure infection, whereas more than one kind are involved in mixed infections. To begin with, these should be regarded as "pure" infections caused by the most sensitive organism.

Mixed and Pure Infections

The most important tool of troubleshooting is common sense! Some microbiological knowledge is required for interpreting correctly the results obtained by the rough identification scheme. All pertinent information has to be considered and *must* fit together. The groups of bacteria identified (figure 4) are indications and not proof. In table 1, an attempt has been made to list some possible sources of contamination together with the effective temperatures for killing the bacteria in different groups.

Rough Identification

Bacillus do not multiply in high-acid products. Typically, they are proteolytic causing a bitter flavour and a soft coagulum. Gas is produced only rarely. *Lactobacillus, Streptococcus* and *Enterobacteriaceae* attack carbohydrates rapidly, forming acid and often gas. The pH usually drops well below 5. *Actinomyces, Micrococcus* and *Pseudomonas* cause limited product changes only. *Pseudomonas* may temporarily even raise the pH and develop a fruity or fishy off-flavour.

Different Information Must Fit Together!

When bacteriological problems occur in long-life milk, the cause can be either (177):

a) the survival of heat-resistant spores; or

b) a secondary contamination (reinfection).

Indicator Organism	Effective Killing Temperature	Process Survivors Reinfection	Source
Bacillus	> 100°C	Process Survivors	Intermediate Product, Equipment, Pack.Mat.,
Actinomyces	> 90°C	Both	Air, Dust
Lactobacillus	> 80°C	Reinfection	Milk, Aerosols, Human
Streptococcus	> 80°C	Reinfection	Milk, Aerosols, Human
Micrococcus	> 80°C	Reinfection	Milk, Aerosols, Human
Enterobact.	> 75°C	Always Reinfection	Milk, Human
Pseudomonas	> 65°C	Always Reinfection	Water, Milk, Aerosol

Table 1. *Source and Heat Sensitivity of Indicator Organisms*

A high percentage of the defective packages in 25 cases of unsterility contained pure cultures of spore-forming bacteria growing at 30°C (177).

Type of Infection	Number	%
Total Number Tested	25	100
Mesotrophic Spoilage	18	72
Thermophilic Spoilage	16	64
Spore-Formers Only	19	76
Mesotrophic Spore-Formers	6	24
Thermophilic Spore-Formers	8	32
Mixed Infections	3	12

Table 2. *Spoilage Flora in Unsterile Packages*

The raw milk used had unusually high counts of bacterial endospores: average 2,100/ml, range: 1 to 13,600/ml (177). This may have caused the majority of cases of unsterility. However, a reinfection with spore-forming and other bacteria is clearly indicated because of the incidents of mixed infection.

3.1.7 The Distribution of Spoiled Packages

When plotting the number of unsterile packages found against the production time, a pattern will emerge (84) which can provide valuable information. A sufficient number of unsterile packages is therefore needed, amounting to five or even more. Re-sampling is sometimes necessary to achieve that number. However, it should be borne in mind that usually other data have to be gathered and fitted into the overall picture: patterns indicate areas alone, but are seldom proof in themselves.

Basically, the following regularities can be observed:

The defect rate may be high at start-up, followed by a “wash out”. Towards the end of production, the unsterility rate may or may not increase (Pattern A, figure 5). Such a situation is often, but not always, encountered when plant sterilisation (cleaning) problems exist. Due to a pressure drop during start-up of the filling operation, leakage in the cooling section of the UHT equipment can also result in such a pattern. Other explanations are possible.

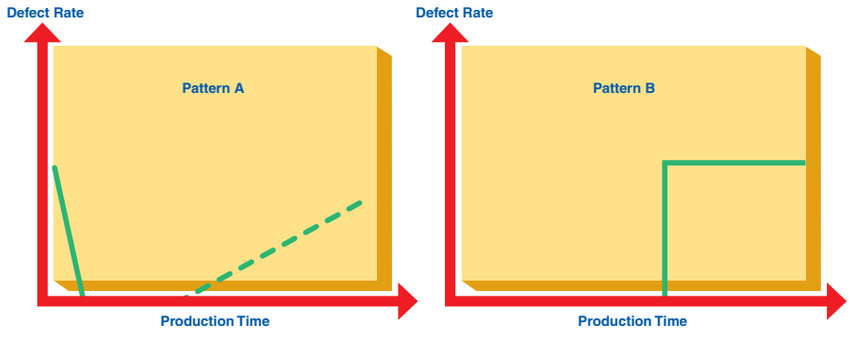


Figure 5. *Distribution of Unsterile Packages*

The first part of production may be sterile followed by a sudden (Pattern B, figure 5) or a slow (Pattern D, figure 6) increase in unsterility.

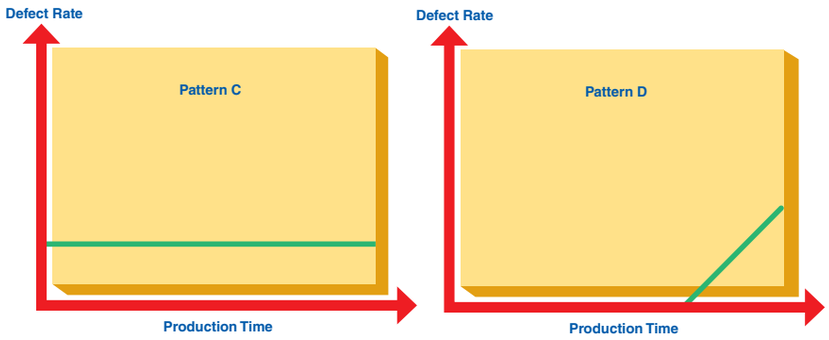


Figure 6. *Distribution of Unsterile Packages*

Operative errors or machine faults may cause such a pattern. The change to a new batch of intermediate product should also be considered as a possible cause. A change of packaging material in combination with a lack of personal hygiene, as well as other sporadic failures, can explain such a pattern.

Defective packages may be evenly distributed over the total production run (Pattern C, figure 6). Faulty sterilisation of the product and/or packaging material, and many other reasons, may result in such a picture.

At a certain time, one or more peaks of unsterility may be observed with no unsterility before or after (Pattern E, figure 7). Such sporadic unsterility is rather frequent (80). Often operative faults are the explanation.

Obviously, these pictures indicate different causes.

A better and more informative way of presenting such a diagram, especially if the installation contains more than one filling machine, is shown in figure 8.

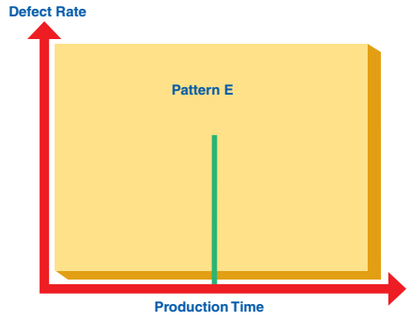


Figure 7. *Distribution of Unsterile Packages*

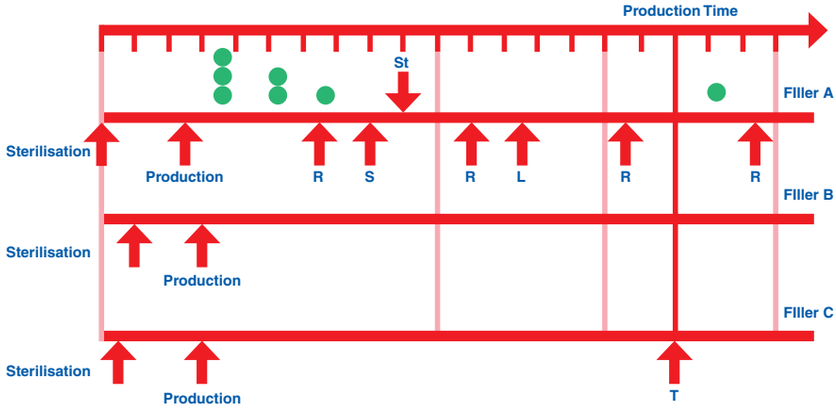


Figure 8. *Incidences of Unsterility and Changes in Production*

A time axis represents the total production run, starting from sterilisation of the equipment to the end of production. For each individual filling machine, occurrences, such as changing the packaging material blanks, reels (R) or longitudinal strip (L), stops (S) and starts (St), etc., are entered. All the relevant changes in production conditions are included, such as changing from a steriliser to a sterile tank and back, etc. In the above diagram, the incidence of unsterile packages is marked accordingly. Often there is a correlation between an event and unsterility which indicates that the event may have caused the problem.

As Much Information As Possible Should Be Included!

The information included in the above diagram is obtained from records. These may either be electronically recorded or automatic "print-outs" from the equipment recording systems or manually prepared machine-operator protocols. Automatic records should contain the date of production, the time that production was started, and should be signed by the machine operator and supervisor in charge.

Machine Operator Records

Manual records should be treated with caution:

a) the primary task of machine operators is to attend to the equipment. Record-keeping is of secondary importance. If a problem occurs, the operator is supposed to attend to the problem first. At best, protocols are filled in "after the event". Consequently, time statements are usually not accurate to the minute (only in the order of magnitude);

b) if a machine operator makes a mistake, it is not likely that his error will be mentioned in the record. Either the event will not be listed at all, or something else "documented" in its place.

3.1.8 History

What has happened in the past? Is unsterility a single occurrence or not (84)? Often such information is not available and cannot be generated. By studying laboratory records and consumer complaints, valuable information can sometimes be gathered from the past. Again, diagrams should be prepared (figures 9 and 10).

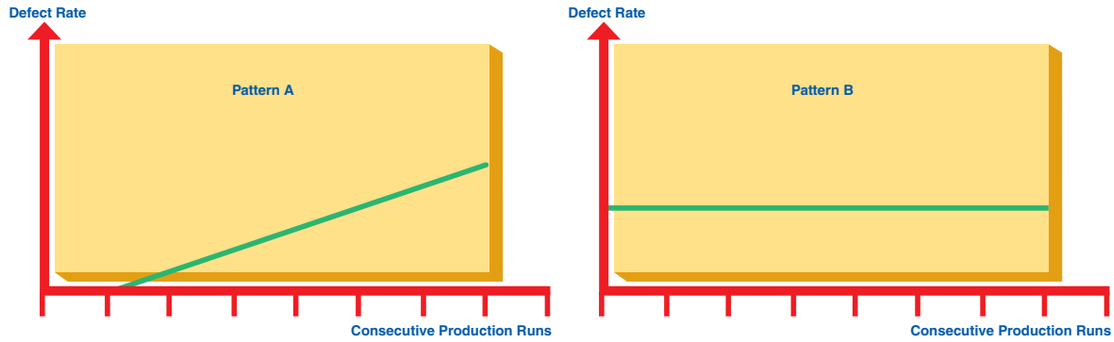


Figure 9. *Unsterility: Its History*

In such diagrams, certain regularities may or may not be evident. Unsterility may increase from production run to production run (figure 9, Pattern A). Such a pattern indicates a systematic fault, an example of which is defects caused by the wear and tear of equipment (159). The defect rate may be approximately the same all the time (figure 9, Pattern B), for instance, a persistent fault such as insufficient soaking procedures for powders. Sometimes no regularity is discernible at all (figure 10, Pattern C). This may be due to the lack of regularity or may be caused by choosing the wrong group. Rearrangement by, for example, plotting the days of the week, may result in a pattern. This can be done by accumulating the results from the production runs on Mondays, Tuesdays, Wednesdays and so on. Unsterility may be so sporadic, so occasional that a systematic analysis becomes impossible. Such faults have to be included in the AQL. (159).

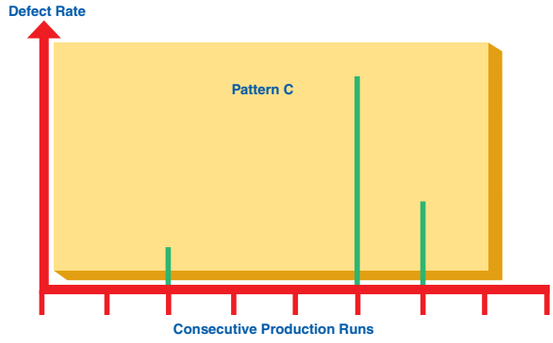


Figure 10. *Unsterility: Its History*

In this type of diagram, different product groups should not be mixed. High-acid and low-acid products (even if produced on the same production line) must be treated separately.

3.2 Zero Sampling

The purpose of systematic troubleshooting is to narrow, step by step, the area in which the fault most likely exists. This can be done either by identification or by elimination. In this connection, the answer to the question: "Is the problem located before, during, or after the filling operation?" is of considerable interest. For this purpose, zero samples are often used.

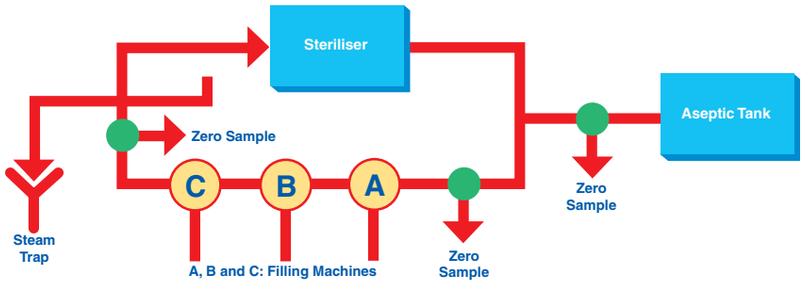


Figure 11. *Zero Sampling*

Sampling devices are introduced at different places in the production line (figure 11). The product is sampled at these spots, incubated and analysed for microbial growth. If the sample is found to be negative, it is assumed that the problem originated after the spot at which the sampling device was installed. The problem is the limited volume of product that can be taken for analysis (262).

This is generally true but particularly if the characteristic under study is an attribute (such as "unsterility"). This is shown in figure 12.

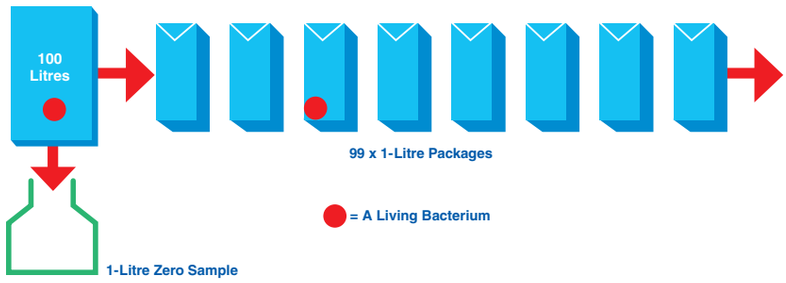


Figure 12. *Zero Sampling of an Attribute*

In the example above, a receptacle contains 100 litres of product in which one living bacterium is present. When the product is filled into 1,000 ml cartons, one of them will be unsterile: a defect rate of 1%! Simultaneously with the filling operation, a zero sample of one 1-litre volume is drawn. The probability of catching the bacterium in the zero sample is small: on average, one per 100 trials will give a positive result, and 99 will indicate that the problem is after the receptacle! As the volume of the zero sample increases, the probability of finding the bacterium in the zero sample also increases. The same applies if a larger number of living bacteria (i.e., a higher defect rate) is present in the container.

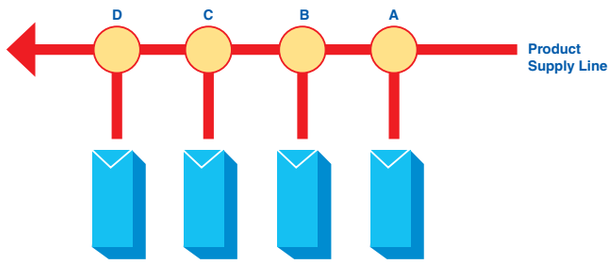


Figure 13. *An Alternative Procedure to Zero Sampling*

In production lines with more than one aseptic filler, a better and more accurate way of locating the problem is to compare the defects sampled from different filling machines (figure 13).

Multiple Filling Machine Installation

Packages for incubation are drawn from each filling machine (A, B, C and D) in the production line. Observe that equal volumes have to be compared. If each of the fillers produces the same package volume, the same number of packages should be sampled from each filling machine. Assume filler A produces 1,000 ml packages and filler B 250 ml packages. Four times as many packages will have to be sampled from filler B than from filler A.

Prior to incubation, four 250 ml packages are "bundled" and regarded as a single unit. Irrespective of whether one, two, three, or all four packages are unsterile, they are still regarded as one package (figure 14).

Using statistics, it can be established whether any difference in the number of unsterile packages found is significant or not. A significant difference indicates a fault in the filler, while absence of significance points to a common cause to the problem, i.e., the product supply.

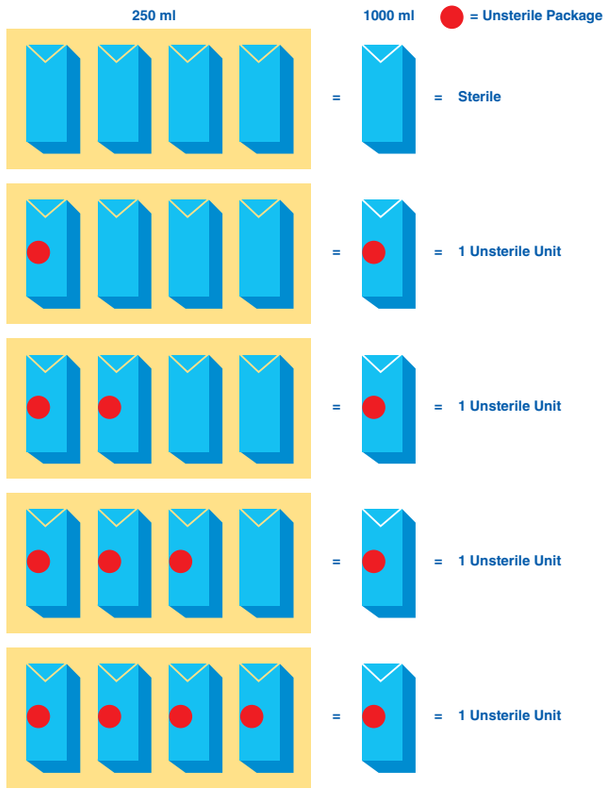


Figure 14. *"Bundling" Packages When Different Volumes Are Produced*

Lowest No.	20% Error Risk	10% Error Risk	5% Error Risk
0	4	5	6
1	6	7	8
2	7	9	10
3	9	10	12
4	10	12	13
5	12	13	15
6	13	15	17
7	14	16	18
8	16	18	20
9	17	19	21
10	18	20	23
11	20	22	24
12	21	23	25
13	22	24	27
14	23	26	28
15	24	27	29
16	26	28	31

Table 3. *Significance of the Difference in Sampling from Two Fillers in the Same Production Line*

Table 3 has been calculated (197) in order to assess whether or not the difference found in two different samples is significant.

Using the installation shown in figure 13 and assuming the same package volume for all four fillers, the application of table 3 can be demonstrated by the example below. An equal number of packages is drawn from each filler, incubated and evaluated.

Example 1:
Two defective packages are found in the sample from filler A and none in the other three fillers. Does this result indicate a fault in filler A? Is the difference between two defective packages and no defective packages significant? Consulting table 3, a minimum of four defective packages is needed to reach the "20% error risk" level: the difference cannot be regarded as significant. To clarify the situation, more samples should be drawn from all fillers.

Example 2:
Eight defective packages are found in filler B, one in filler D and none in the other two fillers. Is there a significant difference between filler B and the others? Again, consulting table 3, the "5% error risk" level is reached in a comparison between filler B and the others. In all likelihood there is a fault in filler B. The one defective package found in filler D should be taken as a strong indication that re-sampling is necessary (see Example 1).

Of course, table 3 can be used to evaluate any comparative attribute sampling situation.

4. Examples of Troubleshooting

1. General

Considering the very large number of long-life, low-acid food products on the market, very few cases have so far been reported in the literature of long-life products and long-life milk being suspected of causing food poisoning.

Usually, the information available on cases of microbial spoilage of long-life products is insufficient to identify the cause of the problem. This is particularly true if a mistake has caused unsterility. This can be illustrated with two examples.

2. Operational Faults

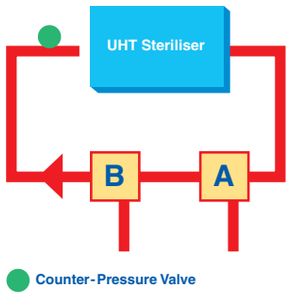


Figure 1. *Example of Unsterility: Installation*

2.1 "Sudden Unsterility"

The normal production schedule of a manufacturer of long-life milk was about 22 hours followed by cleaning and sterilisation of the plant: a typical three-shift operation. The installation was straightforward (figure 1): a UHT steriliser connected to two aseptic fillers.

On one occasion, the following situation evolved: a production run of 36 hours was scheduled. One of the filling machines was "clean" throughout the entire production period. The other was sterile for about 24 hours and then suddenly showed ~100% unsterility (figure 2).

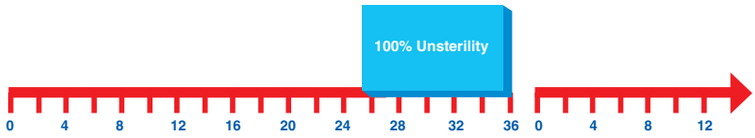


Figure 2. *Unsterility*

After cleaning and sterilisation, production was resumed the next day and both fillers showed a satisfactory result.

This is a typical example of an operational mistake. However, neither the print-out chart in the filler nor the machine operator protocol gave any indication of a fault. The exact reason for the failure will never be identified, it can only be speculated on.

2.2 "Sudden Unsterility"

A very serious situation evolved in a plant producing high-acid, long-life products. The production schedule of the plant was rather unusual in that it violated GMPs. Production commenced on Monday morning and continued until noon on Saturday. The system was cleaned on Saturday afternoon and sterilised on Monday morning before production restarted. The installation was straightforward and simple.

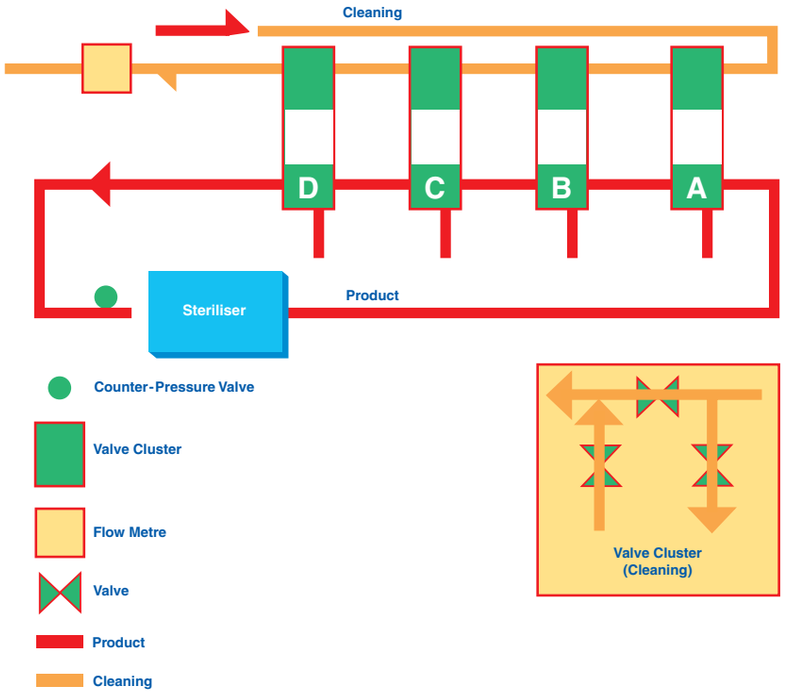


Figure 3. *Example of Unsterility: Cleaning Arrangement*

A product steriliser was directly connected to four TBA/9 fillers. A separate cleaning line was connected by manually operated valves to each of the four fillers (figure 3).

The following situation developed:

Figure 4. *Example of Unsterility: Pattern*

Up to the Saturday, production was acceptable. On the following Monday, from start-up to the end of the run on Saturday, one of the four fillers became 100% unsterile. After cleaning and sterilisation, production commenced on the following Monday morning, and all four machines showed a satisfactory result: a typical indication of an operational error. Again, records did not reveal anything unusual. The exact cause will never be identified. In this case, however, it appears most likely that the manually operated cleaning valves were operated incorrectly, excluding the affected machine from the cleaning cycle.

Even though, the actual cause of the above problems was never clearly identified, the fact that they were caused by operational errors is of some help. The likelihood of repetition can be reduced by either better supervision and/or by revising operational procedures (QACP concept).

3. Cases From the Literature

Some cases of microbial spoilage of low-acid, long-life products have been described in the literature. However, the information which can be extracted is very limited, as illustrated by the three cases below. A clear identification of the cause of spoilage is therefore not possible.

3.1 Example One

A normal production run. No complaints from the market were registered. Nevertheless, quality control regularly found large numbers of samples having severe sensory faults: after incubation, the milk tasted sour and had a pH of 6.15. Microbiological checking revealed a count of about 10^8 spore-forming bacteria/ml. Vegetative microorganisms were not found. Milk samples collected from the market were also infected but the number of germs was too low to cause recognisable spoilage. The laboratory incubated the samples at 40°C, a temperature high enough to permit multiplication of thermophilic organisms (80), but higher than the normal environmental temperature, hence no complaints.

3.2. Example Two

A total production run was affected. The problem was not detected in the normal quality control but rather from market complaints. The milk had a very bitter taste and an unpleasant smell. Plating and incubation at 30°C did not reveal any microbial growth. However, after 24 hours at room temperature, abundant development of *Pseudomonas* was observed (80).

3.3. Example Three

Long-life cream developed an offensive flavour. The inner polyethylene coating had become partially removed from the aluminium foil. Microbiological analysis showed that the massive infection was caused by *Pseudomonas*. In order to clarify whether or not delamination was the cause or the consequence of the infection, good packages were inoculated with material from spoiled ones. All the inoculated packages developed the same delamination fault (80).

4. Hypothetical Examples

The following examples are based on more than 25 years of field experience in microbiological troubleshooting. The cases are slightly simplified and adapted to demonstrate systematic troubleshooting.

4.1 Leakage in the Final Cooler of the Steriliser

Systematic troubleshooting requires the collection of information.

4.1.1 The problem

During normal production, about 50 packages are randomly sampled from each filler and production run. In addition, aimed samples are taken at start-up of the filling operation and after each stop and re-start and change of packaging material. The sampled packages are incubated for five days at 30°C and evaluated by sensory change and pH determination. One day, one defective package was found from filler A. Re-sampling was carried out on all three filling machines, collecting 500 packages from each filler. After incubation, an additional four defective packages were detected. By plating (streak), it was determined that the spoilage was caused by microbial growth.

4.1.2 Equipment and Installation

The UHT steriliser is a tubular heat exchanger produced by company A. It has been in operation for approximately five years. The capacity of the steriliser is stated to be 9,400 litres/hour. Three aseptic filling machines supplied by company B are connected to the steriliser. Each of the filling machines has a nominal capacity of 3,000 litres/hour. The filling equipment is of the same age as the product steriliser (figure 5).

A spring-loaded, counter-pressure valve provides the filling pressure needed by the three (A, B, and C) filling machines.

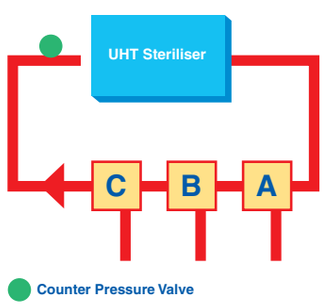


Figure 5. *Example of Unsterility: Installation*

4.1.3 The product

Plain, white, long-life milk.

4.1.4 Re-Sampling

A total of 1,650 random samples and an additional 100 aimed samples were tested. Four unsterile packages were found equalling 0.3% of the sample. A check of package integrity did not reveal any indication of a leakage problem.

4.1.5 Evaluation

The sensory changes recognised were a bitter flavour and a fishy smell. The pH of the spoiled milk was 6.9.

4.1.6 Product Changes and Spoilage Flora

Product changes had already indicated the presence of *Pseudomonas*. A quick test with 3% KOH solution showed slime development. The Nigrosin stain revealed rods. By Gram staining, the result of the KOH test was confirmed: Gram negative rods. The oxidase test resulted in the development of a dark brown colour: oxidase positive, confirming the indication of the "*Pseudomonas*" group.

4.1.7 Pattern

By arranging the five unsterile packages in a time diagram, a pattern was shown. The figure strongly indicates a connection with start-up and re-start of the filling operation (figure 6). Defective packages were also found in all three filling machines at about the same level of unsterility:

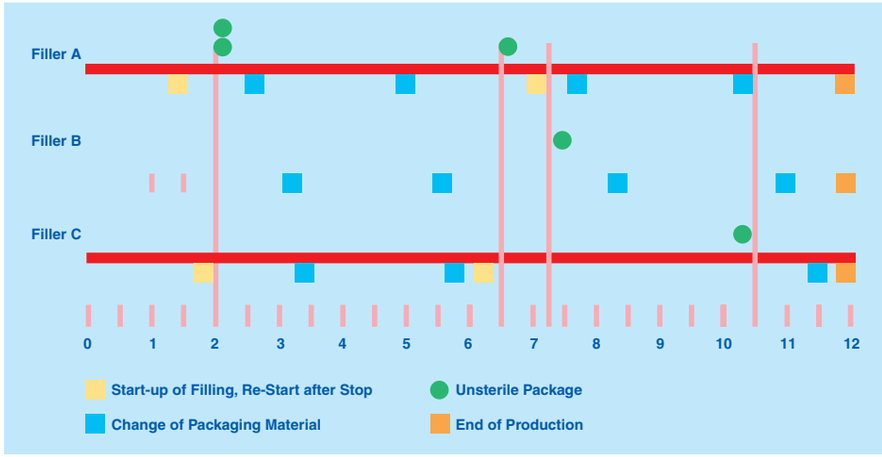


Figure 6. *Example of Unsterility: Pattern and Production*

- Filler A: 3 out of 640 packages tested were not sterile: ~0.5%
- Filler B: 1 out of 630 packages tested was not sterile: ~0.2%
- Filler C: 1 out of 630 packages tested was not sterile: ~0.2%

4.1.8 History

No information was available as to the "history", i.e., previously encountered problems.

4.1.9 Analysis

An evaluation of the aimed samples did not reveal defects among the start-up packages of each respective filling machine, i.e., the first two packages analysed after the filling operation was started at the beginning of the production run and after re-start.

Defective packages were detected in all three filling machines but at a slightly different level. Is there a significant difference between the three, indicating a

fault in the filling systems? Statistics reveal that the difference between the three fillers is not significant (compare table 3, on page 163 in the section on "Trouble-shooting"):

	Lowest No.	20% Error Risk	10% Error Risk	5% Error Risk
Needed	1	6	7	8
Filler		A	B	C
Found		3	1	1

Table 1. *Is the Difference between the Fillers Significant?*

The number and volume of packages checked from each of the three fillers is comparable. To achieve 80% probability (20% risk), which would indicate a significant difference between the results obtained from the different fillers, a minimum of six defective samples is needed, since the lowest number found (fillers B and C, table 1) is one defective package! Only three were found (filler A, table 1), the difference not being significant and pointing to a common source to the problem, i.e., the product supply.

Moreover, it is not very likely that a fault would arise at the same time in all three fillers resulting in about the same number of defective packages. All this points to a common source: the product supply.

The type of spoilage as well as the spoilage flora (*Pseudomonas* group), strongly indicate a reinfection caused by the entry of water. The microorganisms of the *Pseudomonas* group are very sensitive to heat; they are killed at temperatures of > 70°C. At which point can water enter the product after the outlet of the holding cell and at a temperature of < 70°C? A closer look at the equipment and installation may help (figure 7).

The product is fed into the regenerative heating section by means of a centrifugal feeding pump. After homogenisation, the milk passes the final heater, the holding cell, the regenerative section and reaches the final cooler. Water is used in the final cooler of the UHT plant (figure 7).

How can water get into the commercially sterile product? The plant is designed to provide an overpressure on the sterile side. Even if a leakage arises in the final cooler, sterile product will be forced into the unsterile water and not the other way round.

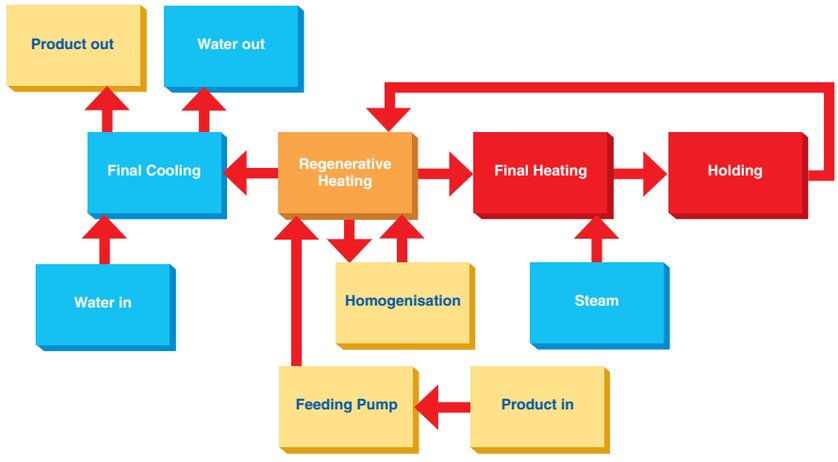


Figure 7. *Example of Unsterility: UHT Steriliser*

If the aseptic filling equipment is started, usually during a short period of time, more product is consumed than the amount corresponding to the nominal capacity of the filler: the system has to be filled up with product while the filler is running.

This additional demand may, and often does, exceed the amount of product supplied by the steriliser. A pressure drop on the sterile product side may reverse the pressure situation in the final cooler: the unsterile water pressure becomes higher than the product pressure. During a very short period of time, water enters the commercially sterile product causing a reinfection which is subsequently washed out of the system. A short period of unsterility results whenever the last filler is started. This infection may manifest itself in any of the three fillers in the line.

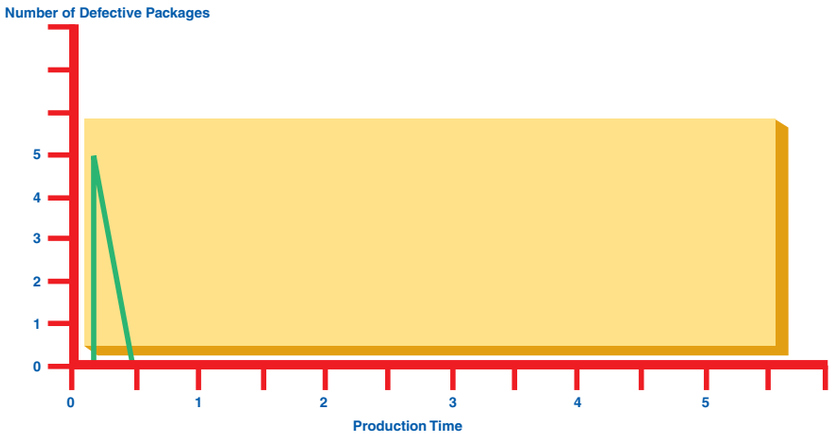


Figure 8. *Example of Unsterility: Typical Pattern Caused by Leakage in the Final Cooler*

Looking at the situation closer, the following pattern can typically be observed (figure 8). After the last machine in the line has been started, a pressure drop is encountered resulting in an infection of the product. However, the product in the line is still sterile; the first packages after start-up are produced from the product in the line and are sterile.

But after a short period of time the "unsterility plug" reaches the filling equipment resulting in infected packages. As long as the leakage is small, only a limited number of bacteria will enter the system. These may show up in any of the three filling machines in the line. The effect can be minimised, possibly even eliminated, by installing a counter-pressure valve at the steriliser outlet or, better, a booster pump at the end of the final heater.

5.2 Counter-Pressure Valve

A plant was undergoing microbiological commissioning. The conditions agreed on were the following:

a) three independent commissioning runs (cleaning and plant sterilisation between each of the runs);

b) from each run, a total of 2,400 packages (total production) are to be taken as samples from each of the three filling machines; the total number of samples to be tested is 3 x 2,400 x 3 = 21,600;

c) conditions of incubation: 5 days at 35°C;

d) evaluation: pH measurement and sensory. A pH deviation of 0.2 units and/or a recognisable sensory change is regarded as failure;

e) acceptance level: for each filler, a total of three defective packages is acceptable, but no more than two packages in any of the three test runs.

After incubation of the test samples, (21,600 packages), a total of 35 defective packages (~0,2%) were detected, which is an unacceptable result.

Points a-e above describe the sampling methods and procedures used. Systematic problem-solving requires the proper use of *all* information available.

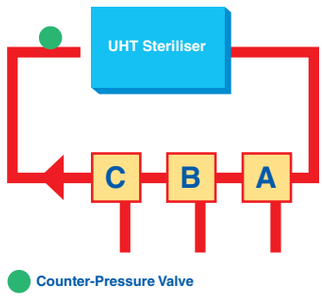


Figure 9. *Example of Unsterility: Installation*

a) *Equipment and installation:* Three aseptic filling machines were connected directly to a UHT steriliser (figure 9). Each of the filling machines produced 1-litre packages and had a capacity of 6,000 packages/hour. The nominal capacity of the steriliser was 19,000 litres/hour.

b) *The product:* Milk was used as a test product.

c) *Sampling:* See above; 3 x 2,400 packages from each filler.

d) *Evaluation:* See above; pH and sensory data..

e) *Product changes and spoilage flora:* Most of the defective packages were blown (gas formation), coagulated and had a pH of 4.6; some were coagulated and had a pH of 6.2. The blown packages contained a mixed flora with both Gram positive and Gram negative rods. The Gram negative rods were also oxidase negative: the *"Enterobacteriaceae group"*. The Gram positive rods were both catalase positive and negative: a mixture of *Bacillus* and *Lactobacillus*.

f) *Pattern:* The packages were put into trays, each of which contained 12 units. The trays were numbered consecutively. Since the majority of unsterile packages were blown, these were what constituted the investigation at this stage. The following picture evolved (figure 10): in all three test runs, blown packages were found at the beginning of the run in filler C only.

g) *History:* not applicable.

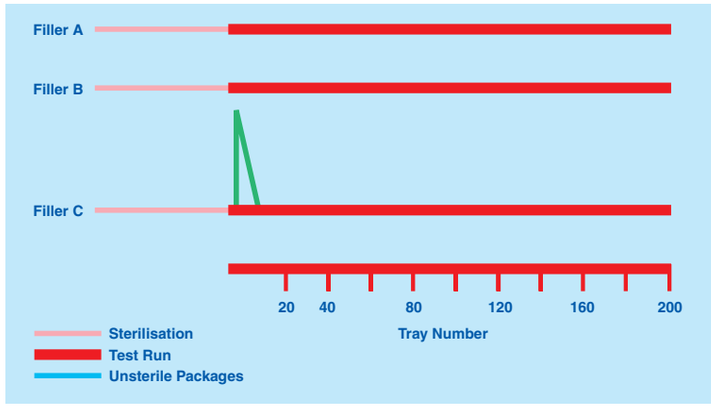


Figure 10. *Example of Unsterility: Pattern*

A close inspection showed that the defective packages were tight.

Statistics reveal a significant difference between filler C on the one hand, and A and B on the other. This clearly indicates a defect in connection with filler C.

A closer look at the tray numbers showed that all the defective packages (figure 11, filler C) were located in the first five trays.

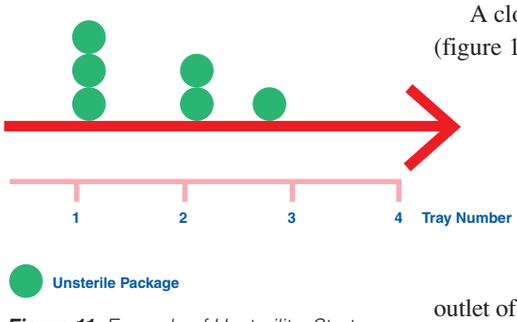


Figure 11. *Example of Unsterility: Start-up Pattern*

An extensive inspection of filler C did not show any abnormalities which would explain the findings. A significant difference between fillers may have been caused by a defect in the filler but it may also have been due to the location, installation, etc., of the equipment. A closer look at the installation showed the following. The counter-pressure valve, which is not an aseptic valve, was placed very close to the last filler (C) in the production line: distance ~ 30 cm (figure 12). The outlet of the return line was above the product level in the balance inlet tank of the steriliser. Due to the design of the filling machine, whenever the filling operation is started more product than the one corresponding to the normal capacity (6,000 l/hr) is needed: the equipment operating at full speed has to be filled with product. The steriliser delivers an amount of product (≈18,500 l/hr) which is only slightly above the total nominal volume needed by the three fillers

(18,000 l/hr). Whenever the last filler is started, the product demand exceeds the supply: an "under-pressure" results.

Unsterile air is sucked into the system through the return line, reaching and entering the last filler in the line (filler C). Irrespective of which filler is started last, filler C will always be affected. The problem can be solved by increasing the distance between the counter-pressure valve and filler C to ~10 metres or more. A further improvement can be made by placing the outlet of the return line below the product level in the balance inlet tank (figure 12).

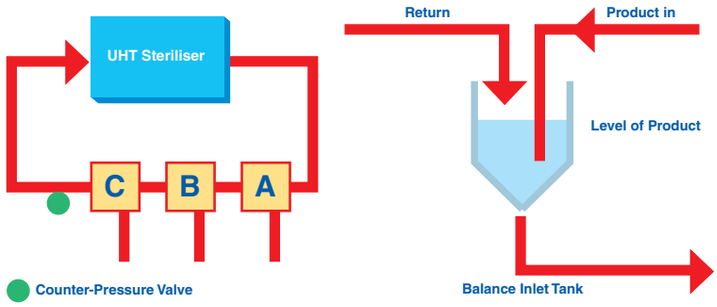


Figure 12. *Location of the Counter-Pressure Valve*

The other defective packages found are within the acceptance level and should for the time being be ignored.

After replacing the counter-pressure valve at a safe distance from filler C, the commissioning test was repeated and gave an acceptable result.

5.3 Poor Quality of Intermediate Product

A plant had been producing long-life products for some time before unsterility was encountered. Market complaints indicated an unacceptable level of spoiled product.

5.3.1 Equipment and Installation

The UHT production line consisted of an indirect UHT plant (plate heat exchanger), an aseptic tank and five aseptic filling machines (figure 13).

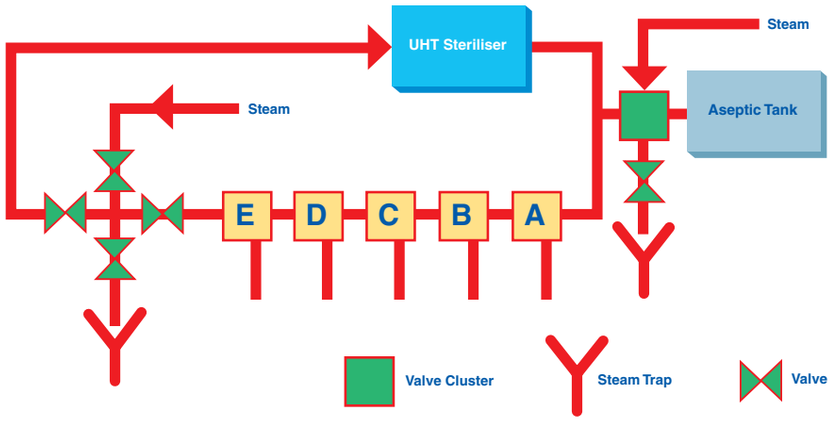


Figure 13. *Example of Unsterility: Installation*

5.3.2 The product

Reconstituted long-life milk.

5.3.3 Sampling

About 20 packages were sampled randomly from each filling machine and production run. In addition, aimed samples were selected at the start-up of each production run.

5.3.4 Incubation and Evaluation

The sampled packages were incubated for five days at 35°C; evaluation was carried out by testing the pH and by sensory procedures.

5.3.5 Product Changes and Spoilage Flora

In the quality control procedures, coagulated packs were found only occasionally. These were ignored. Triggered by the market complaints, a substantial re-sampling procedure was initiated. 500 packs were collected at random from each of the five fillers. They were taken from a production run that had been in storage for five days. Since the temperature in the product storeroom was 25-30°C, the re-sampled packages were opened without any further incubation. A large number (58) of spoiled packages were found. The product had coagulated and had a pH of 6.1 to 6.4. No gas formation was registered. Rough identification revealed Gram-positive, catalase-positive, spore-forming rods: *Bacillus*.

5.3.6 Pattern

Arranging the defective packages in a time diagram, all five fillers showed the same pattern (figure 14).

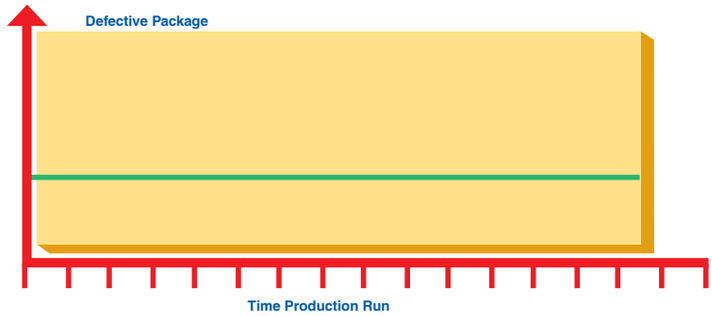


Figure 14. *Example of Unsterility: Pattern*

The spoiled packages were evenly distributed over the entire production run. The number of defective packages found in each filler is shown in table 2. There was no significant difference between them (compare with table 3 on page 163, in the section on "Troubleshooting").

A	B	C	D	E
9	15	8	11	15

Table 2. *Is the Difference between Fillers Significant?*

3.3.7 History

Occasionally, spoiled (coagulated) packages were found at quality control. These and the complaints received from the market, were ignored.

At this stage, the following is what we know:

a) all five fillers are affected to about the same extent indicating a common source to the problem, namely the product supply;

b) the defective packages are evenly distributed over the entire production run;

c) the type of spoilage and the spoilage flora are the same in all the spoiled packages: *Bacillus*. Vegetative microorganisms must have been eliminated by some selective factor. Process stages where such selection can take place are UHT treatment (high spore count of the intermediate product), the sterilisation of the plant (poor cleaning of the equipment), and the sterilisation of the packaging material (high spore count on the packaging material, poor hygienic conditions in the filling area).

A plant sterilisation failure is not very likely since a different pattern of unsterility usually results: high at the beginning of a production run followed by a washout.

All the ingredients used in the preparation of the reconstituted milk were examined: spore counts were rather low. A check on the intermediate product, however, revealed extremely high bacterial endospore counts of the magnitude of ~500,000/ml. Further investigation revealed very poor cleaning in the section where the powder was reconstituted.

15. Long-Life, Low-Acid Products

Summary

Rationalisation of the food industry has resulted in fewer food processing plants and, as a result, greater distances between the points of production and consumption. This in turn has led to growing demands for a longer shelf life for food products. Energy-saving requirements favour the non-refrigerated handling of such commodities. On the other hand, and to an increasing extent, consumers demand natural foods. Changes brought on by processing should be minimised! Since almost all microorganisms can grow in low-acid food products, the risk of spoilage is high. In addition, pathogenic disease-causing bacteria can also develop. Such products represent a public health risk. Commercially sterile long-life products represent an ideal compromise: processing changes are reduced to a minimum, the spoilage risk is insignificant and the public health hazard is practically eliminated.

In the present chapter, the composition of, and processing conditions for some low-acid long-life products are given.

1. Long-Life Milk

For commercial and practical reasons, an increase in the shelf life of milk and milk products is highly desirable. Commercial sterilisation (UHT treatment) together with aseptic packaging considerably extends the shelf life of milk and milk products (2).

A number of problems are associated with the storage quality of long-life milk. The most common of these is age-thickening which can eventually lead to gelation or coagulation and may limit the shelf life of the milk to eight months or less. This defect is caused by thermo-resistant proteases of bacterial origin (mainly *Pseudomonas* and *Bacillus*) and is less common in recombined than in fresh, long-life milk. Inactivation of these enzymes by UHT treatment is limited, often less than 10% (271). The enzyme levels must be kept to a minimum by handling the untreated milk and carrying out the pre-processing treatment in such a way that the number of bacteria capable of producing thermo-resistant proteases is kept at a low level.

Homogenisation of milk subjected to UHT treatment is necessary in order to prevent fat-separation during storage. Homogenisation should result in an average fat globule size of 0.7 µm with a maximum of 1-2 µm (180).

Homogenisation

In Germany, in 1975, 1,580 UHT milk samples produced by 28 producers were purchased and tested for their microbiological properties. 1.3% of the samples were spoiled due to putrefaction, acidification or other anomalies of taste. In 7.8% of the samples, microorganisms which multiplied at 37°C were demonstrated to be present. In part, these microorganisms could only be isolated by enrichment procedures. Most of the strains isolated were identified as *Streptococcus lactis* and aerob *Bacillus*. Only 6% of isolates belonged to groups of Gram negative bacteria (227). In this connection, the usefulness of enrichment culture procedures can be seriously questioned. In the meantime, the situation has improved considerably: today defect rates are much, much lower and failure rates of 0.01% or less are often achieved.

1.1 Lactose-Reduced Milk

Some people suffer from lactase deficiency (lactose intolerance). When consuming milk (lactose), digestive problems arise. A method for lactose hydrolysis in sterile milk or whey with lactase obtained from *Saccharomyces lactis* has been described (103, 104, 195). The properties and stability of this enzyme are very

favourable for use in milk and neutral whey. The shelf life of long-life milk and whey products allows for sufficient reaction time - one to several weeks - at room temperature to lessen the lactose content (figure 1).

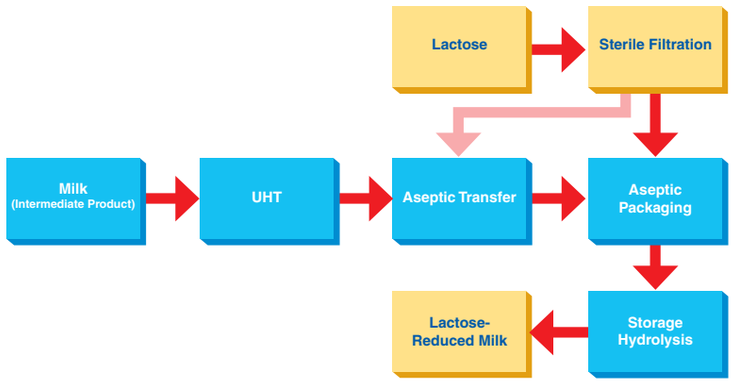


Figure 1. *Addition of Lactase to Long-Life Milk*

Consequences of Lactose Hydrolysis

Before being added to commercially sterile milk, the lactase has to be sterilised by filtration since the enzyme is heat-sensitive and will be inactivated by the UHT treatment. The sterile enzyme solution can either be added before or, preferably, during the aseptic filling operation (figure 1).

When lactose is hydrolysed, galactose and glucose are formed. Both these sugars (monosaccharides) are sweeter than the lactose. Consequently, the hydrolysed milk has a sweeter taste (196). In addition, glucose and in particular galactose react more readily in the Maillard reaction: the hydrolysed milk is more sensitive to browning and to the development of off-flavours associated with the Maillard reaction.

2. Goat's Milk

Long-life goat's milk has been produced by steam injection (169). During storage at different temperatures (4°C, ambient, 30°C, 37°C and 50°C), the following changes were registered (169):

a) at 50°C, the non-protein nitrogen increased from 8.2% to 13.1% of the total nitrogen content within 71 days. The flavour had acquired a sharp, bitter taste already after 7 days. The content of available lysine was reduced. The amount of 5-hydroxy-methyl-furfural had increased (Maillard reaction) and after 14 days the milk was distinctly brown in colour;

b) the non-protein nitrogen also increased at 37°C and to a lesser extent at 30°C and room temperature. Even after two years, the increase at 4°C was insignificant. A sharp, bitter flavour different from that observed at 50°C developed after storage for 21, 71, 105 days and 2 years respectively. The available lysine decreased and the level of 5-hydroxy-methyl-furfural increased during storage at ambient, 30°C and 37°C. The samples became brown after 71 days, 6 months and 10 months at 37°C, 30°C and room temperature respectively;

c) the peptone fraction increased during storage and the casein content decreased. This suggests an enzymatic degradation of the casein. Goat's milk contains a natural proteolytic enzyme associated with the κ-casein, which may have been reactivated during storage.

In conclusion, the shelf life of long-life goat's milk is shorter than the one for cow's milk. Storage at high temperatures should definitely be avoided.

Long-Life Goat's Milk = Shorter Shelf Life!

If the initial pH of goat's milk subjected to UHT processing is below 6.9, rapid and severe sedimentation occurs in the product. The problem may be controlled either by adjustment of the pH of the milk to well above 7.0, or by the

addition of 0.2% di-sodium phosphate to the milk before processing. The higher calcium content of goat's milk compared to cow's milk is probably responsible for the greater instability of goat's milk during UHT processing (272).

3. Recombined Long-Life Milk and Milk Products

3.1 General

The term "recombined" is used to describe a process where milk powder, butter fat and water are used, while "reconstituted" (65) refers to a procedure where only powder and water are utilised.

Ingredients: Recombined Long-Life Milk

Stabilisers and possibly emulsifiers have to be added in the production of reconstituted or recombined long-life milk.

Recombined milk is usually made from skimmed milk powder, anhydrous milk fat (butter oil), a stabiliser and good quality water. Sometimes a portion of sweet cream buttermilk powder may be used to enhance the flavour. Whole milk powder can be utilised but this is not common because of the risk of flavour deterioration during prolonged storage of the powder (271).

3.2 Raw Materials

3.2.1 Water

Water as a raw material plays a dual role in the production and processing of recombined milk, i.e., as process water and as water used for the reconstitution as such (165).

Quantitatively, water is the most important raw material used in the manufacture of reconstituted milk. Water for inclusion in a product must be free from visibly suspended matter, colour, odour, and taste, from disease-causing organisms, and from mineral or organic material dangerous to health or detrimental to flavour. To achieve, this the water should be taken from a "properly protected source" and submitted to an adequate system of purification (165). The most commonly used indicator of bacterial pollution is the organism *Escherichia coli* and the coliform group as a whole. It is generally accepted that no sample of treated water should contain *Escherichia coli* in 100 ml. Biological examination of water is of value in determining causes of objectionable tastes and odours in water and in explaining causes of clogging in distribution pipes and filters (165).

Water for Reconstitution: General Requirements

Water: Microbial Quality:Potable

From a chemical point of view, the quality and composition of dissolved substances must also be controlled, i.e., total dissolved solids, hardness, toxic substances. The "salt balance" is of particular importance in recombined long-life products where the minerals can affect the heat stability or viscosity of the product. Frequent testing of the water quality is essential, even if municipal water supplies are used.

3.2.2 Milk Powder

The packaging used for milk powder should protect the product during transportation from the manufacturer to the recombining plant so that there is a minimum loss of quality. The package must act as a barrier against moisture, oxygen and light. It must withstand physical disruption due to poor handling and give protection against insect infestation (195). Skimmed milk powder is packaged almost entirely in bags with an internal layer of polyethylene, the thickness of which should be at least 76 µm (195).

Storage Package

Skimmed milk powder will remain in good condition for long periods provided its moisture content is low (4%). At higher moisture contents, it will deteriorate with loss of solubility and the development of off-flavours. During storage, attention should be paid to temperature differences: if one side of the

Water Content

storage package (bag) is exposed to heat (for instance, sunlight) and the other is not, the moisture distribution may be affected. Although the total water content may still meet the specification, locally the limit of 4% can be exceeded.

The same requirements are applicable to whole milk powder. However, another type of deterioration occurs, even at low moisture content, caused by the oxidation of fat. As a result, the storage life is very much temperature-dependent and rather limited, normally to no more than 6 months.

Skimmed milk powders are produced to a heat classification (American Dry Milk Institute, ADMI) based on the denaturation of whey protein. The amount of non-denatured whey-protein nitrogen (WPN) is determined by:

Heat Classification

- low-heat powder: >6.0 mg undenatured WPN/g;
- medium-heat powder: 1.51 - 5.99 undenatured WPN/g;
- high-heat powder: <1.5 mg undenatured WPN/g.

Typically, low-heat or medium/low-heat powder is used in the manufacture of recombined milk as well as in filled milk in general and long-life milk in particular. Medium-heat powder is utilised for recombined, sweetened, condensed milk, and high-heat powder for recombined, evaporated or concentrated milk. Only finest grade, spray-dried skimmed milk powder manufactured to the following specifications (table 1) should be used (212):

Component	Content
Moisture	4.0%
Milk Fat	1.25%
Titrable Acidity (lactic acid)	0.10 - 0.15%
Solubility Index (ADMI)	0.5 ml
Scorched Particles (ADMI)	Disc A
Bacterial Count	<50,000/g
Coliforms	absent in 0.1 g
Yeast and Moulds	< 50/g

Table 1. *Specification: Skimmed Milk Powder*

Buttermilk powder should meet at least the following quality requirements (table 2):

Component	Content
Moisture	<4.0%
Milk Fat	>4.5%
Titrable Acidity (lactic acid)	0.8 - 0.16%
Solubility Index (ADMI)	<1.2 ml
Scorched Particles (ADMI)	Disc B or better
Bacterial Count	<50,000/g
Coliforms	absent in 0.1 g
Yeast and Moulds	< 50/g

Table 2. *Composition of Buttermilk Powder*

Milk powders with reduced lactose content (produced by enzymatic hydrolysis, mainly immobilised enzyme, lactase) have been produced and are available on the market (196).

3.2.3 Reconstitution, Recombination

Formulation and Composition

The reconstitution process consists of dissolving the skimmed milk powder in warm water, adding the milk fat, usually melted, and stabiliser. The milk is then homogenised and subsequently processed in a similar way to fresh milk. Emulsifiers such as glycerol monostearate and stabilisers such as carrageenan are often used to increase the stability of the fat dispersion. In long-life products, the addition of at least a stabiliser is essential. Typical formulae for recombined milk are given in table 3, while table 4 shows the composition of the recombined product (271).

Ingredient	Recombined Milk	Recombined Milk with Buttermilk Powder	Recombined Milk with Whole Milk Powder
Skimmed Milk Powder	89.5	80.5	
Buttermilk Powder		9.7	
Anhydrous Milk Fat	34.1	33.3	
Whole Milk Powder			123.7
Water	876.4	876.5	876.3

Table 3. *Formulation for Recombined Milk, kg/tonne*

Composition of Product	Recombined Milk	Recombined Milk with Buttermilk Powder	Recombined Milk with Whole Milk Powder
Total Solids	120	120	120
Fat	35	35	33
Non-Fat Solids	85	85	87

Table 4. *Composition of Recombined Milk (g/Kg)*

Thermo-Resistant Enzymes

As with plain white milk, thermo-resistant enzymes may also be a problem in reconstituted or recombined milk. The raw milk used for powder production as well as the pre-processing treatment of the milk before sterilisation should be done in such a way that the bacteria which produce these enzymes do not reach too high a number (271). Adequate pasteurisation of the recombined milk is essential if it is to be held for prolonged periods before UHT sterilisation.

3.2.4 Equipment Needed for Recombination (figure 2) (271)

- A vat with an efficient agitator and fitted with calibrated sight-glass or mounted on load cells so that its content can be measured, or a water flow meter which measures a calculated amount of water into the vat. The vat should be jacketed or set up so that its contents can be heated by circulation through a heat exchanger.
- A suitably designed powder-liquid blender or dumping funnel for adding and dispersing powder. The powder and water are left in the tank for 30-60 minutes and subjected to agitation (65). Different possibilities for circulation exist in order to improve the soaking process.
- Equipment for melting milk fat in 200-litre drums.
- Clarifier or duplex filters.
- Homogeniser, usually two-stage.
- Equipment for pasteurising the milk.
- Equipment for UHT sterilisation, either an indirect or direct heating system.
- Aseptic packaging machine.

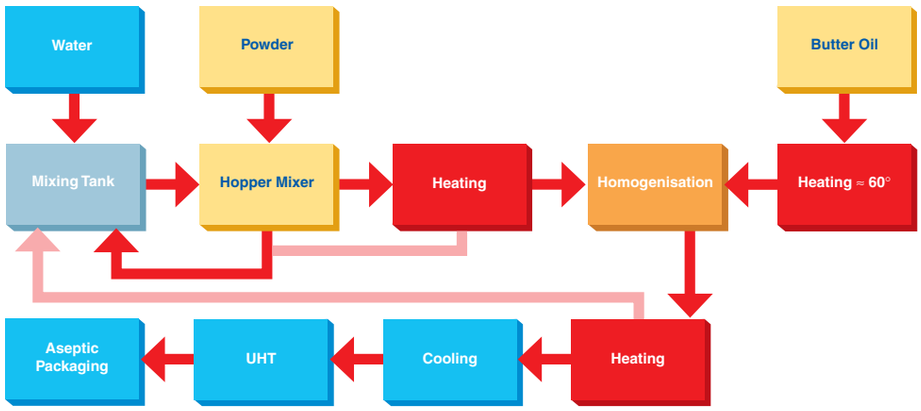


Figure 2. *Flow Chart for the Reconstitution and Production of Long-Life Milk: Production Line*

Medium to low-heat powder (ADMI) is often used with a whey-protein nitrogen content of 4 mg/g or more. The powder should have been made from milk of good bacteriological quality, preferably having a total count of not more than 5 x 10^4 organisms/ml of reconstituted milk (271).

3.3 Filled Milk

Filled milk is similar to recombined milk except that instead of milk fat, vegetable fats or oils are added. For the manufacture of filled milk, only highly refined and bleached deodorised oils of minimum peroxide value and low, free-fat acidity (acid value) complying with the specification shown in table 5 should be used. Points a-e below show the composition of filled milk (271).

Component	Content
Acid Value	0.1
Peroxide Value	1.0
Volatile Matter (105°C)	2.00 g/kg
Insoluble Impurities	0.50 g/kg
Soap	0.05 g/kg
Copper	0.10 mg/kg
Iron	0.15 mg/kg

Table 5. *Specification: Vegetable Fat*

a) Extra-grade skimmed milk powder, with a minimum content of 4.5 mg/g native whey-protein nitrogen.

b) Refined, deodorised and partially hydrogenated coconut fat which is added to the recombined skimmed milk at a temperature of 28°C.

c) Addition of vitamins A and D to give a content of 6,000 or 600 I.U. respectively in the final product.

d) Water which has been treated to a hardness of 230 ppm of $CaCO_3$.

e) Sodium bicarbonate: $NaHCO_3$ content: 0.025% of the total solids.

The procedures for manufacturing different types of filled milk are the same as for recombined milk. However, the difference in the nature of the fat may necessitate the use of different homogenising conditions and the use of emulsifiers such as glycerol monostearate or lecithin (271).

3.4 Recombined Cream

Recombined cream can be used for several purposes. Formulation and manufacture may vary according to the intended use.

Fat Content

Recombined cream can be made from skimmed milk powder, buttermilk powder and anhydrous milk fat to give a fat content ranging from 20% to 40%. For whipping cream, a fat content in the range 35-40% is essential, whereas for coffee cream and dessert cream the fat content is usually 20-25%. The addition of emulsifiers and stabilisers is necessary.

The recombining process is essentially the same as described for recombined milk. Attention should be paid to homogenisation pressures, which depend on the kind of product being produced. Recommended homogenisation pressures are (271):

- coffee cream and dessert cream: about 180 kg/cm^2 at the first stage and 30-40 kg/cm^2 at the second stage.
- whipping cream: about 70 kg/cm^2 at the first stage and 30-40 kg/cm^2 at the second stage.

Low or medium-heat (ADMI) skimmed milk powder is recommended. Typical formulations for coffee cream and whipping cream are given in table 6 (271):

Component	Coffee Cream	Whipping Cream
Skimmed Milk Powder	30.0	40.0
Buttermilk Powder	45.0	55.0
Anhydrous Milk Fat	190.0	400.0
Water	733.2	498.0
Carrageenan	0.3	3.0
Glycerol Monostearate	0.5	1.0
Tween 60	1.0	3.0

Table 6. *Formulation for Coffee Cream and Whipping Cream (kg/tonne)*

4. Fruit-Flavoured Milk

4.1 General

Heating fruit may and often does affect its flavour. Many fruits are acidic. Their addition to milk reduces protein stability which may result in problems when heating at high temperatures. With this background, the procedure shown in figure 3 of adding liquid and dissolved fruit components aseptically to the previously UHT-sterilised milk has been suggested (162).

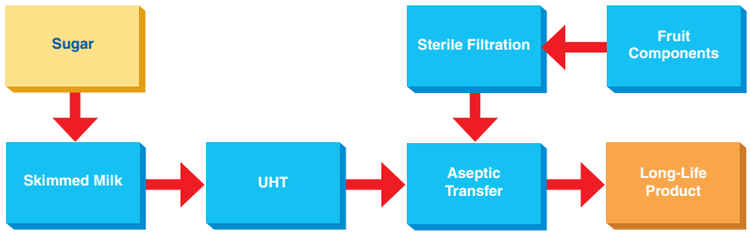


Figure 3. *Addition of Sensitive Fruit Components in the Production of Fruit-Flavoured, Long-Life Milk*

For example 350 kg of concentrated sucrose syrup and four litres of natural red colouring are added to 5,000 litres of high quality skimmed milk. The mixture is UHT-treated by direct steam injection at 148-150°C and homogenised aseptically at low pressure (25 kg/cm^2). Natural aroma is sterilised by filtration and injected aseptically and continuously into the stream of milk passing into the aseptic filling unit, as illustrated in figure 3.

A typical formulation for a fruit-flavoured milk drink is given in table 7.

Fruit-Flavoured Milk: Composition

Component	Content
Milk	56.8%
Fruit Juice	38.0%
Sugar	5.0%
Stabiliser	0.2%

Table 7. *Composition of Fruit-Flavoured Milk*

5. Cream

5.1 Cream-Based Liqueurs

One of the fastest growing markets for cream is in the form of cream-based liqueurs (38, 175). Homogenisation of the cream is essential in order to prevent fat separation during storage. Rather severe homogenisation conditions were found to be necessary: 96% of the fat globules had diameters of less than 0.8 µm. An emulsifier with sodium caseinate was found to be satisfactory. A ratio of fat to caseinate of approximately 4 to 1 was optimal. A disadvantage of the caseinate is its instability at low pH.

The most desirable range of milk fat-content was 12-16%. Addition of carbohydrate to the liqueur performs two obvious functions, i.e., it determines the texture and sweetness, but it also affects the sensation of alcoholic strength in the mouth. An addition of 15-20% sugar was found optimal. Table 8 shows the typical composition of a cream-based liqueur.

Component	Concentration
Milk Fat	12 - 16%
Added Sugars	15 - 20%
Sodium Caseinate	2.6 - 3.5%
Non-Fat Milk Solids	1.0 - 1.4%
Total Solids	35 - 40%
Ethanol	14%
Water	46 - 51%

Table 8. *Typical Composition of a Cream-Based Liqueur (38)*

5.2 Coffee Cream

One of the major problems with long-life coffee cream is flocculation which occurs when cream is mixed with hot coffee. The graph below (figure 4) shows the relationship between two stage homogenisation pressures and flocculation of 12% coffee cream (124).

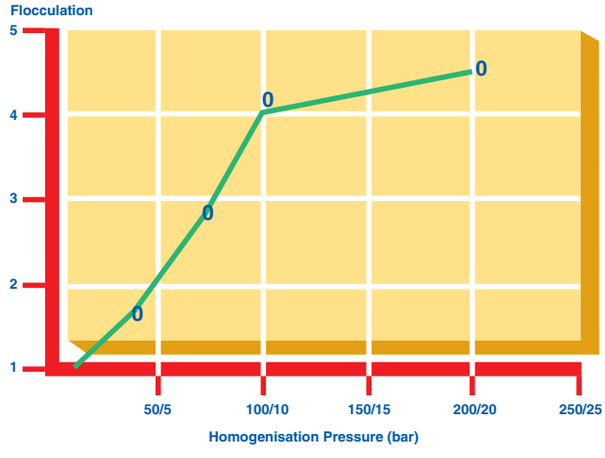


Figure 4. *Coffee Cream: Flocculation and Homogenisation*

Homogenisation of long-life coffee cream is also necessary in order to prevent fat separation during storage. As can be seen from the above graph (figure 4), flocculation increases with increasing homogenisation pressure. The following procedure is recommended for the production of long-life coffee cream (124):

a) homogenisation to obtain small fat globules and aggregation of protein on to the fat globule membranes;

b) pre-heating to denature whey proteins which also aggregate with the fat globules;

c) UHT heating to obtain a commercially sterile product;

d) aseptic homogenisation to split the fat protein aggregates: fat globules of small size are formed, the surfaces of which are covered with denatured milk protein.

5.3 Whipping Cream

Long-life whipping cream is one of the most difficult UHT products to make. It has, however, been produced at both direct and indirect working UHT plants. Homogenising whipping cream with a fat content of 30% is problematic. Sufficient foam (whipping ability) can only be obtained if the size of the fat globule aggregates is between 15 and 20 µm. If the fat globule size is > 20 µm, a high foam stability results but the increase in volume is very limited. The following recommendations are given for the production of long-life whipping cream (154) and are schematically shown in figure 5:

Foam Stability

a) the cream should be obtained from raw milk which has been stored for a short period of time only;

b) low temperature at separation (< 45°C, preferably 10°C);

c) ripening of the cream to a pH of 6.4 to 6.3 before UHT treatment by the addition of a starter culture (rarely if ever done) or, preferably, by adding "acid cream buttermilk powder" in a ratio of 100:15;

d) rapid cooling of the heated cream;

e) homogenisation (28 - 30 kg/cm^2 at 60 - 70°C) to obtain a fat aggregate size of 15 - 20 µm;

f) the addition of 0.5% "acid cream buttermilk powder".

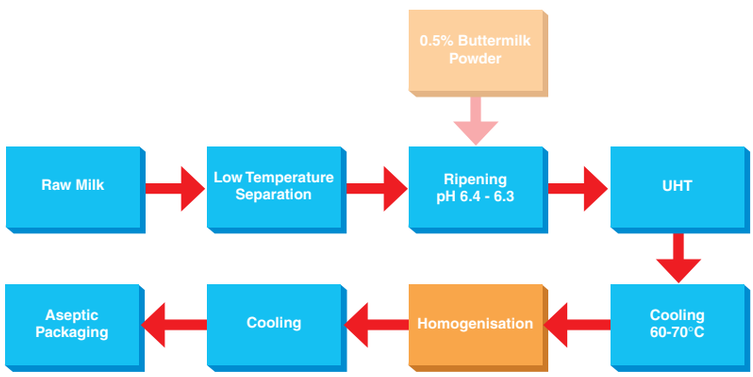


Figure 5. *Production of Long-Life Whipping Cream*

Homogenisation conditions in the manufacture of UHT whipping cream are likely to be of major importance in controlling the properties of the product. Conflict is to be expected between the need for adequate homogenisation to prevent fat separation and the effect of homogenisation on whipping properties. In a pilot plant system (steam injection), optimal homogenisation conditions for a 36% long-life whipping cream were found to be two-stage homogenisation at 30°C and a pressure of 300 + 100 lb/sq inch (40 + 14 kg/cm^2). An increase of 20% in available calcium in the cream markedly improved whipping properties but increased the rate of fat separation (233).

Homogenisation Fat Separation

Whipping Ability

A cream of 40% fat was UHT-sterilised at 130-135°C with a holding time of 15 seconds (162). The heat treatment did not affect whipping ability. Prolonged storage, however, resulted in considerable creaming of the product. Creaming can be prevented by homogenisation, but this interferes with the whipping capacity.

30% cream was heated to 145°C and subsequently homogenised in two stages at 70 + 7 bar and at 70°C. The product was stable for at least six weeks at room temperature (116). Homogenisation pressures below 50 bar resulted in poor whipping ability while high pressures (100 bar) led to a product of too high a viscosity (143). An addition of 0.02% of carrageenan is recommended to improve stability in the whipped cream (143).

A whipping cream having low viscosity, excellent whipping ability and especially suitable for UHT treatment had the composition shown in table 9 (29).

Ingredient	Composition
Dairy Cream (35-40%)	99.2 %
Lactodan P 22	0.6 %
Sobalg FD 155	0.05 %
Sugar	0.1 %

Table 9. *Composition of a Stabilised Long-Life Whipping Cream*

Fat Crystallisation before Whipping!

It is recommended that the cream be stored under refrigeration. At least before whipping, the cream should be cooled down to ca. 5°C to achieve the necessary fat crystallisation.

A whippable cream product of only 20-25% fat was obtained by diluting 30-35% cream with sweet cream buttermilk. The product could be UHT treated and aseptically packaged. The storage life of the product, determined by accelerated storage tests (the suitability of which is debatable), was at least six weeks. The product could be whipped in about three minutes.

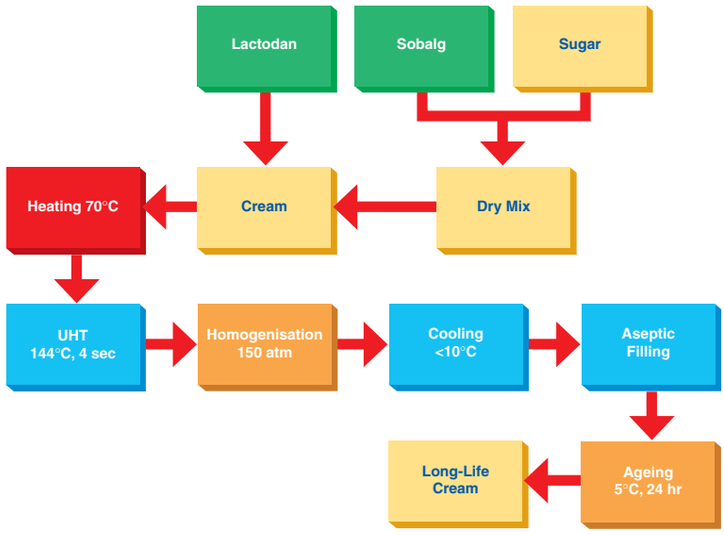


Figure 6. *Flow Chart: Long-Life Whipping Cream with Additives*

A low-fat, long-life whipping cream was developed with good whipping ability (141).

The process parameters were as follows (figure 7):

a) gelatine (0.05 - 0.15%) is mixed at a temperature of 15°C with cream and allowed to swell for 15 minutes;

b) skimmed milk powder or buttermilk powder (about 2%) is dissolved under mild heating (35°C, ca. 15 minutes);

c) both fractions are mixed and stored at 20-25°C. The fat content is adjusted to 22-24%. After ripening, the pH should not exceed 6.5;

d) after UHT treatment, sterile filtered rennet (strength of the solution: 1:10,000) is added in an amount of 0.1%. The enzyme is allowed to act for 20-40 minutes at 30-35°C, followed by cooling (ca. 5°C) and aseptic filling.

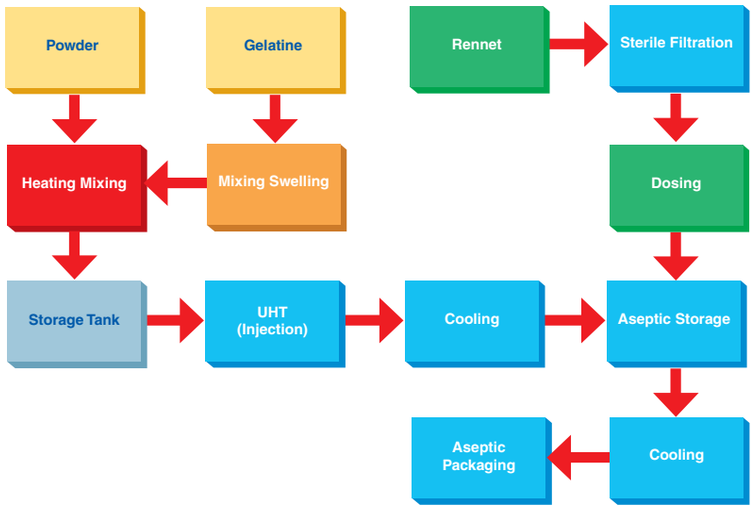


Figure 7. *Flow Chart: Whipping Cream Produced with the Addition of Rennet*

6. Whey-Based Products

Whey, a by-product from the manufacture of cheese, has long been recognised as a health food (16). Refreshing and nutritious still drinks can be produced based on whey and whey concentrate in combination with fruit. The following composition (table 10) has been recommended (28):

Ingredient	0.7% Protein	3.0% Protein
Fresh Whey	75.50	
Whey Concentrate		8.50
Pectin	0.10	0.30
Sugar	6.00	6.00
Citric Acid	0.65	0.65
Tri Sodium Citrate	0.55	0.55
Fruit Juice (65 Brix)	15.00	15.00
Water		69.40

Table 10. *Composition of a Whey-Based Drink*

The following production conditions (figure 8) resulted in an acceptable long-life product:

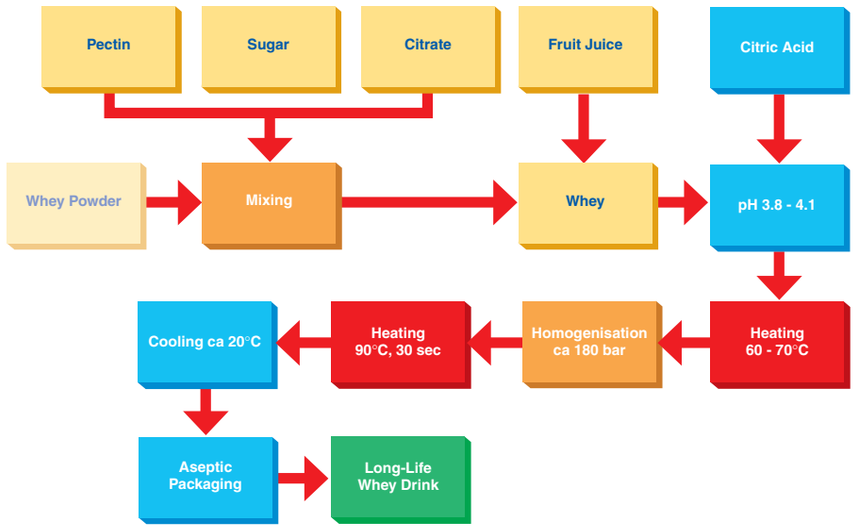


Figure 8. *Flow Chart: Production of a Long-Life, Whey-Based Drink*

When fresh whey is used the protein content can be increased by evaporating part of the water or by adding whey powder.

The use of mango in the formulation of whey-based drinks seems to cover the typical whey flavour. The long-life product shown in table 11 had an acceptable flavour and was successfully marketed.

Production Conditions for a Whey-Based Mango Drink

Natural whey having a pH of ~3.9 and a dry matter content of ~5.4% was used without any additional treatment. The product was homogenised at a temperature of 60-65° C and a total pressure of 200 kg/cm^2 (150 + 50 kg/cm^2). Pasteurisation conditions were 88-90°C with a holding time of 15 seconds. The whey drink had a shelf life of about 6 months at ambient temperature (personal communication).

Component	Concentration
Whey	88.96 %
Sugar	8.90 %
Mango Concentrate	1.78 %
Stabiliser (Frimulsion)	0.36 %

Table 11. *Composition of a Whey-Based Long-Life Product Containing Mango*

7. Custards and Desserts

7.1 General

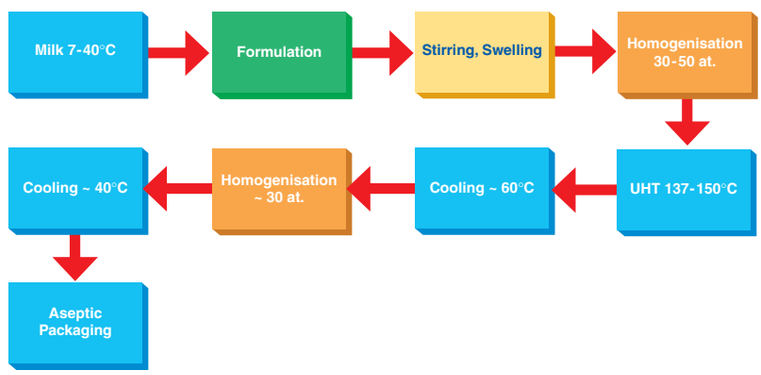


Figure 9. *Flow Chart: Production of a Long-Life Dessert, General Procedure*

Custard powder and sugar are added to milk and stirred continuously at a temperature of about 20°C. The mixture is then pumped through a pasteuriser where it is heated to about 85°C. Subsequently, it is sterilised at 140°C followed by aseptic filling (162).

Thickening Agents

The type of thickening agent, and the jellying agent and their respective levels, determine the rheological properties of the dessert. Formulations containing 3.1% of selected modified starches and 0.2% of selected carrageenan resulted in products of the desired quality. The principle process parameters which influence quality properties are filling temperature, homogenisation pressure and heating intensity (182).

Manufacturing conditions in general are as illustrated in figure 9 (210).

In table 12, a general formulation for a long-life dessert is given.

Component	Content
Milk	1,000 litres
Amylopectin Food Starch	~50 kg
Locust Bean Gum	2 - 3 kg
Phosphates	0.2 - 1.0 kg
Gelling Agents	1.5 - 3.0 kg
Emulsifiers	0.2 - 1.0 kg
Fat	20 - 50 kg
Skimmed Milk Powder	20 - 30 kg
Egg Powder	5 - 7 kg
Sucrose	~140 kg
Dextrose	~30 kg
Malto Dextrin	~30 kg
Colour, Flavour	as desired

Table 12. *General Formulation for a Long-Life Custard (210)*

7.2 Vanilla Custard

Composition

In the manufacture of vanilla custard, 103 kg of milk standardised to 3.00% butterfat is mixed with 4 kg of custard powder (starch) and 8 kg of sugar to obtain 115 kg of mixture containing 3.09 kg of butterfat. Processing is as shown above.

7.3 Chocolate Custard

Composition

103 kg of milk (3% fat), 3.5 kg of custard powder (starch), 2 kg of cocoa powder and 9 kg of sugar were used in the formulation (162).

Special carrageenans were used as jellifying agents. Care should be given to the quality of the cocoa powder used. For it to be suitable for UHT treatment (requiring quick heat transfer because of rapid heating, short holding, followed by rapid cooling), the powder should meet the following specification:

a) highly defatted: oil content: 10-12 %;

b) instant type: lecithin-treated;

c) low shell content: ~ 4 % or less;

d) small particle size; table 13 contains a specification of particle size which was found to be adequate for the production of long-life products (UHT treatment) in general.

If such powders are not available, more intensive soaking procedures must be applied.

A typical flow chart for the production of a chocolate pudding is shown in figure 10.

Size (μ)	Content
< 10	25.0 %
> 10 - < 20	35.0 %
> 20 - < 30	20.0 %
> 30 - < 40	11.0 %
> 40 - < 60	5.0 %
> 60 - < 90	3.0 %
> 90 - < 200	0.6 %

Table 13. *Cocoa Powder Specification: UHT Treatment, Particle Size*

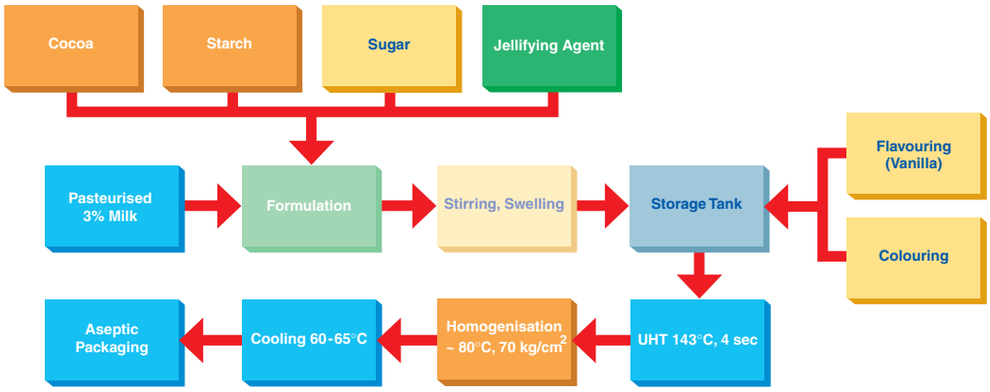


Figure 10. *Flow Chart: Production of a Long-Life Chocolate Dessert*

Filling Temperature

High filling temperatures reduce the safety margins of sealing. The filling temperature of 60-65°C shown in figure 10 depends on the starches used in the formulation. During filling, the product must be in a liquid state: low temperature and late setting starches must be used in the formulation.

8. Formulations For Baby Food

Long-life milk (direct UHT) has been compared to fresh milk and retorted milk for feeding babies. The following advantages have been found (68):

a) it is free from living microorganisms, and thus hygienically safe (169);

b) steam injection heating results in minor losses of nutrients;

c) it is safe to use since "overdosing" often connected with powder products is impossible (169);

d) it is not dependent on the quality of water, which is not always satisfactory;

e) it has six months shelf life; and

f) it requires little effort in application, i.e., it is convenient to handle.

The microbiological benefits of long-life formulations for baby food (adapted, ready-to-use) were confirmed (see table 14) (48).

Product	0 hr	6 hr	12 hr	24 hr
1	8,000/ml	128,000/ml	500,000/ml	1,200,000/ml
2	28,000/ml	420,000/ml	1,400,000/ml	3,100,000/ml
3	32,000/ml	400,000/ml	1,000,000/ml	2,400,000/ml
4	10,000/ml	180,000/ml	520,000/ml	1,800,000/ml
Long Life*	<100/ml	200/ml	680/ml	2,400/ml
Long Life**	0/ml	0/ml	0/ml	100/ml

Home Conditions (68), * = filled into baby food bottles, ** = opened package.

Table 14. *Total Bacterial Counts of Reconstituted Milk After Being Filled into Baby food Bottles*

Microbiological Safety in Production!

Because of the sensitivity of such products, the consequences of failure are very severe. Special measures must be taken to ensure safety. Spoilage rates must be kept to an absolute minimum. This requires giving particular attention to production procedures, quality control and quality assurance.

The composition of long-life milk adapted as a formulation for baby food is given in table 15.

Formulations for baby food can be the only source of nutrition for babies. Consequently, they must provide all the nutrients needed in the necessary amount and the correct proportion. The vitamin content of the product is given in table 16.

Component	Content
Protein	1.90 g
Total Carbohydrates	8.20 g
Lactose	3.50 g
Saccharose	3.00 g
Corn Starch	1.70 g
Total Fat	3.60 g
Butterfat	1.20 g
Mixed Fat	2.40 g
Minerals	0.45 g
Calories	74/100 ml

Table 15. *Composition of an Adapted Long-Life Formulation For Baby Food*

Vitamin	Content
A	2,000 IU/l
B_6	2 mg/l
C	80 mg/l
D	400 IU/l
E	5 mg/l

Table 16. *The Vitamin Content of a Long-Life Formulation For Baby Food*

9. Vegetable Juice

Low-Acid or High-Acid?

Natural vegetable juice usually has a pH value of > 4.6. Consequently, spoilage by bacterial endospores is a possibility (107). Process conditions have to be adjusted accordingly. They can either be treated as low-acid products which require UHT treatment at high temperatures, usually > 130°C, or they must be acidified prior to processing, typically by the addition of citric acid. If this is done, the process step of acidification becomes a critical control point. At the present time, a number of different long-life vegetable juices are available on the market.

10. Wine

10.1 General

Large-scale continuous manufacturing procedures of wine have evolved. Since grapes represent a natural product which is highly susceptible to chemical, biochemical and microbiological changes, rapid processing is of the utmost importance.

10.2 Manufacture of White Wine

The flow chart shown in figure 11 illustrates a typical production line for wine.

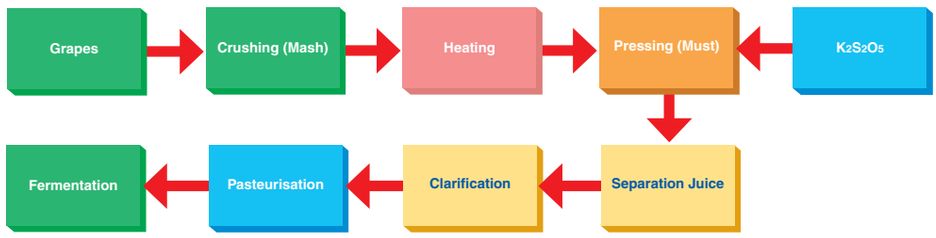


Figure 11. *Flow Chart: Production Line for Wine*

After crushing, sulphurous acid (as $K_2S_2O_5$) is added as an anti-oxidant and a means of preservation. Typical pasteurisation conditions are two minutes at 87°C. Depending on the kind of wine and in combination with aseptic filling procedures, pasteurisation at 63°C for 8 seconds and 90°C for 15 seconds has also been tried (107).

Heat treatment of the mash prior to pressing is often carried out. Such a step offers a number of advantages:

a) the pressing time is reduced (table 17);

b) protein stability is increased;

c) an increased yield is obtained because of reduced viscosity due to the disintegration of pectins. The addition of pectinase in combination with heating to 45°C for two hours further reduces the pressing time to 34 minutes.

Mash	Pressing Time
Pressed Immediately	85 min
Pressed after 3 Days	43 min
Stemmed - Pressed Immediately	83 min
Stemmed - Heated 80°C, 2 min	50 min
Stemmed - Heated 45°C, 2 hours	52 min

***Table 17**. Effect of Heating on Pressing Time*

10.3 Manufacture of Red Wine

Traditional Production

An important quality characteristic of red wine is the colour. The colouring matter (anthocyans) is located in small cells and can only dissolve when the cell plasma has been destroyed. In the traditional method of manufacture, the red grapes are crushed, sulphurous acid is added, and the mash is fermented with air admitted over a period of 3-10 days. The must containing alcohol and obtained by pressing undergoes secondary fermentation to red wine. There are two main shortcomings to this procedure:

a) the development of colour varies;

b) the taste and colour are affected by the release of tannins.

These shortcomings have been overcome by modern processing techniques, as shown in figure 12.

Improved Quality

The mash is heated to 75-85°C for a few minutes, cooled down to 45-55°C, pectinase is added and allowed to act for 1-2 hours.

Otherwise, the process is the same as that used for white wines.

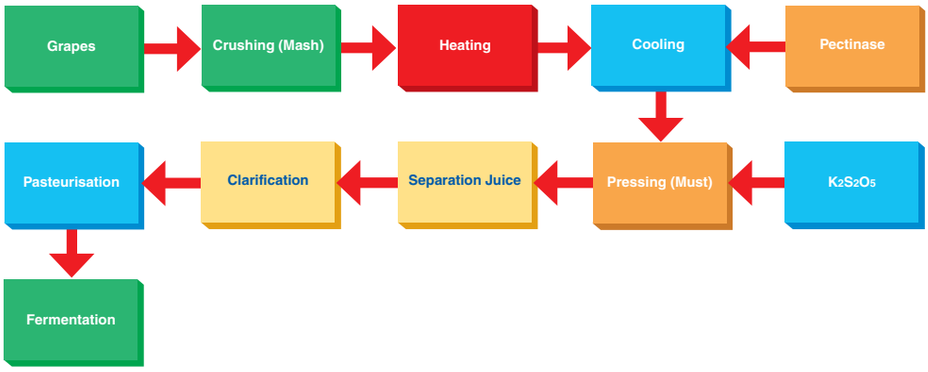


***Figure 12.** Flow Chart: Modern Production of Red Wine*

Wine is also sensitive to oxidative change, both in flavour and in colour. In order to reduce dissolved oxygen and to minimise oxidation, an atmosphere of, and flushing with, sterile nitrogen was used. The processing conditions for the wine tested are shown in figure 13 (35):

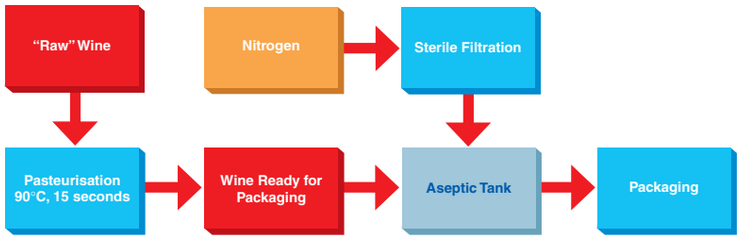


Figure 13. *Flow Chart: Reduction of Oxidation*

The test wine was filled into an aseptic tank and flushed with sterile nitrogen for 24 hours before filling (figure 13). The composition of the wine is shown in table 18.

If pasteurisation is not required, sterile filtration is one of the methods which can be used for the sterilisation of wine (figure 14) (52).

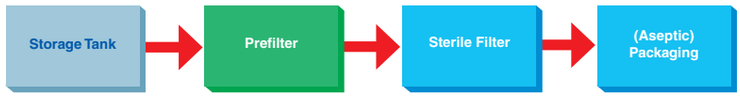


Figure 14. *Flow Chart: Sterilisation of Wine by Filtration*

Component	Content
Alcohol	79.7 g/l
Total Acid	8.9 g/l
pH	3.23
Free SO_2	69 mg/l
Total SO_2	177 mg/l
Volatile Acid	0.44 g/l
CO_2	0.80 g/l
Ethanal	61 mg/l
Sugar	24 g/l

Table 18. *Composition of the Wine*

Comparison of wine filled in a number of different containers is presented in table 19 (35). The results show the suitability of aseptic technology as a packaging system for table wine.

Container	Volume	6 months	12 months
Glass	700 ml	2.32	1.68
Can	330 ml	1.60	0.62
Can	330 ml	1.22	0.57
Can	250 ml	1.49	0.77
Tetra Brik	200 ml	2.29	2.34
PET	250 ml	2.08	2.00
PVC	500 ml	2.47	1.81
PVC	500 ml	2.28	2.29

Table 19. *Sensoric Score During Storage*

White and red table wines were filled into glass bottles and paper-based plastic laminates (Tetra Brik cartons). After storage for one year at ambient temperature and at 37°C, the quality of the wine had been affected by the storage conditions. However, there was no difference in quality specific to the type of package (3).

11. Water

11.1 General

Total Counts in Packaged Water

If untreated packaged water is stored, rapid multiplication of bacteria may take place. Even under optimal conditions, pure drinking water in packages will not retain an acceptable bacteriological quality for more than one week at ambient temperature (228). Samples of packaged mineral water bought in retail outlets showed varying total bacterial counts as shown in table 20 (62).

Total counts of up to 3,000/ml were found in commercial samples of untreated water intended for household use (102). The bacterial growth could be controlled by the aseptic packaging of heat sterilised water (62, 102).

Because of the low-flavour profile of mineral water, the product is very sensitive to flavour changes induced by either processing or packaging. If plastic materials are used, special packaging material qualities are recommended.

Kind of Container	Volume	Total Count/ml
Glass Bottle	0.50 l	780/ml
Plastic Pouch	1.00 l	50,000/ml
Plastic Pouch	0.35 l	1,000/ml
Gable Top	1.00 l	5,600/ml
Gable Top	2.00 l	6,100/ml
Gable Top	2.00 l	25,000/ml
Gable Top	1.00 l	10,000/ml
Gable Top	1.00 l	310/ml

Table 20. *Total Counts in Packaged Mineral Water*

11.2 Water Treatment

Treatment of water with ozone (O_3) in amounts of 0.3 to 1 mg/l kills the micro-organisms present in the water within 6-8 hours. As shown in figure 15, ultra-violet irradiation can also be used in combination with filtration (102):

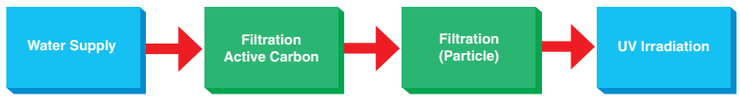


Figure 15. *Flow Chart: Treatment of Water*

UV-Treatment of Water

Water is filtered and, in a thin layer, passes a UV lamp. Attention has to be paid to the removal of deposits which may form on the UV lamp (102).

Another possibility is sterile filtration through filters with a pore size of 0.2-0.15 µm. Local legislation has to be observed carefully.

In many countries, no treatment or only specified procedures are permitted if a water is marketed with the label of "natural mineral water" (figure 16).

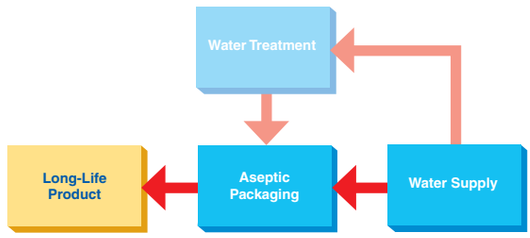


Figure 16. *Flow Chart: Long-Life (Mineral) Water*

Aseptically-filled natural mineral water has been successfully marketed in a number of countries.

12. Liquid Eggs

Handling costs, loss of functionality, and inconvenience associated with frozen liquid whole eggs have stimulated interest in a refrigerated alternative. To maintain adequate shelf life at refrigeration temperatures, liquid eggs were ultra-pasteurised and aseptically packaged. Holding times varied from 3 to 192 seconds, and temperatures between 63.7°C and 72.3°C (Tetra Pak technology: 70°C for 90 seconds). A refrigerated shelf life of 3 months at 4°C was obtained (135). Figure 17 shows a flow chart for the process.

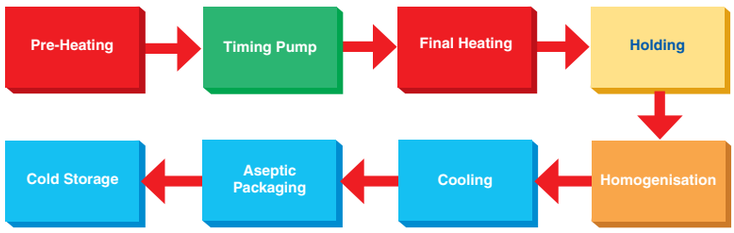


Figure 17. *Flow Chart: Liquid Eggs*

Though not really a long-life product since the heat treatment is not sufficient to obtain commercial sterility and, consequently, refrigerated storage is required, the interest in aseptically packed liquid eggs is none the less increasing.

16. Long-Life Acidified Products

Summary

Acidified products are produced from low-acid raw materials which, prior to final processing, are rendered acidic either by the addition of organic acids, usually citric acid, or by biological acidification by means of a starter culture. Processing conditions are about the same as those for high-acid food products. Long-life acidified drinks are gaining in popularity. Some long-life vegetable juices are also very popular. In the following chapter, a short description of some milk-based, acidified, long-life drinks is presented with regard to the composition and processing of the products.

1. General

The acidified products of interest are mainly cultured products based on milk or soya beans and some vegetable juices. Acidified products originally have a pH value in the low-acid food range (i.e., above 4.6). As shown in figure 1, their pH is lowered either by the addition of acid (chemically acidified) or by the growth of microorganisms (acidified by the addition of starter cultures). The acidification process is a critical control step and its process parameters must be controlled accordingly.

Acidification is a Critical Control Point!

Pre-processing is necessary to achieve the correct pH value and often desirable to increase safety and product quality. This is particularly true for yoghurt-type products. Processing parameters necessary for achieving commercial sterility depend on the product and, most of all, its pH. In general, a temperature of 70-80°C and a holding time of 30-60 seconds are sufficient to eliminate lactic acid bacteria as well as yeast and moulds. The heat resistance of thermophilic lactic acid bacteria was found to be 10-15°C higher than that of mesophilic lactic acid bacteria (207). Often such acidified products are heat-treated at a higher temperature using the same processing conditions as those applied to high-acid products, typically in the temperature range of 85-95°C with holding times between 15 and 30 seconds.

Processing Conditions

In the production of acidified products, the acidification process is a critical control step. Care has to be taken to achieve the correct as well as an evenly distributed pH. Process procedures must be established to ensure that this is accomplished.

The purpose of heat-treating cultured milk products is to prolong their shelf life and, at the same time, minimise any negative influence in their quality.

Heat and acidity favour syneresis of the casein resulting in separation of the whey. Contraction of acid-precipitated casein is influenced by the following factors (207):

- the pH of the product: the pH should be adjusted with an accuracy of 0.05 units and should be in the range of 4.40 to 4.10;
- the content of fat, protein, and sugar: higher fat and sugar contents are favourable while the opposite is true for casein;
- the temperature and duration of heating the milk before fermentation: an increasing load of heat stabilises the end product (reduces syneresis);
- proteolysis;
- the type of hydrocolloid (stabiliser) used: hydrocolloids are indispensable in the manufacture of long-life, acidified milk products. Starch, gelatine, or pectin may be added during the preparation of the milk before or after fermentation, but before the final heat treatment;
- the heating temperature and holding time of the finished product.

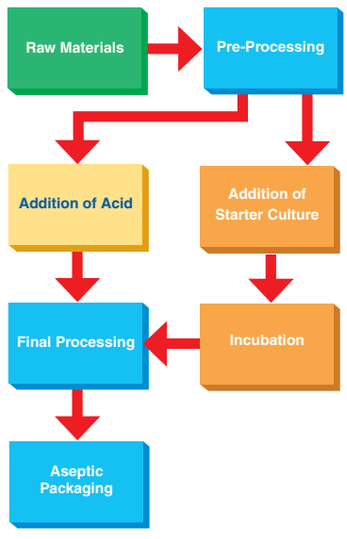


Figure 1. *Flow Chart: Acidification Process*

As shown in table 1, the processing conditions necessary to achieve commercial sterility depend on the pH of the product (60, 231). The time-temperature combinations stated in table 1 are necessary for in-container sterilisation and are much too high for in-flow treatment. However, the values stated in the table clearly indicate the importance of the pH.

pH value	40 sec at	20 min at
4.6	140°C	115°C
4.5	130°C	110°C
4.4	120°C	105°C
4.3	110°C	100°C
4.2	100°C	95°C
4.1	98°C	90°C
4.0	94°C	85°C
3.9	90°C	75°C

Table 1. *Acidified Milk Products: pH and Temperature Treatment Necessary to Achieve Commercial Sterility*

A representative flow chart for the production of acidified milk-based products is given in figure 2. Milk is standardised and homogenised, followed by the addition of the starter culture and proper incubation if microbiological acidification is applied. Stabiliser and fruit, if desired, and/or acid, in chemical acidification, are added.

Acidified, Milk-Based, Long-Life Products

Some time for swelling is allowed for. The product is subsequently pasteurised using a suitable time-temperature combination. After aseptic packaging, a long-life product is obtained.

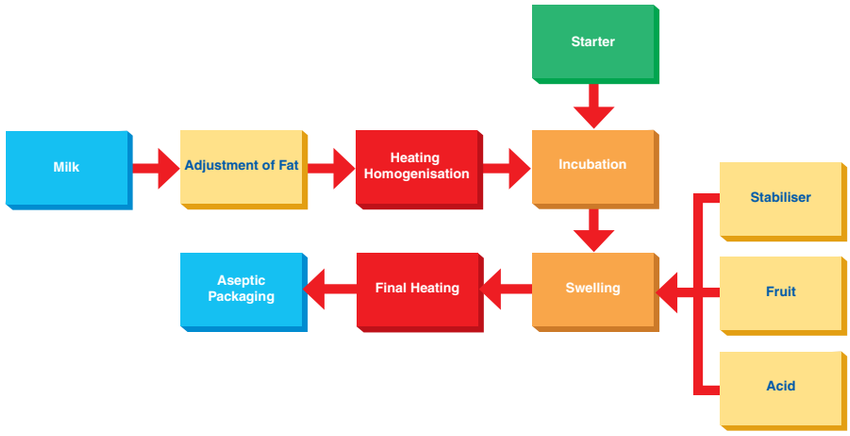


Figure 2. *Flow Chart: Production of Acidified Milk-Based Products*

2. Yoghurt Production

Raw Materials

As the properties of the yoghurt influence to a great extent the quality of the final, long-life yoghurt drink, it is necessary to consider its production carefully in order to obtain an optimal result. The raw material is either fresh skimmed milk, whole milk or skimmed milk with extra solids added.

Furthermore, reconstituted milk - either in the form of powder or condensed milk - may be used. The content of milk solids non-fat (MSNF) may vary between 9% and 20%. A higher content of milk solids will cause a reduction in the size of the casein particles after fermentation resulting in a drink with higher viscosity.

The heat treatment leads to denaturation of the whey proteins: more water can be held between the protein micelles resulting in less whey separation. Fermentation must be carried out down to a pH of ~ 4.0. At a lower pH, the yoghurt becomes less stable.

A general flow chart for yoghurt production is given in figure 3 (26).

Shelf-stable drinking “yoghurts” with a claimed shelf life of up to 6 months at room temperature have been reported (180). The product was prepared from 84 kg of skimmed milk, 12 kg of fruit juice with 50% sugar, and 0.35 kg of Mexpectin RS 450 dispersed in a 65% sugar solution. The final composition of the product is shown in table 2.

Component	Content
Milk Solids, Non-fat	7.6 %
Sugar	8.0 %
Mexpectin	0.35 %

Table 2. *Product Composition*

Figure 3. *Flow Chart: Typical Yoghurt Production*

After mixing, the product was homogenised, pasteurised at a temperature of at least 73°C with a holding time of 20 seconds, cooled and packaged aseptically (figure 4).

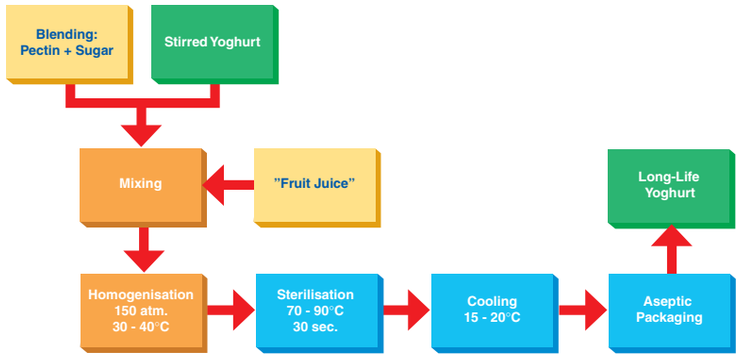


Figure 4. *Flow Chart: Production of Long-Life Yoghurt*

Yoghurt drinks stabilised with pectin may be sterilised to extend the shelf life to 6 months or more. Composition and viscosity of the drink may be varied in accordance with market requirements, but generally the products contain the following ingredients (26):

a) yoghurt: 30 to 80%;

b) sugar: approximately 8%;

c) pectin: 0.25% to 0.6%;

d) fruit juice, fruit concentrate and/or fruit flavour: 0.5 to 10%;

e) water: to 100%.

Homogenisation

Homogenisation disintegrates the yoghurt into casein particles, brings the pectin into solution, and ensures optimum contact between the pectin and the surface of the separated casein particles. Optimum pressure varies with the actual homogeniser used, but as a starting point 150 atm (2100 psi) is recommended. By raising and lowering the pressure 50 atm and observing the influence on stability of the final drink, the actual optimum pressure can be determined. Homogenisation is made at the fermentation temperature. Increasing the homogenisation temperature results in an end product of inferior stability.

Another yoghurt type produced from UHT-sterilised milk and packaged under aseptic conditions contained a living starter culture (figure 5) and had an increased shelf life, but it had to be stored at relatively low temperatures (below 10°C). Because of the requirement for refrigeration, it is unlikely that such a product will be successful in the market. In many countries, a product labelled as "Yoghurt" must contain living starter culture bacteria, a requirement which is met by this product.

The following processing stages are applied (figure 5) (236):

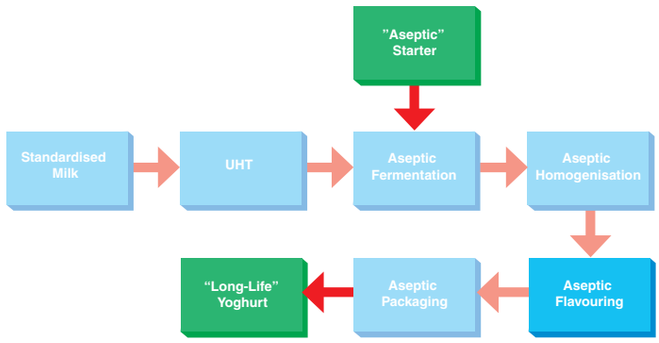


Figure 5. *Flow Chart: "Long-Life" Yoghurt Containing Living Bacteria*

This is, however, a very demanding and expensive technology which is rarely used in practice.

17. Long-Life, High-Acid Products

Summary

Long-Life fruit juices, nectars and fruit juice drinks have become very popular products. They are considered safe from a public health point of view. The main spoilage organisms are yeast and moulds, though some bacteria (primarily *Lactobacillus* and *Streptococcus*) can multiply at low pH values (i.e., below 4.6) and are thus able to damage the product.

Because of the restricted number of possible spoilage organisms, the processing and aseptic packaging of high-acid food commodities is rather simple. This may actually lead to problems: over-confidence results in negligence!

Some processing and aseptic packaging systems have been specifically developed for handling high-acid or acidified beverages, their use being restricted to such products. Some fruit juices, nectars and fruit juice drinks, particularly those prepared from or with citrus fruit, are sensitive to heat-induced flavour changes. Losses of vitamin C also occur in heat treatment. Consequently, the heat load should be kept as low as possible. On the other hand, enzymes, especially pectinase, need to be inactivated and this requires a certain minimum heat treatment.

In the discussion on "Long-life, High-Acid Products", the handbook *Fruit Processing Technology* (Chem. Chin G., Ag Science, 1992) has been a valuable help. It has been used extensively in the section on "Fruit Juices and By-Products".

1. General

High-acid products have a pH value of 4.6 (4.5) or lower. They include:

a) fruit juices;

b) nectars;

c) fruit juice drinks, etc.

Why Different Processing Parameters?

Often, but not always, aseptic packaging conditions are the same for high and low-acid food products. However, the processing parameters are different for four main reasons:

- bacterial spores do not germinate at pH values of 4.6 or below and, consequently, they do not need to be killed since their presence does not interfere with the concept of commercial sterility – they cannot multiply;
- the sterilisation efficiency of any heat treatment increases with a decreasing pH value: less heat is required to achieve the same killing rate;
- pathogenic bacteria cannot multiply at low pH values and, consequently, high-acid products are regarded as safe from the point of view of public health; and
- most fruit juices and fruit juice drinks are sensitive to heat-induced changes in quality: mild processing conditions are preferred. This is particularly true for citrus juices.

Typical Processing Conditions

Fruit juices are usually processed at a temperature below 100°C. Some manufacturers of processing equipment offer special, simplified "sterilisers" specifically designed to operate at temperatures below 100°C. Other equipment features automatic switch-over from low-acid to high-acid processing conditions. Typical parameters for processing shelf-stable, high-acid products are temperatures in the range of 85-95°C with holding times varying between 8 and 40 seconds (107, 174). Some fruit products, especially tomato-based products, may require higher processing temperatures (i.e., above 100°C).

Because of aroma losses, direct heating (expansion cooling) is unsuitable for processing fruit juices, nectars and fruit juice drinks. Often deaeration combined with aroma recovery is used to remove foam and to reduce dissolved oxygen. A typical processing line for the production of long-life juice is shown in figure 1.

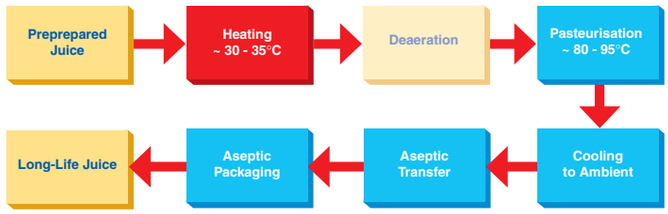


Figure 1. *Flow Chart: Processing and Packaging Line for Long-Life, High-Acid Products*

2. Microbiology of High-Acid Food

Fruit and berries are protected against microorganisms by the epidermis and often, in addition, by a waxy layer called cuticula. If the protective layers are damaged mechanically or by birds or insects, microbial spoilage may take place rather quickly. Because of the low pH in the material, mainly yeast and mould will grow but also some bacteria will multiply and cause product spoilage.

Microorganisms enter the processing plant on the surface of fruit having originated in soil, untreated water, dusty air and decomposed fruit (238). In general, wild fruit and berries are less contaminated with microorganisms than products grown in orchards.

Control of Raw Materials

In a fruit processing operation, it is important to control the raw materials carefully and to reject products that are noticeably damaged. To a certain extent, mechanical control devices are available but usually a visual inspection is practised in combination with manual sorting.

The microflora vary from one orchard to another. It is therefore not possible to give any generally valid information about the microbiology of the raw material. In addition to what is already present at harvest, it has been found that the general standard of hygiene during transportation to the factories may be the most important single factor for the microbiological quality of the fruit delivered.

Prevention of the Growth of Microorganisms

If a lengthy period of time elapses between harvest and final processing it is necessary to prevent the development of yeast and mould and reduce chemical changes. Storage at low temperature has only limited value because many product-spoiling moulds are able to grow even below 0°C. Freezing or storage in controlled atmospheric conditions (CO_2, ethylene oxide, etc.,) or storage of pulp preserved with SO_2, are examples of storage methods.

When the raw material is to be processed, it is just as important for high-acid products as it is for milk that hygiene be kept well under control.

2.1 Bacteria

Coliform bacteria and related types (e.g., *Ervinia*) are frequently present on or in oranges before they are harvested (236). Fruit-flavoured soft drinks (pH 3) were spoiled due to the growth of *Acetobacter* spp., resulting in formation of flocculation, and by other Gram negative bacteria, as well as by *Leuconostoc* and *Lactobacillus* (153, 226).

2.2 Yeast

Thirty-five strains of asporogenous yeast were found to be less heat-resistant than 85 strains capable of ascospore formation. The genus *Saccharomyces* showed the highest heat resistance (208). Heat-resistant spores from yeast (*Saccharomyces cerevisiae*) have been isolated in apple juice.

2.3 Mould

Spore-forming moulds have caused spoilage in a variety of fruit juice products, including grapes and apples, cherries, strawberries and pineapple juice concentrate. Gas is normally not formed by moulds and, if so, very slowly (240) and in small amounts only.

Byssochlamys

Spoilage by *Byssochlamys* was originally observed in England during the early 1930s. The organism was later reported in Switzerland, Holland, the United States, South Africa, Canada, South America and Australia. Spoilage of fruit and fruit products by *Byssochlamys* has been a problem for the industry for a long time. *Byssochlamys fulva* can form extremely heat-resistant spores. In apple juice, heat resistance is affected by sugar concentration: the higher the concentration, the greater the resistance to heat. In an apple juice containing 47% sugar, 10 minutes at 101°C were needed to kill 10^6 spores of *Byssochlamys fulva*, while 10 minutes at 99°C achieved the same effect with spores of *Byssochlamys nivea* (43). Fruit juice needs to be heated above 90°C to achieve sufficient killing of *Byssochlamys fulva* (202).

Elimination of *Byssochlamys fulva* from equipment required treatment with water or vapour at temperatures in excess of 90°C, or alkaline at 90°C, or cold sulphuric acid or chlorine containing disinfectants at 60°C (202).

Other thermo-resistant moulds isolated from fruit juice are *Neosartorya fischeri* and *Talaromyces flavus* (232), their respective D-values and z-values are given in table 1.

Organism	D-value	z-value
N. fischeri	1.4 min (87.8°C)	5.6°C
T. flavus	2.2 min (90.6°C)	5.2°C

Table 1. *D-values and z-Values of Two Thermo-Resistant Moulds*

3. Microbiology of Sugars and Syrups

3.1 Sugar

In the production of high-acid liquid drinks, sugar is often added to the formulation. There are standards available for the microbiological quality of the sugars or syrups used. Bottlers' and canners' standards are given below.

3.1.1 IDF (150)

IDF gives the microbiological specification for sucrose, as shown in table 2.

Type	Average Counts	Limit Single Count
Total Count	20.0/g	50/g
Moulds	3.0/g	10/g
Yeast	2.0/g	6/g
Osmophilic Moulds	2.0/g	6/g
Osmophilic Yeast	0.1/g	2/g

Table 2. *Microbiological Standard for Sucrose (IDF)*

3.1.2 Microbiological Standards for Bottlers: Sugar (7)

a) mesophilic bacteria: max. 200 per 10 g;

b) yeast: max. 10 per 10 g;

c) moulds: max. 10 per 10 g.

3.1.3 Standards for Thermophilic Organisms in Granulated Sugar (7)

For Canners, National Food Processors' Association: samples taken from five bags or barrels of a shipment or lot.

a) *Total thermophilic spore count:* in the five samples examined, there shall be no more than 150 spores and an average of no more than 125 spores per 10 grams of sugar;

b) *flat sour spores:* in the five samples examined, there shall be no more than 75 spores and an average of no more than 50 spores per 10 grams of sugar;

c) *thermophilic anaerobic spores (gas-formers):* they are to be present in no more than three (60%) of the five samples, and to the extent of no more than four (65%) of the six tubes inoculated in any one sample;

d) *sulphide spoilage spores:* they are to be present in no more than two (40%) of the five samples, and to the extent of no more than five spores per 10 grams in any one sample. This would be equivalent to two colonies in the six inoculated tubes.

Storage of Sugar

In the production of shelf-stable, aseptically packed products with very limited availability of oxygen, the standard for bottlers and canners is probably the most relevant. The storage area for crystalline sugar should be dark (prevention of invasion by bees and other insects) and, of course, dry. Special care has to be taken when sacks are emptied into a mixing tank in order to avoid contamination by dust and other foreign matter.

3.2 Syrup

Storage Tanks: Cleaning is Necessary!

In the factory, the cleaning of tanks used for syrup or sugar solutions is critical. There is an obvious risk that yeast will multiply on the surface of the liquid and that mould will develop on the inside of the top of the tank or in shaft seals, etc. This risk is enhanced by condensate formation which will dilute the sugar solution, particularly on the surface. Regular cleaning is important, as is the use of alternate tanks. Refilling a tank containing a residual volume of sugar solution is not permitted.

It is helpful to use ultraviolet light at the top of the tanks and an overpressure of warm, filtered air in order to prevent the formation of condensate (the high Brix sugar solutions are often stored at high temperatures).

4. Water for Reconstitution

Fruit juice may be, and often is, produced from concentrates and water. Since water is quantitatively the main ingredient, its composition is of decisive importance for the quality of the product obtained.

4.1 Physical Requirements

The Joint ECE/FAO Codex Alimentarius (8) specifies the following standard for water quality used in the reconstitution of fruit juice from concentrates:

a) the water has to be free from all substances able to influence negatively the appearance, taste and flavour of reconstituted fruit juice;

b) the water is not to change the main physical characteristics of the reconstituted juice in comparison to the natural juice.

4.2 Microbiological Requirements (8)

The water has to be acceptable microbiologically:

a) water for the reconstitution of fruit juice concentrate shall be free from pathogenic organisms;

b) no sample of water is to contain *Escherichia coli*. In 100 ml of water no coliforms are to be present;

c) the number of colonies obtained from 1 ml of water is not to exceed 100.

4.3 Chemical Requirements

Water with a high degree of hardness influences the organoleptic quality of reconstituted fruit juice prepared from concentrates by neutralising the fruit acids. The water used for the reconstitution of fruit juice should be demineralised and meet the standard shown in table 3 (30).

In reconstituted fruit juice, the content of some chemical components should not exceed the limits given in table 4 (8).

Component	Content
Alkalinity	<50 ppm
Total Solids	<500 ppm
Iron	<0.1 ppm
Manganese	<0.1 ppm
Colour	<5 ppm
Turbidity	Colourless
Residual Chlorine	None

Table 3. *Water Composition*

Component	Content	Component	Content
Sulphate (SO_4)	<200 mg/l	Mercury (Hg)	<0.001 mg/l
Chloride (Cl)	<200 mg/l	Selenium (Se)	<0.008 mg/l
Nitrate (NO_3)	<45 mg/l	Calcium (Ca)	<75 mg/l
Arsenic (As)	<0.04 mg/l	Magnesium (Mg)	<30 mg/l
Lead (Pb)	<0.04 mg/l	Manganese (Mn)	<0.05 mg/l
Copper (Cu)	<0.05 mg/l	Fluoride (F)	<1.5 mg/l
Zinc (Sn)	<2.0 mg/l	Sodium (Na)	<120 mg/l
Iron (Fe)	<0.1 mg/l		
Cadmium (Cd)	<0.006 mg/l		
Cyanides (CN)	<0.05 mg/l		

Table 4. *Water Quality: Reconstitution of Fruit Concentrates*

Water for reconstitution is not to contain phenolic compounds or free chlorine. The pH of the water should be no less than 6.5 and no more than 8.5.

5. Factors Affecting Shelf Life

The factors affecting shelf life are the same as those affecting the shelf life of other foods. They include the quality of the raw materials, holding time before processing, plant sanitation conditions, processing treatment, container characteristics, oxygen content and storage temperature. High storage temperatures have a negative impact on quality by accelerating non-enzymatic browning, flavour change and nutritive value (181).

6. Fruit Juices and By-Products

6.1 Oranges

Citrus fruit may be processed into various products and by-products. The juice is by far the largest component.

6.1.1 Pulpwash Juice

“Washed pulp solids” or “pulp wash” are soluble fruit solids recovered from a pulp finisher by extraction with water. Soluble fruit solids from which the water has been extracted are used for manufacturing juice drinks.

6.1.2 Comminuted Fruit Juice

This product is extracted by cutting up whole fruit and is used in drink-based products. Its production is very limited.

6.1.3 Segments

Segments are peeled fruit cut up into sections or into practically whole sections of membrane. This type of product is very limited in volume and is canned and pasteurised for storage at ambient temperature or canned and chilled to be used in citrus salads.

6.1.4 Pectin

Many of the component parts of citrus fruit such as the flavedo, albedo, membrane, juice vesicles, and the core contain varying quantities of pectin. Pectin is not produced in a typical citrus processing plant since it requires complex manufacturing processes.

6.1.5 Juice Vesicles and Pulp Cells

Juice vesicles (pulp) can be recovered directly from the juice finisher. Both washed and unwashed juice vesicles can be frozen or dried for use as stock material in various types of beverages and food products. Pasteurised frozen pulp may be used to adjust the pulp content of juice, juice concentrates, drinks containing juice, and other beverages.

6.1.6 Essence

Volatile components are recovered during the evaporation-concentration operation by an essence distillation or condensation system. The essence contains oil and aqueous phases. The essence oil floats on the top of the aqueous essence and is decanted off in a tank. The aqueous essence contains aroma and flavour which can be added back to the concentrate. The essence oils are processed by the flavour manufacturers and used as a flavour enhancer.

The aroma fraction can be purchased separately together with the de-flavoured concentrate thus permitting separate processing. The de-flavoured reconstituted juice is heat processed while the essence solution is sterilised by filtration and added back to the product after the heat treatment. This procedure eliminates heat-induced flavour changes of sensitive aroma compounds.

6.1.7 Dried Animal Feed

Dried citrus pulp or pellets are made from the peel, rags, pulp and seed residue ejected by citrus fruit extractors which are treated with lime and dried to 8-10% moisture in the feed mill.

6.1.8 Citrus Molasses

Syrup is produced by evaporation concentration of the pressed liquid obtained from lime-treated peel residues. Citrus molasses can be added back to the peel residues to enhance the nutritional value of dried citrus pulp or used in the fermentation of citrus alcohol.

6.1.9 d-Limonene

Limonene is a by-product that is recovered from the manufacture of citrus molasses. The product comes from the peel oils that have been treated with lime in the feed mill, and recovered by distillation from citrus press liquor or decanted from the condensate of the molasses evaporator. Large quantities of d-limonene are used in the plastics industry.

6.2 Grapefruit

6.2.1 Pulp Wash

Pulp separated during the finishing operation can be washed with water to give a low Brix solution of the adhering juice solids. In industrial practice, pulp is washed with 1-3 kg water/kg pulp in one to eight stages resulting in about an additional 7% recovery of soluble solids. Grapefruit pulp wash can be used in the manufacture of juice drinks, citrus punch and similar products. Grapefruit pulp wash is extremely bitter and should be used in moderation.

6.2.2 Essence Recovery

Commercially, the most widely used method of obtaining essence is through an "Essence Recovery System" connected to the evaporator. When the feed juice enters the first stage of the evaporator, essence-bearing vapours are boiled off under vacuum with the water vapour.

Essence vapour enters the essence system at the vent condenser. The vent condenser condenses about 90-95% of the essence product. Grapefruit essence is a distilled aqueous solution of volatile compounds that possesses a characteristic flowery or fruity aroma typical of freshly squeezed grapefruit juice.

6.3 Lemon and Lime

Most of the lemon juice retailed in the United States is consumed in the bottled single-strength and lemonade beverage product forms. Frozen concentrated lemonade is the leading form in the beverage category.

6.3.1 Lemon Oil

The range of applications of cold-pressed lemon oil is as extensive as that of processed lemon juice. There is a heavy demand for lemon oil as a flavouring in foods and soft drinks, cosmetics, pharmaceuticals and household products. The market value of this product is usually very strong. Lemons contain 6-8 kg of oil per metric ton of fruit. At least 50%, and as much as 90%, of this quantity can be recovered.

Chemically, the essential oil of lemon is composed of sesquiterpene hydrocarbons and terpene oxygenated terpene alcohols, aldehydes and esters and non-volatile material including waxes, lipid esters, sterols, coumarins, psoralens and other substances.

All citrus oils are subject to oxidation, and selection of proper containers and storage conditions is of critical importance. Oxidative reactions in lemon oil are accelerated or catalysed by light, heat and moisture.

6.3.2 Pectin

Pectin is the purified carbohydrate product obtained by aqueous extraction of an appropriate edible plant material. Pectin is an essentially linear polysaccharide containing between a few hundred and about 1,000 building blocks in a chain-like configuration (26).

Because of the high concentration of pectic substances in the albedo of citrus peel, these fruit juice extraction wastes serve as the chief raw materials for the recovery of purified pectin sold to the food, beverage and pharmaceutical industries. Pectin derived from lemon peel is considered to be superior to orange and grapefruit pectins in terms of jelly units, the jelly unit being a measure of both pectin yield from raw material and jellifying power of the refined product. Lemon albedo contains about 33% of its dry weight as total pectic substances.

6.3.2.1 Production of Pectin

Figures 2 and 3 illustrate the process of pectin production.

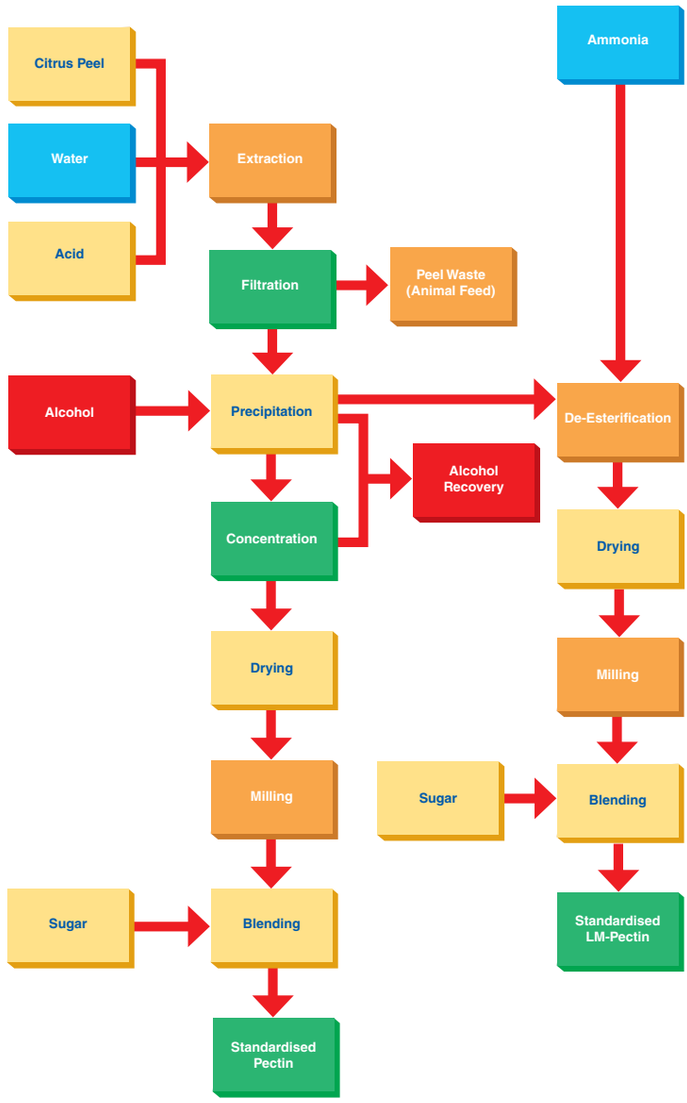


Figure 2. *Flow Chart: Production of Pectin*

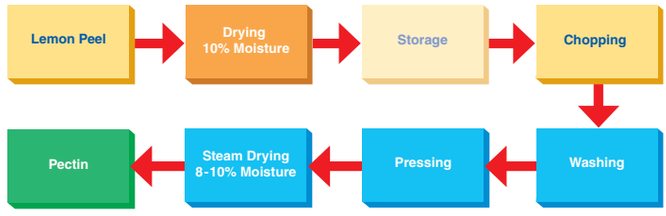


Figure 3. *Flow Chart: Pectin Production from Lemon Peel*

6.3.3 Shaved Peel

There is an essentially limited but constant demand for shaved lemon peel. The product is used primarily in the manufacture of marmalade, spices and teas.

6.4 Passion Fruit

In the juice extracted from passion fruit, about two-thirds of the bulk is refuse, of which 90% is rind and about 10% is seeds.

6.4.1 Composition of Rind Material

Passion fruit rinds were found to be satisfactory as supplementing feed for dairy cows. The rinds dehydrated rapidly without lime pre-treatment and the dried material was acceptable to dairy cattle at 22% of the ratio. The composition of passion fruit rind is shown in table 5.

Compound	Fresh Purple Rind %	Dried Yellow Rind %
Moisture	81.92	16.80
Crude Protein	2.56	4.58
Ether Extract	0.12	0.33
Ash	1.47	6.76
Crude Fibre	5.01	25.66
N-Free Extract	7.14	45.87
Pentosans		15.70
Lignin		6.50
Pectin	1.78	20.00

Table 5. *Composition of Purple and Yellow Passion Fruit Rind*

6.5 Papaya

6.5.1 Puree and Syrup

Papaya puree can be used in the preparation of syrups to improve natural papaya flavours and colours. These syrups can be used as a pancake or waffle syrup, ice cream topping or similar dessert. Their composition is given in table 6.

Citric acid is added to papaya syrup to adjust the acidity to about pH 4. This is necessary to permit mild heat treatment (88°C) in the subsequent pasteurisation of the product. Stabilisers and preservatives are added in amounts that do not alter the final solids content by more than 0.1%.

An invert sugar and sugar are added while stirring and heating to 66°C.

Component	Amount
Water	5 lb
Stabiliser	4.00 oz
Papaya Puree	20 lb
Citric Acid	3.00 oz
Sodium Benzoate	0.80 oz
Potassium Sorbate	0.15 oz

Table 6. *Composition of Papaya Syrup*

6.5.2 Papain Production

Papain is one of the more important proteolytic enzymes used in the food, cosmetics, leather and drug industries. Papain is derived from the latex of papaya fruit. There are two major uses for papain in the food industry: for tenderising meat and as a beer stabilising agent. It is estimated that beer stabilisation accounts for about 75% of the consumption of papain.

6.5.3 Pectin

Papayas that are no longer useful in the production of papain can be used in the production of pectin.

6.6 Guava

6.6.1 Guava Syrup

Guava syrup can be used in the preparation of syrups to improve natural flavours and colours. These syrups are also satisfactory for use as pancake or waffle syrups, as ice cream topping or similar desserts. One syrup formulation for guava is as follows: to 20 lb of guava puree, stir in 40 lb of 64° Brix pineapple ion-exchange syrup and heat to 66°C; stir in an additional 40 lb of sugar and continue to heat to 88°C, then hot-fill. Cool in a water bath.

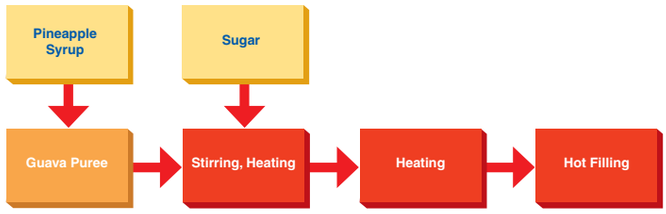


Figure 4. *Flow Chart: Production of Guava Syrup*

6.6.2 Guava Pectin Powder

Guavas are a rich source of pectin. In some countries, they provide an inexpensive and readily available source of this natural food thickener and gelling agent. Figure 5 illustrates the production of pectin from guava puree.

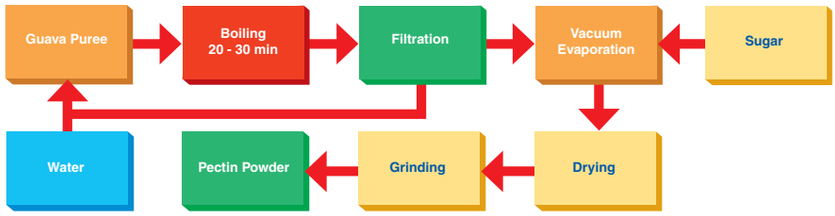


Figure 5. *Flow Chart: Production of Pectin*

Pectin is extracted from guava pulp by boiling for 20 to 30 minutes with equal amounts of pre-filtered water. The hot filtrate is used to extract additional amounts of fresh guava pulp until a total solids content of 8.5% is attained. The extract is further concentrated by vacuum evaporation until a 20% total solids content is reached, at which point 0.2 parts of sugar are added for each part of concentrated extract. This mixture of sugar and pectin extract is vacuum-dried without the application of heat. The dry product is then ground into a fine powder.

7. Standards for Fruit Nectars

Standards for fruit drinks have been developed. The Codex Alimentarius Commission on Standardisation of Fruit Juices (FAO/WHO Food Standard Program 1981) updated the Codex Standards for fruit juices, concentrated fruit juices and fruit nectars. The Codex Standards for apricot, peach, and pear nectars preserved exclusively by physical means (Codex STAN 44-1981) are as follows:

7.1 Description

Unfermented but fermentable pulpy product, intended for direct consumption, is obtained by blending the total edible part of sound and ripe apricots, peaches or pears, concentrated or unconcentrated, with water and sugars or honey and preserved exclusively by physical means.

7.2 Essential Composition and Quality Factors

7.2.1 Minimum Content of Fruit Ingredient

The product shall contain no less than 40% w/w in the case of peach and pear nectars, and no less than 35% w/w in the case of apricot nectars, of single-strength fruit ingredient or the equivalent derived from any concentrated fruit ingredient.

7.2.2 Sugars

One or more solid sugars, as defined by the Codex Alimentarius Commission, is to be added to the product. The total quantity of added sugar or honey, calculated for dry matter, is not to exceed 200 g/kg of the final product.

7.2.3 Honey

Honey, as defined by the Codex Alimentarius Commission, may be used if it is the sole sweetening ingredient added.

7.2.4 Lemon Juice

Lemon juice may be added as an acidifying agent.

7.2.5 Soluble Solids

The soluble solids content of the product is not to be less than 13% w/w as determined by a refractometer at 20°C, with no corrections for acidity and read as Brix on the International Sucrose Scales.

7.2.6 Apparent Viscosity

The apparent viscosity of the product is to be such that the flow time is not less than 30 seconds, as determined by the method of Lamb and Lewis.

7.2.7 Ethanol Content

The ethanol content is not to exceed 3 g/kg.

7.2.8 Hydroxymethylfurfural

The hydroxymehtylfurfural content is not to be more than 10 mg/kg.

7.2.9 Organoleptic Properties

The product shall have the characteristic colour, aroma, and flavour of the fruit from which it is made, taking into consideration the addition of honey as a substitute for sugar.

7.3 Food Additives

The maximum levels of citric acid, malic acid and ascorbic acid are limited by Good Manufacturing Practices (GMPs).

7.4 Contaminants

Limits for the content of metals are shown in table 7.

Contaminant	Level
Arsenic (As)	<0.2 mg/kg
Lead (Pb)	<0.3 mg/kg
Copper (Cu)	<5.0 mg/kg
Zinc (Zn)	<5.0 mg/kg
Iron (Fe)	<15.0 mg/kg
Tin (Sn)	<250.0 mg/kg
Sum of Copper, Zinc and Iron	<20.0 mg/kg
Sulphur dioxide	<10.0 mg/kg

Table 7. *Limits for the Content of Metals*

7.5 Hygiene

It is recommended that the products covered by the provisions of this standard be prepared in accordance with the Recommended International Code of Hygienic Practice for Canned Fruit and Vegetable Products (Ref. No. CAP/RCP 2-1969) and the General Principles of Food Hygiene (Ref. No. CAP/RCP 1-1969, Ref. 1) recommended by the Codex Alimentarius Commission.

When tested by appropriate methods of sampling and examination, the product:

- shall be free from microorganisms capable of development under normal conditions of storage; and
- shall not contain any substances originating from microorganisms in amounts which may represent a hazard to health.

7.6 Weights and Measures

7.6.1 Container Fill

Minimum Fill: The nectar is to occupy not less than 90% v/v of the water capacity of the container. The water capacity of the container is the volume of distilled water at 20°C which the sealed container will hold when completely filled.

7.6.2 Labelling

In addition to Sections 1, 2, 4 and 6 of the Codex General Standard for the Labelling of "Pre-packaged Foods" (Ref. No. CODEX STAN 1-1981), the following provisions apply:

- *The name of the food:* the name of the product shall be "apricot nectar" or "pulpy apricot nectar", "peach nectar" or "pulpy peach nectar", "pear nectar" or "pulpy pear nectar", as appropriate.
- *List of ingredients:*

a) a complete list of ingredients including added water shall be declared on the label in descending order of proportion;

b) the addition of L-ascorbic acid shall be declared in the list of ingredients as:

(i) "L-ascorbic acid as antioxidant"; or

(ii) "Antioxidant".

- *Net contents:* the net contents shall be declared by volume in one or more of the following systems of measurement: Metric, US or British units, as required by the country in which the product is sold; for British units, units of capacity measurement shall be used.
- *Name and address:* the name and address of the manufacturer, packer, distributor, importer, exporter or vendor of the product shall be declared.
- *Country of origin:* the country of origin of the product shall be declared if its omission would mislead or deceive the consumer.
- *Lot identification:* each container shall be embossed or otherwise permanently marked, in code or in plain language, to identify the producing factory and the lot.
- *Additional requirements:* the following additional provisions shall apply:

a) no fruit or nectar may be represented pictorially on the label except the species of fruit present in the nectar therefrom;

b) when the product contains honey the declaration "contains honey" shall appear in close proximity to the name of the product;

c) no claim shall be made in respect of "Vitamin C" nor shall the term "Vitamin C" appear on the label unless the product contains such a quantity of "Vitamin C" as would be accepted by national authorities in the country in which the product is sold, as warranting such a claim or the use of such a term.

d) where fruit nectars require to be kept under conditions of refrigeration, there shall be information on how to keep and, if necessary, thaw the product.

- *Bulk packs:* In the case of fruit nectars in bulk, the information required by Sections 7.1 to 7.74 shall be either given on the container or in accompanying documents except the name of the product and the name and address of the manufacturer or packer which should appear on the container. However, the name and address of the manufacturer or packer may be replaced by an identification mark, provided that such a mark is clearly identifiable with the accompanying documents.

18. Processing and Composition of Fruit Juice

Summary

Heat processing of fruit juice is a compromise between the effect desired, i.e., the control of microbiological spoilage and/or enzyme inactivation, and the undesirable product changes inflicted by such a treatment. The choice of optimal processing conditions becomes even more complicated if different products are produced on the same line. Aspects of safety margins also need to be considered. The microbiological result of a heat process is determined by:

Microbiological Result = Process Parameters + Microbial Load

Since the microbial load is not usually known, a certain amount of over-processing is unavoidable.

A further factor to be considered is the composition of the product to be processed. Of special interest are the pH and the presence and amount of certain organic acids. In this chapter, the composition and most commonly used processing conditions of a number of fruit juices are presented.

1. General

The processing conditions of products in general and of fruit juice in particular are determined mainly by the goal of the process (for instance, commercial sterility), the product, enzyme inactivation and microbiological aspects. The microbiological load is determined by the quality of the raw materials (concentrates or fruit) *and* by the care which is exercised during all the pre-processing stages.

2. Citrus Juice

Citrus fruits are classified botanically as berries (100). The thin outer cuticle covering a flavedo layer is characterised by a green, yellow, or orange colour containing numerous oil glands filled with an aromatic essential oil. Beneath the flavedo is the albedo layer composed of light-coloured spongy tissue of varying thickness. The albedo layer is high in pectic materials and certain bitter principles (100). The flesh of the fruit consists of segments divided by walls which contain juice-bearing vesicles. These sacs are made up of an epidermal wall easily ruptured to release the juice. The central core of the fruit is composed of a light-coloured spongy tissue similar to the albedo.

2.1 Orange Juice

2.1.1 Production of Orange Juice Concentrate (54)

To obtain the juice, it is essential that during mechanical pressing, the extractor should not be adjusted too tightly which will result in extraneous flavours from the skin and larger amounts of pectins in the juice. Modern, highly efficient and multi-effect falling film evaporators include a stabilising unit that inactivates the pectinesterase in the juice (237). A typical process for the production of orange juice concentrate is illustrated in figure 1 which shows the different process stages.

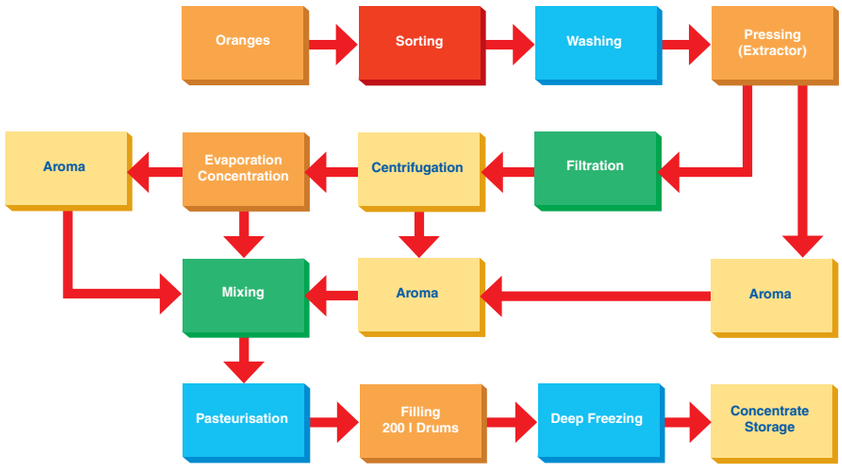


Figure 1. *Flow Chart: Production of Orange Juice Concentrate*

Brazilian orange juice typically has a relatively low Brix/acid ratio of around 13 to 15. Orange juice from Florida and California have a significantly higher ratio.

2.1.2 Pre-Processing

In the pre-processing area, the concentrates are reconstituted with water (figure 2). The water can be obtained from the municipal water supply or from a well. Concentrates are usually stored deep frozen (-18°C) in drums.

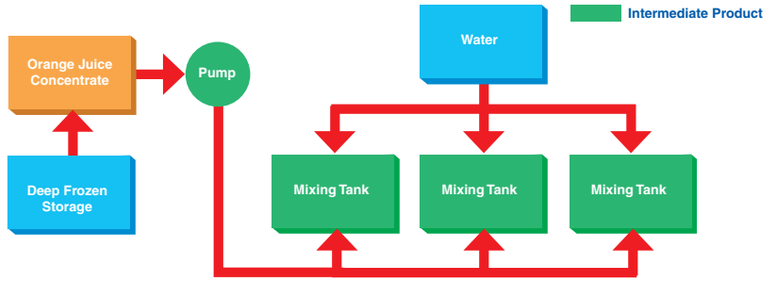


Figure 2. *Flow Chart: Reconstitution of Orange Juice Batch Process*

The quality of the water used for reconstitution is of primary importance (237). To ensure the required quality, the water should be treated prior to use. Figure 3 shows an example of such a treatment.

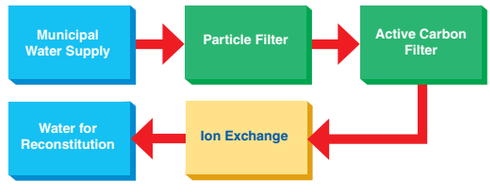


Figure 3. *Flow Chart: Water Treatment*

2.1.3 Processing and Packaging

After reconstitution, the orange juice may be heated to, for example, 95°C with a holding time of 25 seconds in a plate heat exchanger. An aseptic transfer line connects the heat exchanger with the aseptic packaging equipment (figure 4).

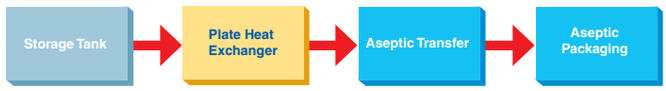


Figure 4. *Flow Chart: Production of Long-Life Orange Juice*

The aseptic transfer can be "straight" piping between the heat exchanger and the aseptic filling equipment, or an aseptic buffer tank may be used. The juice is then packed in aseptic packages using suitable packaging equipment.

2.1.4 Distribution Equipment

If portion packs are used, a straw may be applied to each package after the packs have left the packaging machine. The packages may then be placed in a tray, shrink-wrapped and manually palletised. The outer wrapping used must provide the mechanical protection needed during handling and distribution.

After being palletised, the product is transported into a store.

2.1.5 Product Characteristics

Aseptic processing procedures produce a higher quality orange juice than hot-filling. However, differences in quality may disappear during storage at high ambient temperatures.

Availability of Oxygen

Oxygen dissolved in the product, in the container head space, or permeating through the package accelerates the rate of ascorbic acid destruction and non-enzymatic browning (181) and reduces the shelf life. The most important factor in determining the shelf life of aseptic orange concentrates and single-strength juice is the storage temperature (130, 253).

The objective of aseptic processing is to eliminate microbial and enzymatic activity and to provide a package environment in which an acceptable quality level can be maintained during the entire intended shelf life of the product.

2.1.6 Enzyme Inactivation

The most important enzyme in citrus juice is pectinesterase, the activity of which results in loss of cloud in juice or gelation in concentrates. Insufficient inactivation will lead to alteration of the naturally occurring pectin (100) resulting in gel formation (54). Different time-temperature combinations are stated in the literature for inactivation of the enzyme. One minute's heating at 90°C was found to be necessary to achieve a two log cycle reduction in pectinesterase activity, which is necessary for commercial stability (130). In another study, inactivation required a thermal process of 85-95°C with a holding time of 10 to 20 seconds (237). Table 1 illustrates the effect of pH on the inactivation of the enzyme; higher pH values require higher temperatures (30, 219).

Inactivation of Pectinesterase

pH	Temperature
2.4	74°C
3.2	89°C
3.3	90°C
3.8	94°C

(15 seconds holding time, pH adjusted with citric acid in orange juice)

Table 1. *Pectinesterase Inactivation*

Further investigation revealed that a temperature of 82-87°C is necessary to inactivate pectinesterase in orange juice.

2.1.7 Flavour

Of great significance to the flavour of citrus juice are compounds which contribute to bitterness, mainly limonene and naringin (100). About 0.02% of orange juice are flavour ingredients, including 75-98% hydrocarbons, which are mainly d-limonene, 0.6-1.7% aldehydes, 1% esters, 1% ketons and 1-5% alcohols (237). Volatile flavours in citrus fruit are present as essential oils in the peel of the fruit and as aqueous essence oils in the juice. During commercial juice extraction, process peel oils are captured by means of water spray. The oil-water slurry is then centrifuged to separate the oil from the water. Aqueous essences and essence oils are distilled from the juice in the early stages of evaporation (100).

Peel Oils: Recovery

Limonene

During storage, orange juice or juice concentrates aseptically packed into cardboard cartons lined with polyethylene showed some loss of aroma volatiles, mainly limonene which was absorbed into the plastic layer of the packaging material (181). This is considered advantageous since limonene is known as a precursor of off-flavour components (α-terpineol) (109, 237).

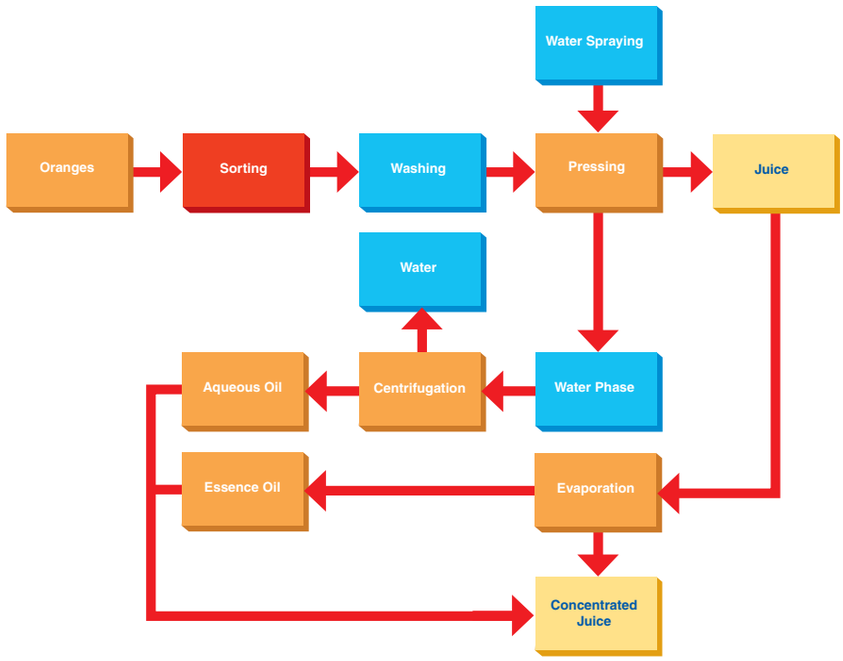


Figure 5. *Flow Chart: Recovery of Aromatic Compounds during the Production of Orange Juice Concentrate*

A total of 330 different volatile compounds have been found in citrus fruits. About 90% of the peel oil is (+)-limonene which is regarded as non-essential to orange flavour (108).

2.1.8 Microbiology

Bacteria

During processing, some bacteria may develop in juice and juice concentrates, especially *Leuconostoc mesenteroides* and *Lactobacillus plantarum*, and spoil the product because of the production of acetoin (acethyl-methyl-carbinol). It is common to control the quality of citrus as well as other products by chemical rather than microbiological analysis. If the raw material has already been microbiologically spoiled, an increase in lactic acid, acetic acid, ethanol, glycerol and acetoin will be found.

During concentration (evaporation), the amount of acetoin initially decreases but after a few hours an increase is found if the product and the evaporator are contaminated with, for instance, *Lactobacillus*. This may be taken as a signal to stop production and clean, disinfect or sterilise the evaporator. The damage in quality caused by vegetative bacteria is primarily an off-flavour.

Moulds

Recently, product spoilage has been caused by unidentified species of *Actinomyces* but it has not been clarified whether this was due to bacteria surviving the process or re-contamination after the heat treatment.

In several studies it was found that different species of *Penicillium* cause blue and green rot on citrus fruit.

Some moulds form rather heat-resistant types of spores, such as chlamydospores. They may survive the pasteurisation process and cause spoilage in finished products. Chlamydospores of *Byssochlamys fulva* and *Fusarium oxysporum* have been found to survive the pasteurisation of citrus products.

Several genera of bacteria can survive in juice concentrate for several weeks but they have not been known to multiply in juice with more than 20° Brix if the temperature is lower than 5°C.

Spoilage Organisms

Spoilage by microorganisms is limited to acid-tolerant populations which are predominately lactic acid bacteria, yeast and moulds. Thermal resistance of yeast ascospores is somewhat higher than that of vegetative cells. Heat resistance is also higher in concentrates than in single-strength juice. The thermal resistance of *Leuconostoc*, lactobacilli and yeast was determined in single strength orange juice and in a 42° Brix orange concentrate. As shown in table 2, the organisms had greater heat resistance in the concentrate (194).

	Single Strength		Concentrate	
Organism	**F_{71}**	**z**	**F_{71}**	**z**
Leuconostoc	0.04 min	7	0.23 min	10
Lactobacilli	0.28 min	7	1.20 min	18
Yeast	0.35 min	6	6.70 min	9

Table 2. *Heat Resistance of Some Spoilage Organisms*

Low oxygen levels in packed products usually prevent or retard the growth of the moulds *Byssochlamys* and *Penicillum* sp. To kill ascospores of several isolates of *Byssochlamys fulva* required 20 to 50 minutes at 88°C with z-values ranging from 4-8°C (130).

High counts of faecal coliform-positive, capsule forming *Klebsiella pneumonia* cells were observed in high densities (10^4 to 10^8 CFU/100 ml) in two commercial batches of frozen orange juice concentrate (table 3) (119).

No. of lots analysed	**No. with Coliforms**	**No. with Faecal Coliforms**
39	29	22
4	4	4

Table 3. *Coliforms in Orange Juice Concentrate*

In orange juice concentrate, the following generation times (table 4) were determined for *Klebsiella pneumonia* (119). The results appear, however, to be unlikely.

Temp. °C	**Generation Time (h)**	**Max. Count/ml**
4	1.84	2.00×10^7
25	0.48	1.65×10^8
34	0.39	1.24×10^8

Table 4. *Effect of Temperature on Generation Time and Maximal Counts of Klebsiella Pneumonia in Orange Juice Concentrate*

2.1.9 Quality Control of Orange Juice

US FDA Definition of Orange Juice

The United States Food and Drug Administration defines the standard of identity of orange juice as follows: “Orange juice is the unfermented juice obtained from mature oranges of the species *Citrus sinensis*. Seeds (except embryonic and small fragments of seeds that cannot be separated by good manufacturing practices) and excess of pulp are removed. The juice may be chilled, but is not frozen”.

2.1.9.1 Microbiological Testing

Methods of testing microbiological quality vary. Most quality control departments utilise thermal abuse testing. Often temperatures between 25°C and 35°C are used for periods ranging from overnight to two or more days. Periods of this length are definitely too short for testing long-life products.

Bacteria	Yeast	Moulds
Acetobacter	*Alternaria*	*Aspergillus*
Bacillus (?)	*Brettanomyces*	*Byssochlamys*
Citrobacter	*Candida*	*Cladosporium*
Enterobacter	*Hanseniaspora*	*Fusarium*
Escherichia	*Hansenula*	*Geotrichum*
Gluconobater	*Klockera*	*Mucor*
Klebsiella	*Pichia*	*Penicillium*
Lactobacillus	*Rhodotorula*	*Rhizopus*
Leuconostoc	*Saccharomyces*	
Proteus	*Torulaspora*	
Serratia		

Table 5. *Genera of Microorganisms Isolated from Citrus Products*

Because of the low pH value, bacterial growth is mainly limited to *Lactobacillus* and *Leuconostoc* (31). The most common spoilage organism is yeast (238). At the end of the abuse period, microbial growth is evaluated by:

a) standard plating on orange serum agar and/or acidified potato dextrose agar; or

b) the visual inspection of packages for obvious fermentation.

Concentrates are most often diluted prior to plating.

Diacetyl!

Production of diacetyl by lactic acid bacteria gives a butter-like or buttermilk off-flavour in citrus juice and has been used as an indicator of microbial spoilage. A level of 0.8 ppm diacetyl is regarded as a cut-off point to indicate when a processing line should be cleaned and sanitised.

2.1.9.2 Chemical and Physical Testing

The orange juice must meet relevant official standards which vary from country to country. Tests which are commonly conducted include: soluble solids (Brix), total titrable acidity (TA) as citric acid, Brix/acid ratio, pH, colour score, cloud, pectin methyl esterase, suspended pulp, screen pulp, quick fibre pulp, juice bitterness, recoverable oil in juice, ascorbic acid (vitamin C), total aldehydes, ash, and chemical oxygen demand.

2.1.9.3 Quality Improvement

Consumers typically describe orange juice as flavourful, aromatic, colourful, healthy, natural and refreshing (100). Quality evaluations of citrus juice are based on objective and subjective characteristics. The final criterion for the quality evaluation of citrus juice is subjective or sensory (100). An improvement in quality of reconstituted orange, grapefruit, apple and grape juice became possible by the sterile injection of a mixture of alcohol (10-15%), water and flavour compounds into the heat-processed juice base. As illustrated in figure 6, this can be done either before or preferably during the aseptic filling operation (20, 237).

Addition of Aromatic Compounds Using Sterile Filtration

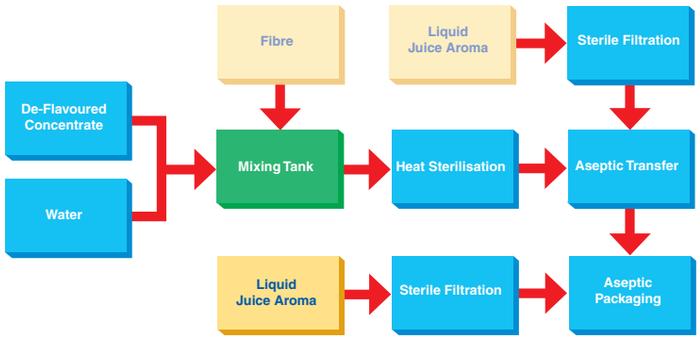


Figure 6. *Flow Chart: Separate, Sterile Addition of Flavour Compounds*

In the process of concentration, the juice base and a flavour fraction are obtained separately. Normally, both fractions are mixed in the process of reconstitution or before shipping of the concentrate. Some of the aroma compounds are adversely affected by the heat treatment. This can be avoided by sterile injection of a liquid aroma solution (figure 6).

In addition, frozen citrus cells may be added in an amount of up to 10-20 g/litre of product (237).

2.2 Lemon Juice

If existing local standards have to be followed, they may vary from country to country (USA: 21 CFR part 146, canned fruit juices) (31). The pH range of lemon or lime juice is 2.2 to 2.6 (238).

2.2.1 Concentrates

Factors of product quality which must be scored to ascertain grade are mainly colour and flavour. In addition, cloud strength and stability should also be tested.

a) *Cloud strength:* is measured by reading the visible light absorbency of a sample of reconstituted juice that has been centrifuged under defined conditions.

b) *Cloud stability:* can be estimated simply by observing the cloud retention in a sulphided sample of reconstituted juice stored at ambient temperature for several days.

2.2.2 Single Strength

USA 21 CFR, Sec. 146.114 (31) specifies: products reconstituted from concentrates must have a titrable acidity (TA) of at least 4.5% by weight and a soluble solids content of at least 6% determined by refractometry but not corrected for acidity. The United Nations Codex Alimentarius Commission has also recommended 4.5% TA and 6° Brix.

Since flocculation of lemon juice cloud by low methoxyl pectin is dependent on interaction with polyvalent metal ions, the quality of the water used for reconstituting the juice has sometimes been of concern.

3. Apple Juice

Experience has shown that in spite of all the very variable parameters which may influence the composition of the apples and their juice, a large number of values are statistically subject to laws which may be quantitatively and qualitatively characteristic of the various types of apple. Collection and consideration of these parameters is justified for assessing the products for their quality, authenticity and identity.

A reference standard is a guideline for what is considered an acceptable juice. European Community Reference Standards (ECRS) for apple juice require that the product meet the limits of composition shown in table 6. In addition, the content of metals is not to exceed the values stated in table 7.

Parameter	Limit
Rel. Density 20/20	> 1.045
°Brix	> 11.2
Volatile Acids as	
Acetic Acid	< 0.4 g/l
Ethanol	< 3.0 g/l
Lactic Acid	< 0.5 g/l
Sulphurous Acid	< 10.0 mg/l
Sodium	< 30.0 mg/l
Nitrate	< 10.0 mg/l
Patulin	< 50.0 µg/l

Table 6. *Absolute Quality Requirements*

Heavy Metal	Limit
Arsenic (As)	< 0.1 mg/l
Lead (Pb)	< 0.2 mg/l
Copper (Cu)	< 5.0 mg/l
Zinc (Zn)	< 5.0 mg/l
Iron (Fe)	< 5.0 mg/l
Tin (Sn)	< 1.0 mg/l
Mercury (Hg)	< 0.01 mg/l
Calcium (Ca)	< 0.02 mg/l

Table 7. *Limits on Metal Content*

Apple juice is obtained, by definition of the EC Directive, from mature and sound fruit by mechanical processes and is treated by physical means and/or diffusion processes, provided that the concentrated juice thus obtained shows the same organoleptical and analytical characteristics as the product obtained by mechanical processes only.

The clearing of apple juice before concentration is recommended and may proceed as illustrated in figure 7 (127).

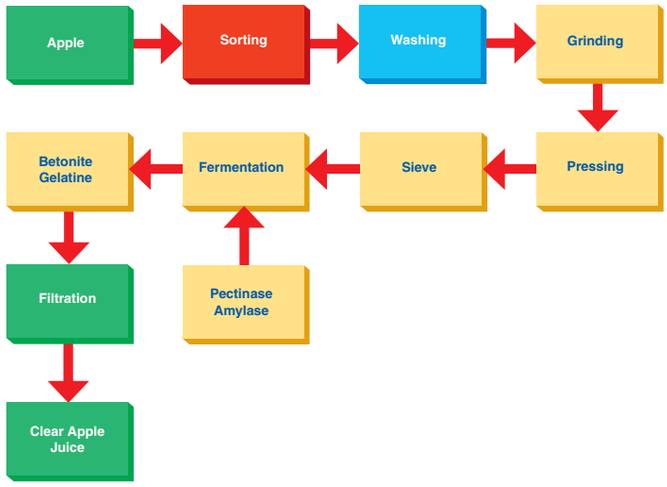


Figure 7. *Flow Chart: Production of Apple Juice*

4. Tomato Products

A rather wide range of different tomato products are on sale today. Many of these are available as long-life products.

4.1 Processing of Tomatoes

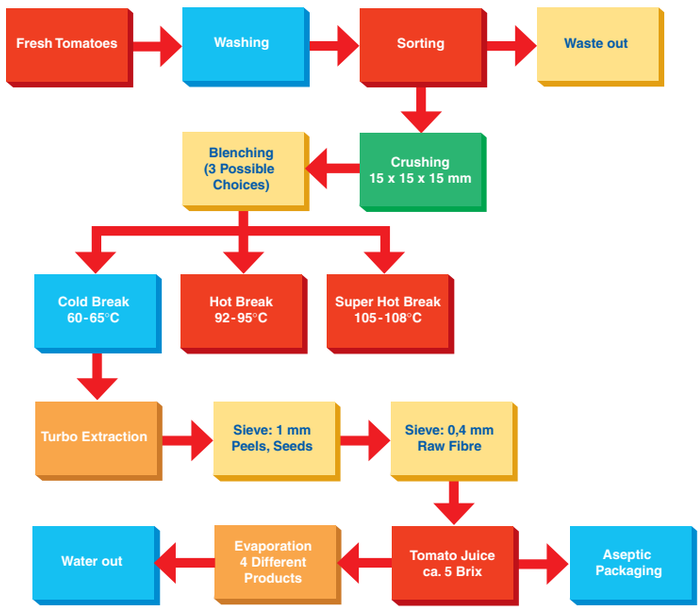


Figure 8. *Flow Chart: Production of Long-Life Tomato Juice*

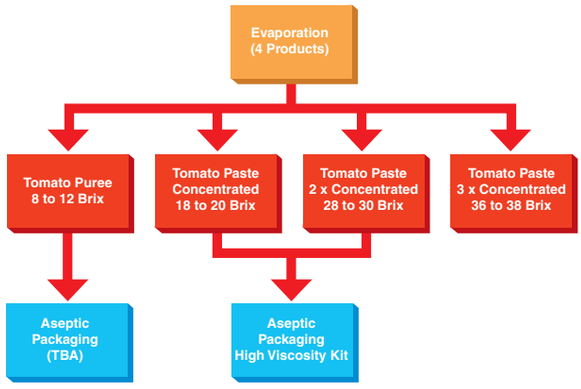


Figure 9. *Flow Chart: Production of Long-Life Tomato Puree and Paste*

As shown in figures 8 and 9, a total of five different products is obtained (tomato juice, tomato puree and three types of tomato paste) which can be marketed as such or used as raw materials for the manufacture of other tomato-based products such as tomato sauce, ketchup, etc. The variety of resulting products is plentiful. Tomato products are rather heat-stable. Typical processing parameters for the sterilisation of tomato juice and tomato products are above 100°C. Values suggested are:

- 100°C, 90 minutes;
- 115°C, 3 minutes;
- 120°C, 1 to 2 minutes;
- 120°C for 15 seconds if followed by aseptic filling procedures (107).

4.2 Microbiology

Whereas in fruit products based on oranges, apricots, apples, etc., spoilage by heat-resistant bacteria is uncommon, tomato-based products are different. It is possible that the pH as such is part of the explanation. Perhaps the types of organic acid and other compounds have a specific inhibitory effect on the germination of spores of heat-resistant bacteria.

pH Variations!

The pH variations observed in tomatoes are very large (figure 10). The pH range registered in California (1975) was from 3.6 to 4.8. pH, and values as high as 5.4 have been reported. Even at pH readings below 4.6, growth of *Bacillus coagulans* and *Clostridium* sp. can occur. *Bacillus coagulans* spores can germinate in tomato juice at pH values down to 4.2, or even 4.0 (107). Growth has even been registered as low as 3.8.

Spoilage by *Bacillus* and *Clostridium*

In tomato products, several strains of *Bacillus* and *Clostridium* have been found to cause product spoilage. Extensive studies have shown that pH levels increase in mould-damaged tomatoes. Visual inspection, and sorting and trimming the raw material become critical for the quality of the juice, paste or other products. If mouldy tomatoes are processed the result is an increased load of bacteria which can grow in the tomato because of the higher pH: the risk for product spoilage thus increases. If the raw material received by the food processor is already pulp or tomato concentrate, an often recommended method of quality control is the Howard mould count. In principle, a smear of product is studied in the microscope and the number of fields containing mould elements is counted. The test has, however, limited value when the product has been homogenised. During homogenisation, larger pieces of mould growth are split up into numerous elements which gives a misleading result.

Howard Mould Count

4.3 Composition

The composition of tomatoes is shown in table 8.

Component	Content
Dry Matter	5 - 6%
Total Sugar	1.5 - 3.0%
Total Acid	0.4 - 0.7%
Vitamin C	20 -30 mg/100g

Table 8. *The Composition of Tomatoes*

4.4 The pH of Tomatoes

When the pH of tomato juice begins to approach 4.3, consideration should be given to acidification. If the pH averages 4.3, there is the possibility that the pH of some individual tomatoes may exceed 4.5. The risk of economic spoilage increases greatly. Above pH 4.8, the danger of botulism begins to appear (206). In order to avoid different processing temperatures and to obtain a product with a more uniform quality, some producers adjust the pH of tomato products by adding acid (usually citric acid).

The wide variation in pH of mature tomatoes is shown in figure 10.

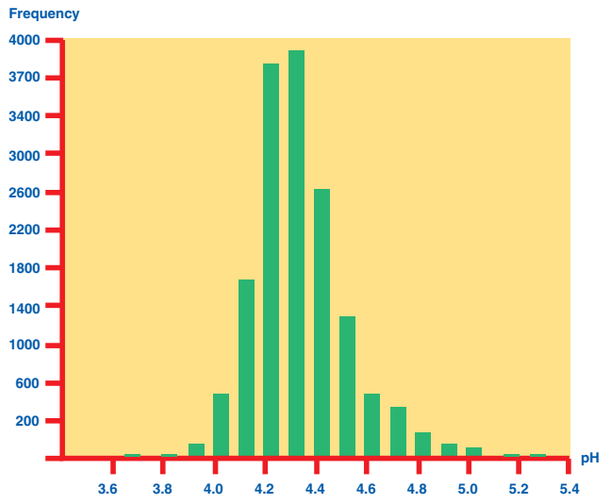


Figure 10. *pH Variations in Tomatoes*

Organic acids and sometimes salt (NaCl) and other spices are needed in the formulations for other tomato-based products. In particular, acids of small molecular size (acetic acid) may cause problems (corrosion of the aluminium foil) in laminated packaging material structures. Special inner coatings are required to prevent this defect.

5. Cranberry Juice

The production of cranberry juice is illustrated in figure 11. The composition of the resulting juice is shown in table 9.

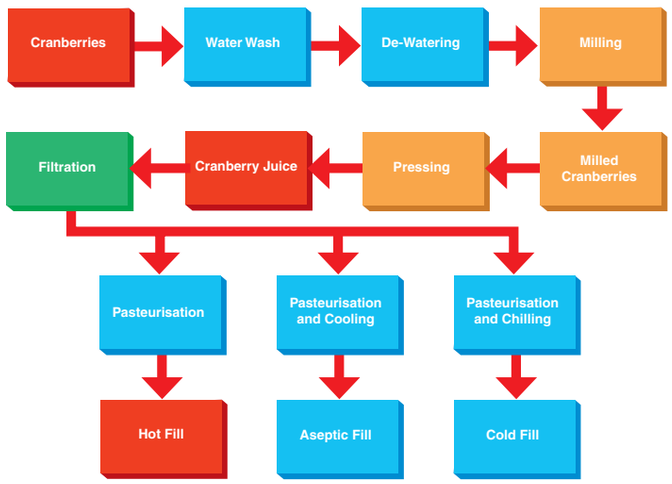


Figure 11. *Flow Chart: Production of Cranberry Juice*

Component	Per cent (%)
Water	93.0
Carbohydrate	6.8
Ash	0.2
Fat	<0.1
Protein	<0.1
Vitamin C	2 mg/100 ml
Crude Fibre	0.0

Table 9. *Composition of Cranberry Juice*

6. Strawberry, Raspberry and Blackberry Juice

Because of similarities in processing procedures, strawberry, raspberry and blackberry juice are presented together. The production of long-life juice is shown in figure 12.

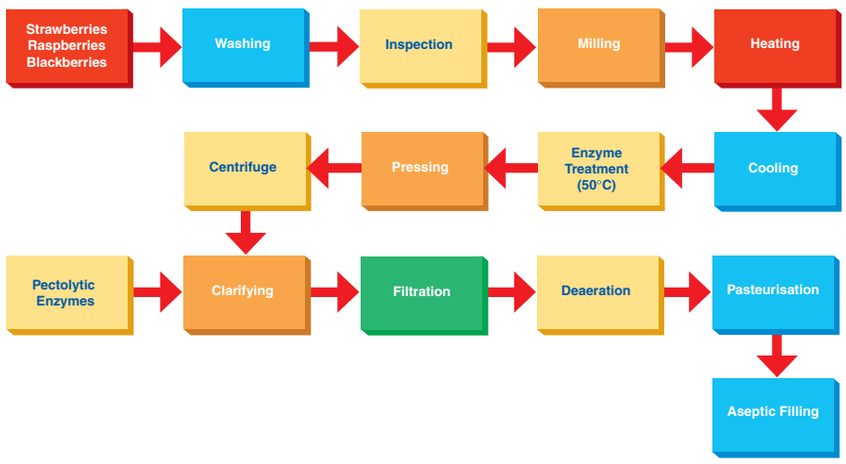


Figure 12. *Flow Chart: Production of Long-Life Strawberry, Raspberry and Blackberry Juice*

Table 10 contains the composition of strawberries and raspberries, while table 11 reveals the vitamin content of strawberries, raspberries and blackberries. The mineral content of these berries is given in table 12.

Component	Strawberry Per cent (%)	Raspberry Per cent (%)
Water	91.57	89.40
Protein	0.61	0.31
Fat	0.37	0.00
Carbohydrates	7.02	7.14
Energy (KJ)	127	124

Table 10. *Composition of Strawberries and Raspberries*

Vitamin	Strawberry Content/100 g	Raspberry Content/100 g	Blackberry Content/100 g
Ascorbic Acid	56.7 mg	25 mg	13 mg
Thiamine	0.02 mg	0.03 mg	0.048 mg
Riboflavin	0.066 mg	0.09 mg	0.05 mg
Niacin	0.23 mg	0.90 mg	0.359 mg
Panthothenic Acid	0.34 mg	0.24 mg	0.093 mg
Vitamin B_6	0.059 mg	0.057 mg	0.036 mg
Folacin	17.7µg		
Vitamin B_{12}	0 µg	0 µg	0 µg
Vitamin A	27 IU	130 IU	100 IU

Table 11. *Vitamin Content of Strawberries, Raspberries and Blackberries*

Minerals	Strawberry Content/100g	Raspberry Content/100g	Blackberry Content/100 g
Calcium	14 mg	22 mg	6 mg
Iron	0.38 mg	0.57 mg	0.17 mg
Magnesium	10 mg	18 mg	5 mg
Phosphorus	19 mg	12 mg	10 mg

Table 12. *Mineral Content of Strawberries, Raspberries and Blackberries*

7. Pineapple Juice

7.1 Quality of Raw Material

Both the US and the Codex Pineapple Juice Standards specify the use of "sound, ripe" pineapples. Sound is interpreted to mean the absence of cracked, split, diseased or fermented fruit.

7.1.1 Holding Time Before Processing

Short Storage Time!

Although some tropical fruits are, or almost are, sterile when protected by an unblemished exterior skin or shell, pineapples are not. At ambient tropical temperatures, pineapples must be transported and processed promptly to prevent the fruit from fermenting or decaying. In the processing plant, the problem becomes potentially worse after juice extraction.

Thus, processing within 24 hours after harvesting and final packaging within two hours after juicing are recommended.

The pineapple has a characteristic flavour with a delicate aroma and a pleasing sweet/sour taste. The fruit when ripe has a soluble solids content of 14°Brix and an acid content of 0.75%. Fruit which has a Brix/acid ratio of approximately 20 produces a high quality juice which is usually a by-product (233). Pineapple juice is produced from the fruit pulp that adheres to the skin, the core parts, clean parts of the trimmings, undersized fruit and "drain juice".

7.1.2 Product Standards

United States: single-strength pineapple juice is regulated by:

a) the mandatory Standards of Identity, Quality, and Fill of Containers of the Food and Drug Administration; and

b) the voluntary Standards for Grades of Pineapple Juice of the US Department of Agriculture - Agricultural Marketing Service.

Worldwide: single-strength pineapple juice is covered by the voluntary FAO/WHO Codex Alimentarius Commission 1981 Codex Standards for Pineapple Juice Preserved Exclusively by Physical Means, and by standards promulgated by individual countries.

7.1.3 Quality Control/Assurance

The appropriate legal standards, including test methods, imposed on pineapple juice prepared from fresh fruit or concentrate may vary from the basis of a quality control or quality assurance programme. The main objective is to produce pineapple products that meet these standard specifications. Disposition of products that do not meet the standards include:

a) downgrading of product quality declared on the label;

b) reprocessing;

c) donating product to charity; or

d) discarding.

To ensure that minimum standards are met for the production lot, the following checks are necessary at regular intervals:

a) °Brix;

b) percentage total acidity;

c) Brix/acid ratio;

d) percentage insoluble solids;

e) container fill;

f) colour (subjective);

g) flavour (subjective);

h) defects (subjective);

i) mould count, using the Howard Mould Count Method;

j) vitamin C content;

k) dimethylpolisiloxane (antifoam) content;

l) percentage added sweetener;

m) dissolved metal content: arsenic, copper, iron, lead, tin and zinc;

n) ethanol content;

o) sulphur dioxide content;

p) product pH;

q) processing parameters; and

r) packaging conditions.

Component	Content
Total Sugar	7.5 - 18.4 %
Acidity (Citric Acid)	0.60 - 1.62 %
Vitamin C	8 - 165 mg/100g
β–Carotene	0.16 - 0.29 mg/100g
Phenolics	3 - 7 mg/100g
Juice Yield	72%

Table 13. *Composition of Pineapples (223)*

7.2 Composition

The composition of pineapples is shown in table 13. Very large variations can be observed in the vitamin C and sugar content.

7.3 Production of Concentrate

Pineapple juice concentrate is available on the market. It has to be handled carefully because it easily develops a brownish colour defect, probably caused by oxidation.

Figure 13 illustrates the processing steps involved in the production of pineapple juice concentrate.

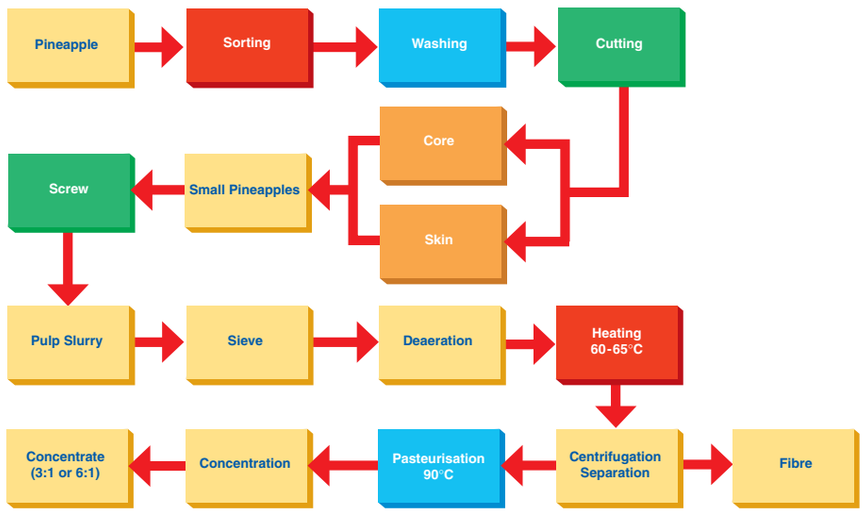


Figure 13. *Flow Chart: Production of Pineapple Juice Concentrate*

Volatile flavour material is stripped from the juices in the concentration process. The product is intensified a hundredfold and added back to the pineapple concentrate (223).

7.4 Microbiology

Some strains of *Byssochlamys* are capable of producing patulin, a mycotoxin of considerable potency. Patulin has also been confirmed in pineapples.

Byssochlamys fulva!

The soil-borne *Byssochlamys fulva* presents unique problems because thermal processes of 100°C or less that are typically used with acid foods including pineapples, do not destroy its chlamydospores. Increasing the severity of the process is considered an unsuitable alternative due to the resulting poor quality of the product. The best method for preventing spoilage is to prevent mould growth on equipment, transport containers and fresh fruit.

Spoilage of pineapple fruit concentrate and juice by heat-resistant *Talaromyces* has also been reported. The organisms had comparable $D_{90°C}$ values of 2 to 8 minutes depending on the strain, with an approximate z-value of 10.3°C.

Component	Content
Total Sugar	7.4 - 13.3 %
Acidity (Citric Acid)	2.40 - 4.80 %
Vitamin C	22 - 70 mg/100g
β–Carotene	1.07 - 1.70 mg/100g
Phenolics	3 - 6 mg/100g
Juice Yield	22 - 53 %

Table 14. *Composition of Passion Fruit*

8. Passion Fruit

The passion fruit is small and oval, with a hard leathery skin. Two types of fruit are generally cultivated: the yellow and the purple passion fruit. The latter is the more commonly used in the production of juice due to its intensive flavour. The juice is high in acidity while the °Brix/acid ratio is very low, in the order of 5 (223).

Table 14 shows the composition of passion fruit (16).
The processing steps involved in the production of passion fruit concentrate are illustrated in figure 14.

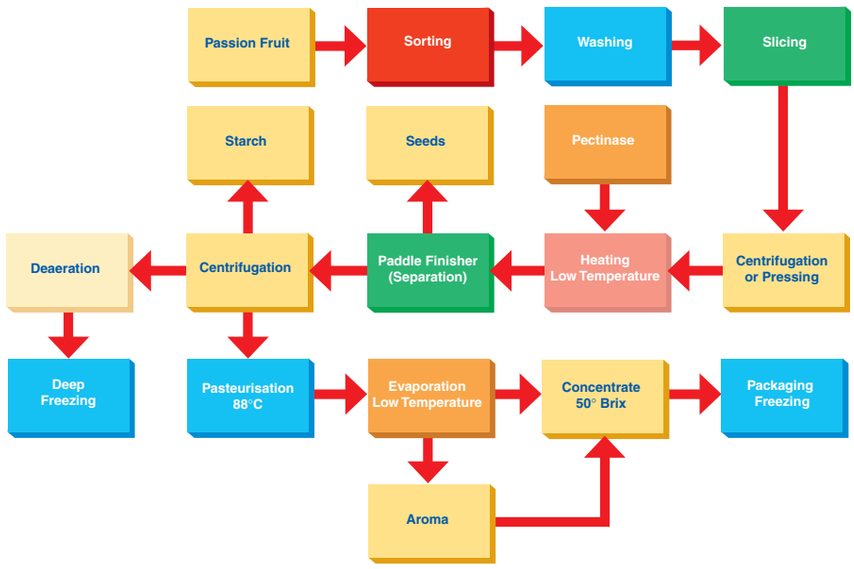


Figure 14. *Flow Chart: Production of Passion Fruit Concentrate*

The low temperature treatment in combination with pectinase break the juice sacks, reduce the juice viscosity and increase the yields. The distinctive aroma of passion fruit is very sensitive to heat treatment. Deaerated single-strength juice is therefore preferably preserved by freezing without heat treatment (223).

9. Mango Juice

The mango fruit is low in acidity with a °Brix/acid ratio of about 40. Its vitamin C content is moderately high. Mango fruit has a pronounced flavour with a distinctive taste and a very attractive yellow colour (223).

The composition of mango fruit (223) is shown in table 15.

Concentrates of mango fruit are available as a frozen pulp product. Typical manufacturing steps involved are illustrated in figure 15.

Component	Content
Total Sugar	10.0 - 15.0 %
Acidity (Citric Acid)	0.20 0.70 %
Vitamin C	10 - 100 mg/100g
ß–Carotene	2 - 6 mg/100g
Phenolics	32 - 75 mg/100g
Juice Yield	31 %

Table 15. *Composition of Mango Fruit*

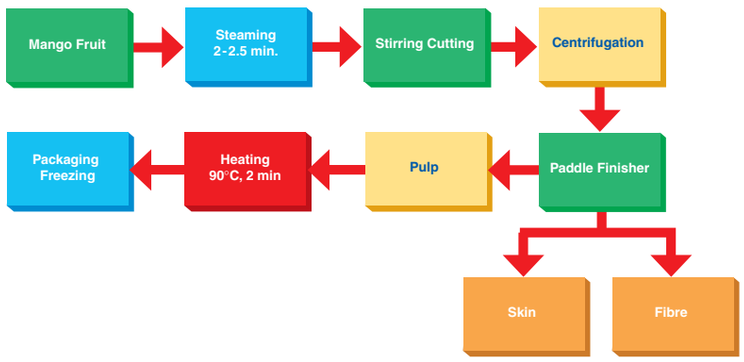


Figure 15. *Flow Chart: Production of Mango Pulp*

Mango juice concentrate has been prepared experimentally by freeze concentration or vacuum evaporation of the entire pulp, as well as by pulp-serum separation followed by freeze concentration.

Component	Content
Total Sugar	3.0 - 11.0 %
Acidity (Citric Acid)	0.36 - 0.66 %
Vitamin C	200 - 700 mg/100g
β–Carotene	2 - 6 mg/100g
Phenolics	37 mg/100g
Juice Yield	25 - 30 %*

* without enzyme treatment

Table 16. *Composition of Guava Fruit*

10. Guava

Guava juice has an average °Brix/acid ratio of 20 with low pH values in the order of 3.2. The low pH and the high phenolics content explain the rather sour, astringent taste. The fruit is a good source of vitamin C, see table 16 (223).

The quality attributes of guava puree which are monitored are flavour, colour, acidity, soluble solids and pH. Because of the wide variation between guava varieties and regional preferences, there is no singular quality standard for guava puree or juice.

In figure 16, typical steps for the production of guava juice are given.

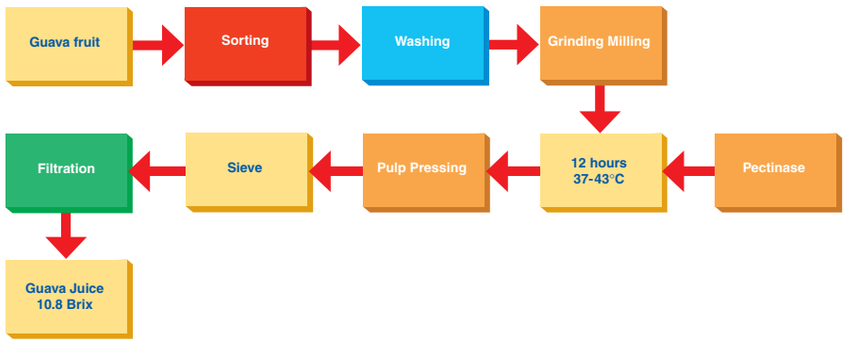


Figure 16. *Flow Chart: Production of Guava Juice*

The composition of the guava fruit (223) is shown in table 16.

The traditional method for increasing juice yield involves boiling the fruit pulp before pressing. Such a process spoils the natural flavour and destroys valuable nutrients such as vitamin C. A newer method is treatment with pectolytic enzymes. The 12-hour treatment can be shortened to 90 minutes by increasing the amount of pectolytic enzyme added. This may also increase the juice yield to about 77%. Processing without peeling has been recommended because of the high vitamin-C content of the peel (223). Concentration may lead to processing problems due to the high viscosity of the product.

Component	Content
Total Sugar	9.0 - 10.0 %
Acidity (Citric Acid)	0.05 - 0.10 %
Vitamin C	18 - 180 mg/100g
β–Carotene	0.06 - 0.24 mg/100g
Phenolics	3 - 6 mg/100g
Juice Yield	70 %

Table 17. *Composition of Papaya Fruit*

11. Papaya

Papaya has a low-acid content resulting in a °Brix/acid ratio of more than 100 and pH values between 5.0 and 5.5. Most of the papaya crop is consumed as fresh fruit. Only a small amount is processed into puree (223).

The composition of papaya fruit (223) is given in table 17.

Attributes for which papaya puree is generally analysed during quality control are: pH, total acid, odour, flavour, colour and black speck count. The black specks are particles from the spicules of the seed coat.

Black Specks: ten millilitres of puree are placed on a Kontes ground glass, thin-layer, chromatographic plate. A clear glass plate is positioned on top which presses the puree into a layer about 0.25 mm thick. The plates are placed on a light box fitted with a green filter. The black specks are counted within one square inch. The generally accepted range for black specks is 0.0 to 1.0 per square inch.

Figure 17 shows the typical production steps involved in the manufacture of papaya puree.

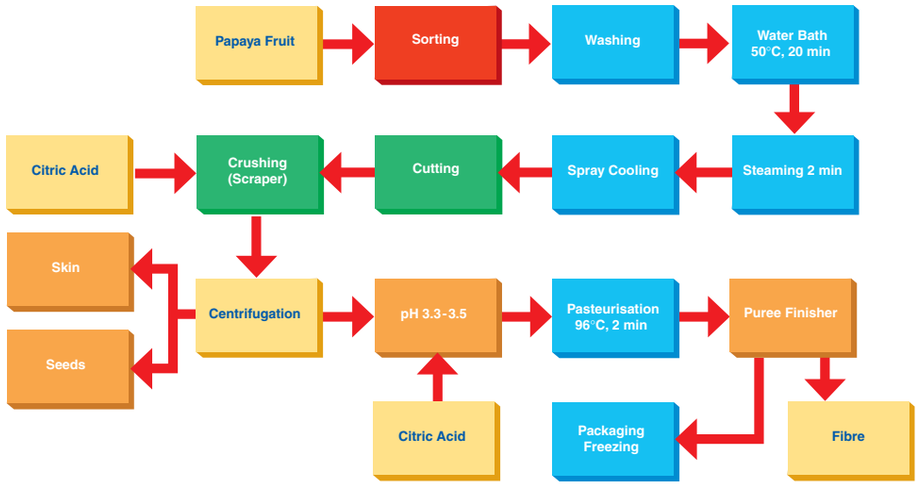


Figure 17. *Flow Chart: Production of Papaya Puree*

Pasteurisation (96°C for two minutes) is necessary to inactivate the enzymes present in the fruit. During storage, off-flavour development and gelation occurs in papaya puree which has not been acidified and sufficiently heat-treated.

12. Cashew Apple

Cashew apple is the flesh to which the cashew nut is attached. The fruit is grown for the nut. Cashew apple is therefore considered to be a waste material.

The composition of cashew apples (223) is shown in table 18.

Component	Content
Total Sugar	8.4 - 21.0%
Acidity (Citric Acid)	0.33 - 0.64 %
Vitamin C	15 - 455 mg/100g
β–Carotene	0.12 - 0.36 mg/100g
Phenolics	60 - 200 mg/100g
Juice Yield	55 - 80 %

Table 18. *Composition of Cashew Apple*

Cashew apple juice has a characteristic flavour with a °Brix/acid ratio of approximately 20. A high tannin content makes the juice very astringent. The fruit deteriorates rapidly and must be processed as quickly as possible. The juice obtained (without enzyme treatment) has a soluble solids content of 9 to 12.5 °Brix.

In figure 18, the typical processing steps involved in the manufacturing procedures of cashew apple juice are presented.

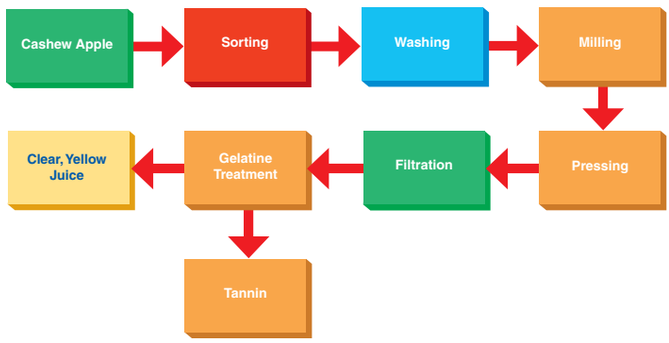


Figure 18. *Flow Chart: Production of Cashew Apple Juice*

Component	Content
Peel	27.80 %
Yield	61.50 %
pH	4.60 %
Total Soluble Solids	17.00 %
Titrable Acidity	0.28 %

Table 19. *Composition of Litchi Juice*

13. Litchi Juice

To obtain a non-bitter litchi juice, peeling is necessary prior to pressing. The juice must be pasteurised after pressing to retard further microbiological deterioration. A clear juice is obtained only by centrifugation and filtration, since pectolytic and cellulolytic enzyme preparations do not appear to assist the clarification process (211).

19. Soya Bean Products

Summary

Soya bean products in general and soya bean drinks in particular are gaining in consumer interest. Long-life soya bean drinks have been on the market for a long time, mainly in the Far East. Traditionally, Chinese people have consumed large quantities of soya bean products including drinks. To the European palate, the "beany" taste of soya bean drinks has been a hinder to its acceptance. Modern technologies have developed procedures by which this "beany" flavour can be eliminated or reduced to a sufficient degree.

Another problem, flatulence, is associated with oligosaccharides present in soya beans. The content of these can be reduced to an acceptable level. Today, soya bean drinks are accepted even by the European consumer.

1. General

Soya Bean Production and Consumption

From a total of 95 million tons of soya beans produced worldwide, 70.5% were utilised as animal feed, 13.7% for oil, 8.4% for lecithin, 5.3% for human consumption and 2.1% to produce edible proteins (71).

Soya drink production aims at manufacturing a consumer-acceptable, nutritious, good quality product with a high yield and at low cost. Soya drinks should be promoted as products in their own right made from soya beans. They should not be disguised as pseudo-products or as an imitation of dairy products (99).

Soya beans are processed by two different procedures (71).

1) In the "western procedure", the components are extracted and separated. Hydrocarbons are used for fat extraction; the proteins are extracted with alkaline and subsequently precipitated by acid.
2) The "Far Eastern method" is based on water extraction of the components which are processed together.

Soya Bean Drinks

A soya bean drink is the water extract of soya beans, a white-coloured emulsion which, in appearance, resembles cow's milk. Traditionally, the product is obtained by soaking soya beans overnight, followed by wet grinding and filtering through a cloth. The product has an undesirable beany flavour which can be eliminated by modern technology so that an organoleptically enjoyable product can be produced.

2. Processing

Large-scale industrial production of soya drinks consists of 11 steps (99) as shown in the flow chart in figure 1.

In the following section, these 11 process steps are discussed in some detail.

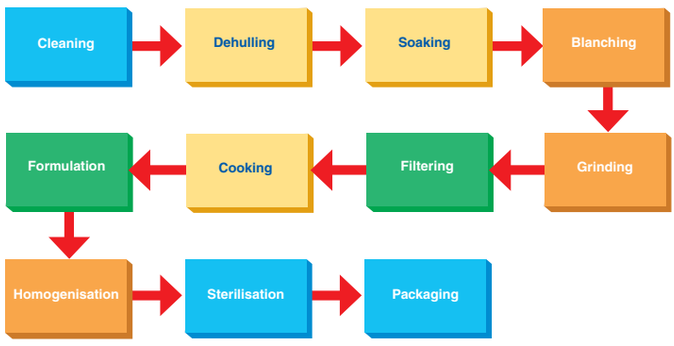


Figure 1. *Flow Chart: Production of soya Bean Drinks*

2.1 Cleaning

Washing Procedure

Commercial soya beans contain microorganisms, dirt, and dust on their surfaces. Foreign materials such as straw, stones, metals and weeds should be removed. Just as important is the removal of damaged soya beans.

It is an advantage to wash the soya beans before they are processed. The beans swell to a very high degree when immersed in water. Washing should be done at ambient temperature (~ 30°C) for a minimum of two hours, but for not more than five hours. Separating is necessary to remove stones, etc., from the soya bean material.

2.2 Dehulling

Dehulling refers to the removal of the outer seed coat. The hull constitutes about 9 % of the whole soya bean by weight. Soya bean drinks can be produced without de-hulling, but the advantages of dehulling are:

a) improved protein recovery;

b) improved soya drink flavour by the elimination of a beany taste and a bitter flavour component;

c) improved digestibility;

d) a slightly whiter colour;

e) reduced oligosaccharides; and

f) reduced bacterial count.

In the dry-grinding process, the beans are cracked on rolls and then passed over a multi-deck screen shaker with subsequent dehulling. The beans are dry-heated at about 65°C. At higher temperatures, denaturing of the protein would start. The hulls are separated from the beans by air in a cyclone.

The soya bean dehulling process consists of three steps (figure 2).

a) *Heating:* breaks the bonds between hulls and cotyledons: for instance, at 93°C for 15 minutes.

b) *Cracking:* is often done by a roller and a stationary concave blade; efficiency is up to 88%.

c) *Separation:* an air blower is used to remove the hulls from the remainder of the beans. In wet dehulling, the steps are essentially the same but the hulls are removed by running water.

Figure 2. *Dehulling of soya beans*

2.3 Soaking

Soaking Conditions

Soya bean drinks can be made from either soaked or dry beans. Soya beans can be soaked in about three times their weight of water. Typically, the soaking time is 8-10 hours at 20°C. The soaking times are shorter at higher temperatures. After proper soaking, soya beans will expand about 2.0 to 2.3 times their original weight. Soaking results in a reduced energy requirement for grinding, enables better dispersion and suspension of solids during extraction, increases the yield and decreases cooking time.

For soaking, alkaline solutions (sodium bicarbonate, sodium citrate, etc.,) have been used, resulting in the following benefits:

- reduction (but not elimination!) of the beany flavour;
- tenderisation of the soya beans giving shorter cooking times and better homogenisation;
- reduction of the oligosaccharide content; and
- acceleration of the inactivation of the trypsin inhibitor.

At soaking temperatures above 45°C, the total solids content decreases from 60% to 55%. There is also a small decrease in the recovery of fat and protein.

Dehulled soya beans reach full hydration much faster than whole soya beans: only 2-3 hours are required at 30°C or 1 hour at 50°C, compared to 7 hours at 30°C, and 3-4 hours at 50°C for whole beans.

2.4 Blanching

Blanching or steaming soya beans in a solution of sodium bicarbonate at high temperatures starts to inactivate the beany flavour. It also washes out all water soluble oligosaccharides and inactivates trypsin inhibitors.

2.5 Grinding

Grinding may be done with hot or cold water to obtain a 150 mesh colloid solution. Grinding in a hot water solution at > 80°C will inactivate the lipoxydase, thus preventing formation of the beany flavour.

To produce a slurry, the soya bean flour is suspended in water adjusted to pH 3.5 and heated to about 97°C. A holding time of about 10 minutes is sufficient to inactivate the lipoxygenase enzyme and to reduce the flatulent properties by dissolving the carbohydrates.

2.6 Filtering

The insoluble soya residues are removed from the slurry by a decanter centrifuge to improve flavour, texture and to dispose of oligosaccharides.

2.7 Cooking or Heating

The purpose of cooking a soya bean drink is to destroy microorganisms and to improve flavour. Heating to 100°C for 8 to 22 minutes resulted in 80%-90% inactivation of the trypsin inhibitor. It has been suggested that the soya slurry be heated to 100°C for 30 minutes to obtain optimal nutritional value and flavour. Heating also lowers the viscosity, improves extraction and results in higher yields of protein and solids.

Trypsin Inhibitor

2.8 Formulation

One way to increase acceptance of soya bean drinks is through an appropriate formulation that uses sweetening and a variety of flavouring agents. The addition of soya oil, lecithin or emulsifiers improves the richness of a soya bean drink. Another common additive is cow's milk (~20%). Nutritional fortification can be made by adding amino acids (methionine), vitamins and minerals.

2.9 Homogenisation

Homogenisation is necessary to stabilise the fat (oil). It will also make the soya bean drink creamier. Homogenisation at 90°C and 2,000 to 3,500 psi (200 to 350 kg/cm^2) is usually sufficient.

2.10 Sterilisation

A soya bean drink is an ideal medium for microbial growth. It can therefore spoil easily and rapidly due to the survival and subsequent multiplication of bacteria and fungi from inadequate heat treatment during processing.

a) Pasteurisation: heating to ~ 72°C for 15 seconds destroys all vegetative disease-causing microorganisms.

b) Sterilising (retorting): typically, heating to ~ 121°C for 15 to 20 minutes to kill all heat-resistant moulds, bacteria and their spores. The shelf life of the product is about six months or more.

c) UHT treatment: both direct and indirect heating can be used, though the direct systems (steam injection) are often preferred. Heating to 140-145°C with a holding time of ~ 4 seconds is adequate. If combined with aseptic packaging, a shelf-stable product is obtained with a shelf life of three months or more.

2.11 Packaging

Packaging requirements depend on the kind of drink produced and the demands for shelf life connected with processing. The full benefit of the UHT treatment is only encountered if the soya bean drink is packaged under aseptic conditions. The shelf life is about 3 months or more if the package provides protection against the entry of light and oxygen.

3. Quality Improvement

The following methods can be used to improve the quality of soya bean drinks (99).

3.1 Methods for Eliminating the Undesirable Beany Flavour

Beany Flavour

The beany flavour in soya drinks is caused by the activity of lipoxygenase on fat. This enzyme can be inactivated by adjusting the pH to either below pH 3 or above pH 10, or at high temperatures > 80°C. Soya bean blanching (100°C for 10-30 minutes) and hot grinding (> 80°C) are two methods widely used to remove the beany flavour.

Lipoxygenase

Hot-grinding the whole-soaked soya beans with boiling water or steam to yield a soya slurry with a temperature of 80°C or above, followed by holding the slurry at this temperature for 10 minutes, inactivates lipoxygenase which causes the beany flavour.

Blanching in boiling water for 10 minutes or placing the soya beans directly into boiling water for 20 minutes inactivates lipoxygenase. The addition of 0.25%-0.5% sodium bicarbonate to the water used for blanching or soaking has also been used. The soya beans are then drained and ground with water to produce a soya drink of little beany flavour.

Using defatted soya flour (elimination of soya oil from the product which will be acted on by lipoxygenase): 40-mesh full-fat soya flour is extracted with 95% ethanol followed by a mixture of equal volumes of 95% ethanol and n-hexane. The solvents are filtered off, followed by drying at 40°C under vacuum.

Deodorisation

Vacuum deodorisation (removes volatile off-flavour): passage of the soya drink through a vacuum pan at high temperature and expansion cooling in direct UHT-treatment systems.

Using *isolated soya protein* removes lipoxygenase. An acceptable soya drink can be prepared from isolated soya protein by adding water, sugar, emulsifier, refined vegetable oil, flavours, vitamins and minerals.

Removal of Beany Flavour

Enzyme fermentation (eliminates off-flavour): the beany flavour can be removed by fermentation with *Lactobacillus acidophilus*, *Aspergillus oryzae* or *Rhizopus oligosporus*.

Adding flavour: flavouring with vanilla, cow's milk, egg, chocolate, honey, etc., is an effective way of masking the beany flavour.

Alkaline soaking and/or blanching (inactivates lipoxygenase): soaking and/or blanching in 0.5% $NaHCO_3$ improves the soya drink flavour while removing oligosaccharides and lowering the cooking time.

Acid-grinding (inactivates lipoxygenase): at pH below 3, the lipoxygenase will be inactivated and no beany flavour will develop.

Dehulling (prevents bitter taste).

Direct UHT treatment and expansion cooling remove volatile off-flavours and thus correct the flavour; the mild heating reduces loss in nutritional value.

4. Methods for Inactivating of Anti-Nutritional Factors (Trypsin Inhibitors)

Inactivation of trypsin inhibitors can be achieved by heating soya slurry at 100°C for 30 minutes or at 110°C for 22 minutes. In addition, the activity of trypsin inhibitors can also be reduced by:

1) various heating processes; and
2) alkaline soaking.

4.1 Methods for Removing Oligosaccharides (Raffinose and Stachyose)

Whole soya beans contain 5% sucrose and 5.1% of other sugars (arabinose, glucose, verbascose, etc.,). In addition, two oligosaccharides, raffinose (1.1%) and stachyose (3.8%), are present in soya beans. Flatus is caused by the fermentation of sugars of low molecular weight - raffinose (galactose-glucose-fructose) and stachyose (galactose-galactose-glucose-fructose). The flatulence caused by these oligosaccharides can be reduced by:

1) alkaline soaking;
2) fibre removal;
3) heat treatment;
4) enzyme treatment (use of the enzyme a-galactosidase): the enzyme soya drink mixture is incubated for 3 hours at 55°C, then boiled for 10 minutes to stop the enzyme reaction.

5. Soya bean Drinks

Soya bean drinks manufactured in the traditional way suffer from a number of drawbacks. They have a strong unpleasant smell and are difficult to digest. The smell is caused by the action of an enzyme, the bean-lipoxydase.

Depending on the formulation, and in particular the addition of sugar, soya bean drinks can show a wide variation in composition (table 1).

The processing conditions in general and pre-processing in particular depend on the kind of product that is to be produced and the raw materials (ingredients) that are to be used.

Component	Soya Bean	Soya Drink	Soya Drink
Carbohydrates	38%	1.5-3.1%	10%
Minerals	4%	0.5-0.6%	
Protein	40%	4.0-4.4%	1.5-2.0%
Oil	18-20%	2.5-2.6%	1.0-1.5%
Water		89.7-89.5%	86.5-87.5%

Table 1. *Composition of Soya Beans and Soya Bean Drink*

The typical composition of a soya bean drink (1,000 kg) is:

- 44 kg of soya beans;
- 88-100 kg sugar; and
- about 850 litres of water.

Soya Bean Drinks are *not* Infant Formulations!

"I must emphasise strongly that the use of soya drink products as infant formulations or as the sole source of nutrients can result in severe protein-calorie malnutrition, multiple vitamin and mineral deficiencies, and ultimately death if not diagnosed and treated promptly" (83).

6. Tofu and Sufu

6.1 General

Tofu (soya bean curd) was developed in China. It is made by the precipitation of soya protein from soya drink by a coagulant such as calcium sulphate ($CaSO_4$). Soya bean protein coagulates if (71):

1) the pH is < 5.5;
2) calcium sulphate ($CaSO_4$) is added at a temperature of > 60°C;
3) the protein concentration exceeds 5% and the temperature 100°C; and
4) the solution is frozen.

Tofu is an excellent protein food made from vegetable protein curd - normally soya beans. Tofu has the advantages from a health standpoint of being high in protein, low in saturated fats and reasonably low in calories. It has a bland flavour and, if made from vegetable protein, is free of cholesterol (83).

Fresh tofu can spoil quickly if not refrigerated and kept covered with fresh water. Even under the best storage conditions, fresh Tofu will not maintain its quality for an extended period of time, with shelf life usually considered to be not more than a week to 10 days (99).

6.2 Production Line for Standard Tofu

In the industrial manufacture of Tofu, a number of process steps are involved (figure 3). Starting from a soya bean drink to which a coagulant is added, the mixture is stirred and heated to boiling. Liquid is removed by filtration. After cooling, the Tofu is ready for sale. Such products are sold in large quantities by street vendors in Japan. Today, shelf-stable long-life Tofu is produced in some countries (Japan and the United States).

Long-Life Tofu

In this technology, the soya bean drink is sterilised by UHT treatment. Subsequently, and under aseptic conditions, the sterilised (by filtration) coagulant is added. The product is packaged aseptically and heated to about 80°C by passage through a water bath. Coagulation takes place at this stage.

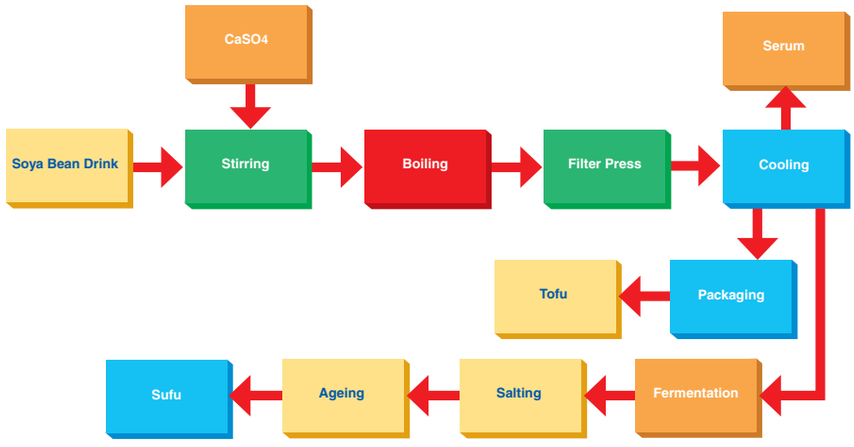


Figure 3. *Flow Chart: Production of Tofu and Sufu*

7. Examples

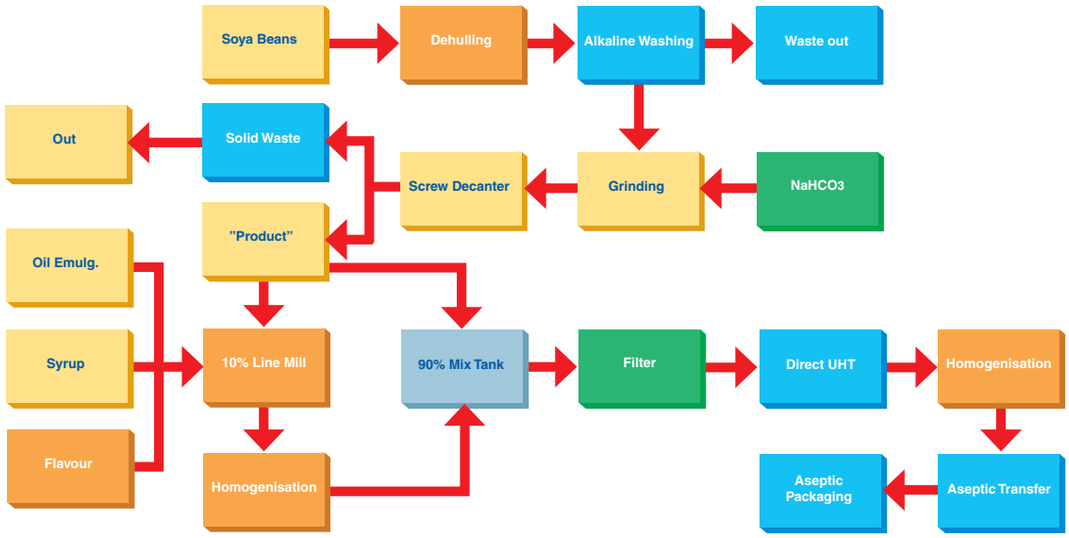


Figure 4. *Flow Chart: Production of Long-Life soya Bean Drinks*

1) *Line mill*: palm oil and emulsifier are added at 80°C and 1,800 rpm which is subsequently increased to 3,600 rpm during the addition of cane sugar syrup and flavouring.
2) *First homogenisation*: 170-180 kg/cm^2, 60°C in two stages.
3) *Filtration*: bucket filter, 80 mesh.
4) *UHT:* ~135°C.
5) *Second homogenisation*: 170-180 kg/cm^2, 85°C.

The total process from grinding to aseptic filling takes no more than 3 to 4 hours.

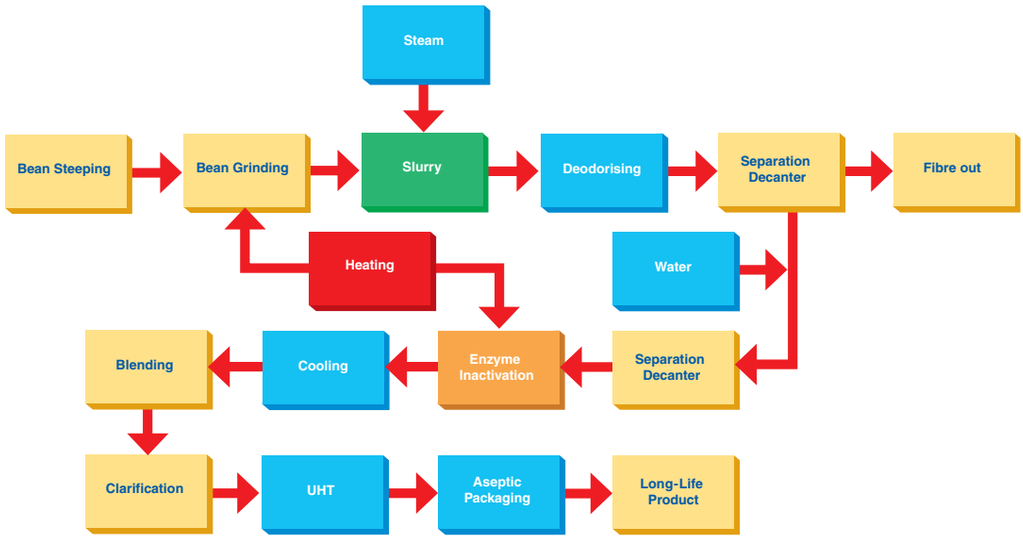


Figure 5. *Flow Chart: Production of a Clarified, Long-Life soya Bean Drink*

Soya bean seeds, removed from their pods but still in their hulls, are steeped and ground and then mixed with water to make a slurry. After enzyme inactivation and deodorisation (steam injection, time-temperature and vacuum treatment), the slurry goes through a set of centrifugal separators; the separated fibre residues may be used for animal feed or similar purposes. In the blending section, sugar, fat, flavouring, colouring, etc., are added to give the product its character and final composition. After clarification and UHT treatment, which includes homogenisa-

tion (typically ~ 57°C, 200-350 kg/cm^2, two stages), the product is aseptically packaged and ready for distribution. If soya flour instead of soya bean seeds is used as a raw material, the steeping and grinding sections are omitted, and the line begins with a slurry mixer.

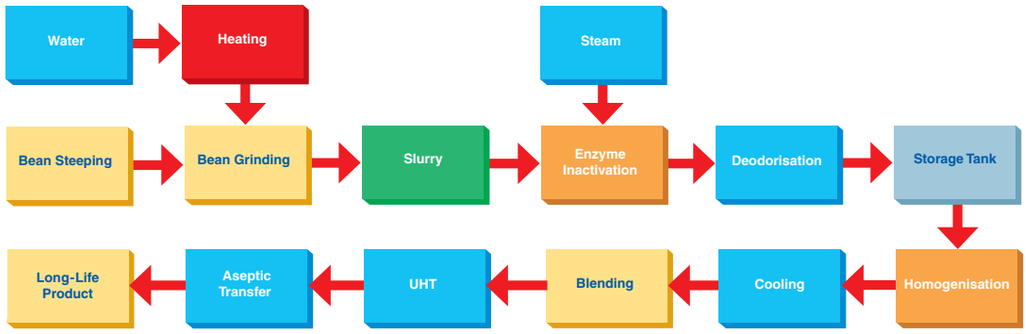


Figure 6. *Flow Chart: Production of a Suspended, Low-Bean Flavour, Long-Life Soya Bean Drink*

If the final product is to be suspended, i.e., retain the fibres, the soya bean seeds must be dehulled prior to grinding. Dehulling can be done by soaking the beans in a 0.2% sodium bicarbonate ($NaHCO_3$) solution for about 3 to 4 hours.

Typical sterilisation conditions (UHT treatment) are 138-145°C with an average holding time of four seconds. Direct and indirect systems are used.

20. Literature

1. Adams, D.M.: Inactivation of *Clostridium perfringens* Type A Spores at Ultrahigh Temperatures; Applied Microbiology, **26**, 282-287, 1973.
2. Aggarwal, M.L.: Commercial Sterilization and Aseptic Packaging of Milk Products; J. Milk Food Technol. **37**, 250-255, 1974.
3. Amati, A.: Versuche zum Abpacken von Wein in Behältern aus "Tetra Brik"; Verpackungs-Rundschau, technisch-wissenschaftliche Beilage, **35**, 7-10, 1984.
4. Anderson, A.M.: Recombined Milk Products: Nutritional Aspects and Role of New Products; International Dairy Federation, Seminar on Recombination of Milk and Milk Products, DOC 6, Singapore, 7-10 October, 1980
5. Anonymous: A Study of the Number and Types of Aerobic Mesophilic Spores in Milk before and after Commercial Sterilisation; J. appl. Bact., **19**, 46-53, 1956.
6. Anonymous: Eignung von H-Milch für die Säuglingsernährung; Welt der Milch, **22**, 367, 1968.
7. Anonymous: National Soft Drink Association (formerly American Bottlers of Carbonated Beverages), Quality Specifications and Test Procedures for Bottlers; Granulated and Liquid Sugar, Washington D.C. 1975.
8. Anonymous: Draft Code of Practice for the Quality of Water Used for the Reconstitution of Fruit Juices from Fruit Juice Concentrates; Economic Commission for Europe, Committee on Agricultural Problems, Joint ECE/FAO Codex Alimentarius group of experts on standardisation of fruit juices, Geneve, July 1976.
9. Anonymous: Staphylokokken waren die Ursache für die H-Milch-Vergiftung; Deutsche Milchwirtschaft, **30**, 1600, 1979.
10. Anonymous: General Dairy Code of Hygienic Practice for the Dairy Industry; International Dairy Federation, Document 123, 1980.
11. Anonymous: Pasilac Therm has Developed a Continous Sterilisation Plant Based on Indirect Heating; Nordeuropaeisk mejeri-tidskrift., **8**, 275-280, 1981.
12. Anonymous: Merkblatt 45: Prüfung von Heissiegelnähten auf Dichtigkeit; Verpackungs-Rundschau, **33**, 10-33, 1982.
13. Anonymous: Abhängigkeit der Anzahl unsteriler Verpackungen von der Ausgangskeimzahl des Packmittels und dem Keimabtötungseffekt des Packmittel-Sterilisationsverfahrens; Deutsche Molkerei-Zeitung, **103**, 1216, 1982.
14. Anonymous: The Three-Shift Machines; Food Engineering, **56**, 36-38, January/February, 1984.
15. Anonymous: Food Tehnical News, 19, December 1985.
16. Anonymous: Erfolgreiches Comeback für ein bewährtes Naturprodukt: Kur-Molke im aktuellen Gesundheits-Trend; Combibloc-Letter No 1/1985.
17. Anonymous: US Department of Health and Human Services; PMO Grade A Pasteurised Milk Ordinance, 1985.
18. Anonymous: Food Europe Germany; Food Europe, 28-29, October/November 1985.
19. Anonymous: Nordisk Tidskrift för Renlighetsteknik, nr 5, 1986.
20. Anonymous: New Flavour Enhancement Process Improves Taste of Fruit Juices and Concentrates; Modern Dairy, 8-10, August 1986.
21. Anonymous: Merkblatt: Prüfung von Aseptikanlagen mit H_2O_2-Packstoffsterilisationsvorrichtungen auf deren Wirkungsgrad; Verpackungs-Rundschau, **38**, 45-47, 1987.
22. Anonymous: UHT-Kaffeesahne, aseptisch verpackt; Deutsche Molkerei-Zeitung, **108**, 177-180, 1987.
23.Anonymous: High Temperature Resistance of Bacterial Spores; Dairy Industry International, **53**, 37-38, 1988.
24. Anonymous: Aseptische Prozesslinien bei der Hochwald Nahrungsmittelwerke GmbH; Deutsche Molkerei-Zeitung, **110**, 1188-1192, 1989.

25. Anonymous: ISO 9000 International Standards for Quality Management; SIS Förlag, Box 6455, 113 82 Stockholm, 1996.
26. Anonymous: Pectin; an information leaflet of the "The Copenhagen Pectin Factory LTD", DK 4623 Lille Skensved, Denmark.
27. Anonymous: Stabilisation of Fermented and Directly Acidified Sour Milk Drinks; leaflet of the "The Copenhagen Pectin Factory LTD", DK 4623 Lille Skensved, Denmark.
28. Anonymous: Whey Drinks; leaflet of the "The Copenhagen Pectin Factory LTD", DK 4623 Lille Skensved, Denmark. Technical Data, Bulletin 1107-2.
29. Anonymous: Grindsted Products A/S Edwin Rahrs Vej 38, DK-8220 Brabant, Denmark, Technical Memorandum TM 311-2e.
30. Anonymous: Product Application: 2. Fruit Juice; Tetra Pak International AB, Information Department, Fack, S-221 01 Lund Sweden.
31. Anonymous: US 21 CFR (US FDA): part 146.
32. Ashton, T.R.: The Planning, Building, Installation, Operation, and Maintenance of UHT Plants; International Dairy Federation, Malmö, Sweden, 24-27 May, 1971.
33. Ashton, T.R. and A.J.D. Romney: Quality Assurance; International Dairy Federation, Methods for Improving the Quality of Heat Treated Milk (proposed monograph by group B21), June 1978.
34. Ashton, T.R. and A.J.D. Romney: UHT Milk; International Dairy Federation, Cleaning and Sterilisation, (proposed monograph by group B21, Appendix I), June 1978.
35. Bach, P. and K.H. Hess: Der Einfluss einiger Portionspackungen auf den Wein im Laufe der Lagerung im Vergleich zur Glasflasche und unter Berücksichtigung der Lichteinwirkung; Neue Verpackung, 78-82, 1986.
36. Badings, H.T.: A Method of Preventing the Occurence of Cooked Flavour in Milk and Milk Products; Nordeuropaeisk Mejeri-Tidskrift, **4**, 379-384, 1977.
37. Badings, H.T. et al.: The Effect of Oxygen Content on Quality and Keeping Properties of Packaged, Pasteurised or Sterilised Milk and Milk Products; Nordeuropaeisk Mejeri-Tidskrift, **7**, 101-113, 1980.
38. Banks, W. et al.: The Formulation of Cream-Based Liqueurs; The Hannah Research Institute, Ayr, Scotland.
39. Banks, W. et al.: The Heat Stability of Milk-Its Effect on Products and Processes; NIRD Research News, Hannah, 2-4
40. Barach, J.T. et al.: Low Temperature Inactivation in Milk of Heat-Resistant Proteases from Psychrotrophic Bacteria; J.Dairy Science, **59**, 391-395, 1976.
41. Barach, J.T. et al.: Stabilization of a Psychrotrophic *Pseudomonas* Protease by Calcium Against Thermal Inactivation in Milk at Ultrahigh Temperature; Appl. Environmental Microbiology, **31**, 875-879, 1976.
42. Barron, W.: Controlling Cleaning Costs; Dairy Industries, 30-31, July 1988.
43. Baumgart, J. and G. Stocksmeyer: Hitzeresistenz von Ascosporen des Genus *Byssochlamys*; Alimenta, **15**, 67-70, 1976.
44. Bayliss, C.E. and W.M. Waites: The Combined Effect of Hydrogen Peroxide and Ultraviolet Irradiation on Bacterial Spores; J.Appl.Bact., **47**, 263-269, 1979.
45. Bayliss, C.E. and W.M. Waites: The Synergistic Killing of Spores of *Bacillus subtilis* by Hydrogen Peroxide and Ultra-Violet Light Irradiation; Microbiology Letters, **5**, 331-333, 1979.
46. Behringer, R. et al.: Sterilisierungsbedingungen für Milchkonzentrate; Deutsche Milchwirtschaft, **41**, 1190-1194, 1990.
47. Betz, W.: Zum aseptischen Abfüllen von Lebensmitteln in tiefgezogenen Verpackungen aus Kunststoff; Verpackungs-Rundschau, **19**, 1032-1036, 1968.
48. Bergmann, K. and O. Hövels: Klinische Prüfung der adaptierten, uperisierten Säuglingsnahrung Pomila; Kinderklinik, **119**, 559-562, 1971.
49. Berlage-Weining, L.: Sensorische Qualität und Vitaminwertigkeit von UHT-Milch und pasteurisierter Milch; Deutsche Milchwirtschaft; **34**, 1021-1023, 1983.

50. Berlage-Weining, L.: Untersuchungen über Qualitätskriterien von UHT-Milch und pasteurisierter Milch; Deutsche Molkerei-Zeitung, **104**, 1330-1334, 1983.
51. Bernhard, K. et al.: Die biologische Wertigkeit der uperisierten Milch, Bulletin der Schweiz.Akad.Med.Wiss., **9**, 313-324, 1953.
52. Berres, H.L.: New Approach in Wine Filtration; Die Weinwirtschaft, No 9. 1984.
53. Betz, W.: Zum aseptischen Abfüllen von Lebensmitteln in tiefgezogenen Verpackungen aus Kunststoff; Verpackungs-Rundschau, **19**, 1032-1036, 1968.
54. Bielig, H.J. et al.: Herstellung von Orangensaft und Orangensaftkonzentrat; ZFL, **26**, 140-143, 1975.
55. Blanc, B.: The Effect of Technological Processes on the Hygienic and Nutritional Value of Dairy Products, XX Int. Dairy Congress, Paris, 1978.
56. Blanc, B. et al.: Veränderungen biochemisher, technologischer und sensorischer Merkmale von UHT-Milch im Verlaufe der Lagerung; Alimenta-Sonderausgabe, 27-47, 53-60, 1980.
57. Blanc, B. et al.: Einfluss der Kühllagerung der Rohmilch mit und ohne Anreicherung psychrotropher Keime auf die Qualität von UHT-Milch; Alimenta, **23**, 71-79, 1984.
58. Blanc, B. and R. Sieber: Comparison of Nutritional Value of Raw, Pasteurised and UHT Milk on the Basis of Long-Range Studies on Rats; XX Int.Dairy Congress, Vol.E, 708-709, 1978.
59. von Bockelmann, B.: Aseptische Verpackungen für Milch und Milchprodukten; Deutsche Milchwirtschaft, **22**, 136-139, 1971.
60. von Bockelmann, B.: Aseptic Packaging of Liquid Food in Tetra Pak Systems: Microbiological Aspects; Supplement to South African Food Review, Feb/March, 1980.
61. von Bockelmann, B. and I. von Bockelmann: Aseptic Packaging of Liquid Food Products: A Literature Review; J.Agri.Food Chem., **34**, 184-193, 1986.
62. von Bockelmann, I and B. von Bockelmann: Bakteriologische Untersuchungen an verpacktem und gelargertem stillen Trinkwasser; Verpackungs-Rundschau, **23**, 1036-1039, 1972.
63. von Bockelmann I. and B. von Bockelmann: The Sporicidal Action of Hydrogen Peroxide-A Literature Review; LWT Report, **5,** 221-225, 1972.
64. von Bockelmann I, and B. von Bockelmann: Die Mikroflora in Papier; Alimenta, **13**, 181-183, 1974.
65. Boejgaard, S.E.: Recombination of Milk; Scan. J. Dairy Technol. and Know-How; 52-55, 1984.
66. Bosett, J.O. and M. Rüegg: Einfluss der thermischen Behandlung, der Entgasung und der Lagerung auf den Gefrierpunkt und die Dichte der Milch, Alimenta, **23**, 163-170.
67. Boto, K.G. and L.F.G. Williams: Rapid Determination of Trace Amounts of Hydrogen Peroxide; Anal. Chim. Acts, **85**, 179-183, 1976.
68. Brehmer, J. and L. Höppner: Tetraederverpackte Säuglingsmilch: ein Weg zur optimalen Säuglingsernährung im häuslichen Milieu; Kinderarzt, **5**, 244-248, 1974.
69. Brody, A.L. and D.E. Ryan: Food Canning in Rigid and Flexible Packages; CRC Critical Reviews in Food Technol., 187-243, 1971.
70. Brown, K.L. and C.A. Ayers: Thermobacteriology of UHT Processed Foods; (Literature review) Developments in Food Microbiology 1, R.Davies, Applied Science Publishers, London and New Jersey, 1982.
71. Brunner, J.C. and Z. Puhan: Milchprodukte-Sojaprodukte: Ein Vergleich der Technologie; Deutsche Molkerei-Zeitung, **110**, 416-421, 1989.
72. Buchberger, J.: Untersuchungen zum Gefrierpunkt der Milch; Deutsche Molkerei-Zeitung, **107**, 375-380, 1986.
73. Buchner, N.: Aseptisch arbeitende Thermoform-, Füll- und Schliessmaschine TFA 240 AS; Deutsche Milchwirtschaft, **27**, 494-496, 1976.

74. Buchner, N.: Die aseptische Verpackung von Lebensmitteln-Entwicklungsstand und Entwicklungsaussichten (I); Verpackungs-Rundschau, **29**, 788-796, 1978.
75. Buchner, N.: Die aseptische Verpackung von Lebensmitteln-Entwicklungsstand und Entwicklungsaussichten (II); Verpackungs-Rundschau, Interpack 78, Sonderausgabe.
76. Buchner, N.: Aseptisches Verpacken in Thermoform-. Füll- und Verschliessmaschinen; Deutsche Molkerei-Zeitung, **106**, 1250-1257, 1985.
77. Burton, H.: Ultra-High-Temperature Processing and Aseptic Packaging in the Dairy Industry; The Process: Its Basic Principles and Development, J. Soc. Dairy Technology, **18**, 56-65, 1965.
78. Burton, H.: Comparison of Milks Processed by the Direct and Indirect Methods of Ultra-High-Temperature Sterilisation. III. A Note on the Results for Spore Destruction obtained with an Experimental Ultra-High-Temperature Milk Steriliser; **37**, 227-231, 1970.
79. Burton, H.: A Comparison between Direct and Indirect UHT Processing Methods for Milk, Dairy Industries, **37**, 197-201, (April) 1972.
80. Busse, M.: Probleme der Betriebsberatung; Deutsche Molkerei-Zeitung, **95**, 1624-1631, 1974.
81. Busse, M.: Mikrobiologische Aspekte bei der H-Milch-Herstellung; Deutsche Milchwirtschaft, **26**, 1800-1802, 1975.
82. Busta, F.F.: Thermal Inactivation Characteristics of Bacterial Spores at Ultra High Temperatures; Applied Microbiology, **15**, 640-645, 1967.
83. Cal, A.: Tofu: A Nutritious, Healthful Food Gaining Wide Acceptance; Food Processing, May 1985.
84. Campden Food Preservation Research Association: Examination of Suspect Spoiled Cans and Aseptically Filled Containers; Technical Report No. **18**, 1-57, 1987.
85. Carlson, V.R.: Current Aseptic Packaging Techniques; Food Technol., **38**, 47-50, December 1984.
86. Cerf, O.: UHT Milk; International Dairy Federation, Methods for Improving the Quality of Heat Treated Milk (proposed monograph by group B21), June 1978.
87. Cerf, O. and J. Hermier: Diversite de la résistance des spores de *Bacillus* à l'eau oxygénée; Le Lait, **511/512**, 1-20, 1972.
88. Cerf, O. and F. Metro: Tailing of Survival Curves of *Bacillus licheniformis* Spores Treated with Hydrogen Peroxide; J.Appl.Bact., **42**, 405-415, 1977.
89. Cerney, G.: Wirkung von Wasserstoffperoxid auf Sporen von *Bacillus subtilits*; Naturwissenschaften, **62**, 299-300, 1975.
90. Cerney, G.: Entkeimung von Packstoffen beim aseptischen Abpacken: 1. Mitteilung, Untersuchungen zur keimabtötenden Wirkung konzentrierter Wasserstoffperoxid-lösungen; Verpackungs-Rundschau, **27**, 27-32, 1976.
91. Cerney, G.: Mikrobiologische Aspekte der Lebensmitteltechnologie und -verpackung; Verpackungs-Rundschau, **29**, 49-54, 1978.
92. Cerney, G.: Entkeimen von Packstoffen beim aseptischen Abpacken: 7. Mitteilung, Untersuchungen zur Sterilisierung von Packstoffoberflächen durch eine Kombination von Wasserstoffsuperoxid-Bedüsung und UV-C-Bestrahlung; Verpackungs-Rundschau, **36**, 29-32, 1985.
93. Cerney, G.: Sterilisieren von Verpackungsmaterialien; Deutsche Milchwirtschaft, **44**, 169-174, 1993.
99. Chen, St.: Principles of Soymilk Production; presented at AOCS sponsored short course on food uses of whole oil and protein seeds, Honolulu, Hawaii, May 12-14, 1986.
100. Cook, R.: Quality of Citrus Juices as Related to Composition and Processing Practices; Food Technology, **37**, 68-133, 1983.
101. Coster, D.R.A.: Aseptische Abfüllung in Papier und Kunststoff; Flüssiges Obst, 253-256, 1971.

102. Dachs, E.: Biologische Aspekte beim Verkauf von Wasser für den Haushalt; Brauwelt, **113**, 311-314, 1973.
103. Dahlqvist, A. et al.: Hydrolysis of Lactose in Milk and Whey with Minute Amounts of Lactase: J.Dairy Research, **44**, 541-548, 1977.
104. Dahlqvist, A. et al.: Hydrolyse von Laktose in Milch und Molke mit geringen Mengen von Laktase; Deutsche Milchwirtschaft, **29**, 1330-1337, 1978.
105. Damerow, G.: Bau- und Wirkungsweise von UHT-Anlagen, Deutsche Milchwirtschaft, **31**, 1456-1463, 1980.
106. Davis, J.G. and P.J. Stubbs: The Measurement of the Colour of Sterilised Milk, Dairy Engineering, September 1958.
107. Duong, H.A. and P. Vögele: Mikrobiologische Aspekte aseptisch verpackter Fruchtsäfte und stiller Getränke; Flüssiges Obst, 2/1984.
108. Dürr, H.A.: Aroma Quality of Orange Juice: A Brief Review; Alimenta, **19**, 35-36, 1980.
109. Dürr, P. et al.: Aroma Quality of Orange Juice after Filling and Storage in Soft Packages and Glass Bottles; Alimenta, **22**, 91-93, 1981.
110. Eckett, A.: The US Accepts the Aseptic Challenge; Food Processing, 23-25, 1985.
111. Edwards, J.L. et al.: Thermal Inactivation Charateristics of *Bacillus subtilis* Spores at Ultra High Temperatures; Applied Microbiology; **13**, 851-857, 1965.
112. van Ejk, T.: Ultrahocherhitzte und aseptisch abgepackte Milchmischgetränke-Aromabildung und Aromatisierung; Deutsche Molkerei-Zeitung, **113**, 995-1001, 1992.
113. Farahnik, S.: A New Method of Hydrogen Peroxide Application "Nebulization"; Dairy and Food Sanitation, **2**, 136-138, 1982.
114. Fink, R. and H.G. Kessler: Erfassung sensorischer Veränderungen in H-Milch während der Lagerung; Deutsche Molkerei-Zeitung, **105**, 1444-1454, 1984.
115. Fink, R. and H.G. Kessler: Reaction Kinetics Evaluation of the Oxidative Changes in Stored UHT Milk; Milchwissenschaft, **41**, 90-94, 1986.
116. Fink, R. et al.: Die Lagerstabilität von aseptisch homogenisiertem Rahm in Abhängigkeit von unterschiedlichen Erhitzungsbedingungen; Deutsche Molkerei-Zeitung, **108**, 1376-1377, 1987.
117. Flückiger: personal communication.
118. Ford, J.E. et al.: Effects of UHT Processing and of Subsequent Storage on the Vitamin Content of Milk, Proc. Nutr. Soc. **27**, 60A-61A, 1968.
119. Francisco, A. et al.: *Klebsiella pneumonia* in Orange Juice Concentrate; Appl. and Environmental Microbiology, **49**, 1527-1529, 1985.
120. Franklin, J.G. et al.: Methods of Assessing the Sporicidal Efficiency of an Ultra-High Temperature Milk Sterilising Plant. II. Experiments with Suspensions of Spores in Milk; J. Appl. Bact., **21**, 47-50, 1958.
121. Franklin, J.G.: Spores in Milk: Problems Associated with UHT Processing; J. Appl. Bact., **33**, 180 -191, 1970.
122. Fussnegger, B.: Vitamine and Provitamine: Ihre Stabilität in Milch und Milch-produkten; Deutshe Molkerei-Zeitung, **109**, 476-482, 1988.
123. Geller, A.: Mikrobiologie und Papierfabrikation; Verpackungs-Rundschau, technisch-wissenschaftliche Beilage, **34**, 1-6, 1983.
124. Geyer, S. and H.G. Kessler: Herstellung flockungsstabiler Kaffeesahne; Deutsche Molkerei-Zeitung, **109**, 1060-1066, 1988.
125. Glaser, H. and E. Hangst: Einfluss der Kühllagerung auf den Geschmack der H-Milch; Deutsche Molkerei-Zeitung, **108**, 4-6, 1987.
126. Görner, F. et la.: Sulphur Compounds and the Flavour of UHT Milk; 20th Int. Dairy Congress, 713-714, 1978.
127. Görtges, E.: Rohwareneinfluss auf Klärung und Stabilisierung beim Apfelsaft; Flüssiges Obst, 16-19, 1980.

128. Granzer, R.: Veränderungen von Fruchtsäften in Verbraucherpackungen während langer Lagerzeiten; Verpackungs-Rundschau, Technisch-wissenschaftliche Beilage, 35-40, 1982.
129. Grasshoff, A.: Toträume in CIP-gereinigten Rohrleitungssystemen; Deutsche Milchwirtschaft, **34**, 407-412, 1983.
130. Graumilch, T.R. et al.: Aseptically Packed Orange Juice and Concentrate: A Review of the Influence of Processing and Packaging Conditions on Quality; J. Agri. Food Chem., **34**, 402-405, 1986.
131. Gravini, R.B.: The pH of Foods; Dairy and Food Sanitation, **9**, 112-113, 1986.
132. Gravini, R.B.: Training: Creative Ways to Make it Work for You; Dairy and Food Sanitation, **6**, 204-207, 1983.
133. Green, A.: Applying Quality Assurance Principles to Food Control; Food Control, **4**, 2-6, 1993.
134. Hahn, G: Reinfektionsfreies Abfüllen von Milchprodukten in Bechern mit H_2O_2; Deutsche Molkerei-Zeitung, **102**, 518-519, 1981.
135. Hamid-Samimi, M.H. et al.: Aseptic Packaging of Ultrapasteurised Egg. North Carolina State Univeristy, P.O. Box 7624, Raleigh, NC, 27695-7624, (USA).
136. Hartmann, G.: Mikrobiologische Probleme beim Einsatz von Packmitteln aus Kunststoff-Folien; Neu-Verpackung, 18-23, 1981.
137. Hartmann, A.: Schnellnachweis der Sterilität von UHT-Milch mit Hilfe der Impedanzmessung; Diplomarbeit, Fachhochschule für Technik Sigmaringen.
138. Hathaway, S.C.: Risk Assessment Procedures Used by the Codex Alimentarius Commission and its Subsidiary and Advisory Bodies; Food Control, **4**, 189-201, 1993.
139. Hauser, G.: Hygienische Gesichtspunkte bei der Konstruktion von lebensmittel-verarbeitenden Anlagen; Deutsche Milchwirtschaft, **36**, 1733-1738, 1985.
140. Heeschen, W. et al.: Hygienische Qualität von Milch und Milch-produkten-Sicherheit statt Risiko; Deutsche Molkerei-Zeitung, **109**, 766-771, 1988.
141. Henningsen, D.: Herstellung einer fettverminderten UHT-Schlagsahne; Deutsche Molkerei-Zeitung, **94**, 2011-2013, 1973.
142. Hersom, A.C.: Quality Control of Aseptically Packed Foodstuffs; Behr´s Seminare "New Trends in Aseptic Processing and Packaging of Foodstuffs", October 1987, Munich.
143. Hinrichs, J. et al.: Einfluss der Herstellungstechnologie auf die Schaumstabilität von UHT-Schlagsahne; Deutsche Milchwirtschaft, **43**, 1304-1311, 1992.
144. Holzinger, T.W.: Quality Assurance: National and International; J. Food Protection, **44**, 553-555, 1981.
145. Huber, J.: Entkeimen von Packstoffen beim aseptischen Abpacken; Verpackungs-Rundschau, **30**, 33-37, 1979.
146. International Dairy Federation: Control Methods for Sterilised Milk, International Standard (Draft), **113**, 1982, General Secretariat, 1040 Brussels, Square Vergote 41.
147. International Dairy Federation: Control Methods for Sterilised Milk, International Standard **48**, 1969, General Secretariat, 1040 Brussels, Square Vergote 41.
148. Ito, K.A. et al.: Resistance of Bacterial Spores to Hydrogen Peroxide; Food Technology, **28**, 58-66, Nov. 1973.
149. Ito, K.A. and K.E. Stevenson: Sterilisation of Packaging Materials Using Aseptic Systems; Food Technol., **38**, 60-62, 1984.
150. Jones, R.E.: Recombination of Milk and Milk Products - Technology and Engineering Aspects; Raw Material Specifications; Sucrose. International Dairy Federation, Report of group B, 3-10, 20-23 June 1978.

151. Jordan, W.K.: Sterilisation and Aseptic Packaging of Milk Products: Changes in Products, J. Dairy Science, **51**, 1144-1146, 1968.
152. Jowit, R.: Hygienic Design and Operation of Food Plants; Society of Chemical Industry, London, published by Ellis Horwood Ltd., 1980.
153. Juven, B.J. and I. Shomer: Spoilage of Soft Drinks Caused by Bacterial Flocculation; J. Food Protection, **48**, 52-53, 1985.
154. Kammerlehrer, J.: Schlagrahm (pasteurisiert, ultrahocherhitzt)-Verbesserung der Festigkeit, Vermeidung des Absetzens; Deutsche Mokerei-Zeitung, **95**, 1820-1825, 1974.
155. Kiermeyer, F: Ultrahocherhitzung und ihr Einfluss bei Milch and Milch-produkten, Deutsche Milchwirtschaft, **23**, 841-844, 1972.
156. Kieffer, R.G.: Validation, Risk-Benefit Analysis; PDA J. Pharmaceutical Science and Technology, **49**, 249-252, 1995.
157. Klann, D.: Qualitätssicherung-Produkthaftung-Schadenverhütung; Deutsche Milchwirtschaft, **41**, 1358-1362, 1990.
158. Kleeberger, A.: Bakteriologische Untersuchungsmethoden zur Qualitäts-beurteilung; Deutsche Milchwirtschaft, **29**, 1380-1385, 1978.
159. Kleeberger, A. and M. Busse: Die bakteriologische Kontrolle von H-Milch; Deutsche Milchwirtschaft, **30**, 1337-1342, 1979.
160. Klupsch, H.J.: Haltbarmachung von Sauermilchprodukten durch Pasteurisieren; Deutsche Molkerei-Zeitung, **92**, 1759-1762, 1971.
161. Konietzko, M. and H. Reuter: Schnellmethode zum Nachweis von Veränderungen in Milch während der UHT-Behandlung; Deutsche Molkerei-Zeitung, **103**, 308-312, 1982.
162. Lang, F. and A. Lang: More New Ideas for Long-Life Dairy Products; The Milk Industry, **68**, 18-21, 1968.
163. Langeveld, L.P.M. et al.: Estimation of the Lowering of the Oxygen Tension in a Flow-Through Cell as a Practical Means of Detecting Non-Sterility in Aseptically Packed UHT-Sterilised Products; Neth. Milk Dairy J., **32**, 69-79, 1978.
164. Langeveld, L.P.M. and A.C. Bolle: Duration of the Pre-Incubation Period in the Sterility Control of UHT-Sterilised Milk; Neth. Milk Dairy J., **33**, 172-180, 1979.
165. Langesrud, T.: Changes in the Protein Fractions and Flavour of UHT-Sterilised Goat's Milk During Extended Storage; 18th International Dairy Congress, A.A.1, 188, 1970.
166. Leaper, S.: Comparison of the Resistance to Hydrogen Peroxide of Wet and Dry Spores of *Bacillus subtilis* SA22; J. Food Technol., **19,** 695-702, 1984.
167. Leaper, S.: Sterilisation of Packaging Materials Using Oxidising Agents; Behr's Seminar: New Trends in Aseptic Processing and Packaging, Munich, October 1987.
168. Lechner, E.: Über den Gehalt der H-Milch an Sauerstoff und dadurch bedingte Veränderungen während der Lagerung; Deutsche Milchwirtschaft, **28**, 123-126, 1977.
169. Leighton, F.R.: Recombination of Milk and Milk Products-Technology and Engineering Aspects; Raw Material Specifications; Water International Dairy Federation, Report of Group B, 3-10, 20-23 June 1978.
170. Lembke, A. and E. Wartenberg: Verbesserung von UHT-Milch durch Einsatz einer Baktofuge; Milchwissenschaft, **37**, 737-741, 1982.
171. Linke, D.: Das aseptische Abfüllen von Molkereiprodukten in tiefgezogene Kunststoffpackungen, Milchwissenschaft, **26**, 543-546, 1971.
172. Lippert, K.D. and T. Claussen: Qualitätssicherung in der Lebensmittelindustrie; Deutsche Milchwirtschaft, **42**, 248-249, 1991.
173. Lisiecki, R. et al.: Aseptic Package Adresses a Variety of Needs; Food Technol., **44**, 126-127 (June) 1990.

174. van der Loo, L.G.W.: Aseptic Packaging of In-Flow Preserved Fruit Juices; Nordeuropaeisk Mejeri-Tidskrift, **4**, 66-85, 1977.
175. Lovio, A.: Tutteli- the Infant Formula of the Future; Scandinavian J. of Dairy Technology and Know-How, 114-115, 1984.
176. Lubieniecki-von Schelhorn, M.: Keimfreimachung: Stand der Sterilisierung von Packstoffen; Die Ernährungswirtschaft, **10**, 810-814, 1970.
177. Lück, H. et al.: Bacteriological Evaluation of UHT Milk; S. Afr. J. Dairy Technol., **10**, 83-85, 1978.
178. Mankowski, A.: Innovation im Bereich der Milchprodukte durch die aseptische Kartonpackung; Deutsche Molkerei-Zeitung, **105**, 582-586, 1987.
179. Mankowski, A.: Innovation in der Lebensmittelindustrie durch die aseptische Kartonpackung; Verpackungs-Rundschau, **38**, 356-360, 1987.
180. Mann, E.J.: Lactic Beverages (Part 2); **50**, Dairy Industries International, 15-16, March 1985.
181. Marshall, M. et al.: Factors Impacting on the Quality of Stored Citrus Fruit Beverages; The Shelf Life of Foods and Beverages, Proceedings of the 4th International Flavor Conference, Rhodes, Greece, 237-254, 23-26 July, 1985.
182. Mayr, W.: Homogenisierung von Konsummilch; Deutsche Milchwirtschaft, **39**, 12-29, 1988.
183. McKellar, R.C.: Colorimetric Assay for Lipases; Dairy Industries International, **51**, 19, 1986.
184. McNutt, K.: Consumer Attitudes and the Quality Control Function; Food Technology, **42**, 97-99 (December) 1988.
185. Meier, P.: Total Quality Management (TQM); Deutsche Milchwirtschaft, **43**, 1678-1682, 1992.
186. Meier, P.: -ISO 9000/EN 9.000-Zertifikat und Total Quality Management, ein Vergleich oder warum sich die Intermilch AG für Total Quality Management entschieden hat; Deutsche Milchwirtschaft, **44**, 285-286, 1993.
187. Meursing, E.H.: Cocoa Powder: Types, Uses and Quality Control; Candy and Snack Industry, (June) 1980.
188. Moisio, T. and M. Kreula: A Checking Method for Fluid Milk in Closed Packages; Milchwissenschaft, **28**, 477-478, 1973.
189. Mostert, M.A.: European Hygienic Equipment Design Group (EHEDG), personal communication, 1991.
190. Mottar, J. et al.: Sensoric Changes in UHT Milk during Uncooled Storage, Milchwissenschaft, **34**, 257-262, 1979.
191. Mottar, J.: Heat Resistant Enzymes in UHT Milk and their Influence on Sensoric Changes during Uncooled Storage; Milchwissenschaft, **36**, 87-91, 1981.
192. Mottar, J.: La Fabrication de Desserts Lactes a Laide du Procede UHT Indirect et Leurs Properties; Revue de L'agriculture; **37**, 1167-1178, 1984.
193. Mrowetz, G. and J. Thomasow: Zum Nachweis und Verhalten von Wasserstoffperoxid in Milch; Deutsche Molkerei-Zeitung, **92**, 639-641, 1971.
194. Murdock, D.I. et al.: Thermal Resistance of Lactic Acid Bacteria and Yeast in Orange Juice and Concentrate; Food Research, **18**, 85-89, 1953.
195. Nermark, C.: Tetra Pak´s New System for the Hydrolysis of Ultra-Sterilised Aseptically Filled Milk; North European Dairy J. **5**, 1- 8, (No 1), 1978.
196. Nichols, L.E.: Recombination of Milk and Milk Products-Technology and Engineering Aspects; Packaging and Storage of Raw Materials; International Dairy Federation, Report of Group B, June 1978.
197. Objerg, L.: Tetra Pak, personal communication
198. Ostermann, A.E.: Aseptic Packaging of Liquid Food with Particulates; Newsletter of the International Dairy Federation No. **18**, April 1990.
199. Parodi P.W.: Recombination of Milk and Milk Products-Technology and Engineering Aspects; Raw Material Specifications; Vegetable Oils. International Dairy Federation, Report of Group B, 3-10, 20-23 June 1978.

200. Pflug, I.J. and E. Odlaug: A Review of z and F Values Used to Ensure the Safety of Low-Acid Canned Food; Food Technology, **32**, 63-70, (June) 1978.
201. Pflug, I.J.: Factors Important in Determining the Heat Process Value, FT, for Low-Acid Canned Foods; J. Food Protection, **50**, 528-533, 1987.
202. Peter, A.: Fruchtsaftinfektion durch Schimmelpilze und ihre Bekämpfung; Lebensmittel Industrie, **12**, 256-259, 1965.
203. Pichhardt, K.: Krisenmanagement in der Ernährungswirtschaft, Deutsche Milchwirtschaft, **44**, 1993.
204. Pien, J.: Chemical and Physico-Chemical Aspects: Laboratory Control; IDF Seminar on UHT Milk, Malmö (Sweden), (May), 1971.
205. Porter, J.W.G. and S.Y. Thompson: Nutritional Aspects, IDF Seminar on UHT Milk, Malmö, Sweden, 1971.
206. Powers, J.J.: Effect of Acidification of Canned Tomatoes on Quality and Shelf Life; Critical Review in Food Science and Nutrition, 371-396, June 1976.
207. Puhan, Z.: Heat Treatment of Cultured Dairy Products; J. Food Protection, **42**, 890-894, 1979.
208. Put, H.M. et al.: Heat Resistance Studies on Yeast spp. Causing Spoilage in Soft Drinks; J. Appl. Bact, **40**, 135-152, 1976.
209. Püschel: Vortrag, International Presskonferenz der Milchversorgung Bochum, 19. März, 1968).
210. Rand, A.G. and P.M. Linklater: The Use of Enzymes for the Reduction of Lactose Levels in Milk Products; Australian J. Dairy Technol., 63-67, June 1973.
211. Redelinghuys, H.J.P. and P.A. Torline: The Preparation of Litchi Juice; (Proceedings of the Fifth Biennial Congress, Milner Pak, Johannesburg), Supplement to Food Review, 118-119, 1979.
212. Reinders, M.A.: The Continuous Manufacture of Ready to Use Dairy Desserts; The Milk Industry, June 1969.
213. Renner, E.: Milchprodukte in der Ernährung des Menschen, Deutsche Milchwirtschaft, **22**, 84-94, 1971.
214. Renner, E.: Milch und Milchprodukte in der Ernährung des Menschen, Verlag Th. Mann OHG, Hildesheim, 1974.
215. Renner, E. and R. Schmidt: Untersuchungen über die sensorische, chemische und ernährungsphysiologische Qualität vershiedener Konsummilchsorten in unterschiedlichen Verpackungen, Deutsche Milchwirtschaft, **26**, 1620-1629, 1975.
216. Renner, E.: Ernährungsphysiologische Veränderungen der Milch durch Be- und Verarbeitung, Deutsche Milchwirtschaft, **27**, 1021-1031, 1976.
217. Renner, E. and H. Dorguth: Untersuchungen zur Eiweissqualität; Deutsche Milchwirtschaft, **31**, 505-508, 1980.
218. Renner, E.: Gehalt an freien Fettsäuren in der Rohmilch in Abhängigkeit von verschiedenen Produktionsfaktoren und in der H-Milch; Deutsche Milchwirtschaft, **33**, 233-238, 1982.
219. Renner, E.: Milch und Saft; Deutsche Milchwissenschaft, **42**, 1629-1638, 1987.
220. Rippen, A.L.: Aseptic Packaging of Grade A Dairy Products; J. Dairy Science, **53**, 111-115, 1970.
221. Robson, J.N. and G. Barnes: Hygienic Design and Operation of Food Plants; Society of Chemical Industry, "Plant Design to Minimise Non-Microbial Contamination," London, published by Ellis Horwood Ltd., pp. 121-136, 1980.
222. Rose, D.: Good Manufacturing Practice Guidelines for the Processing and Aseptic Packaging of Low-Acid Foods; The Campden Food Preservation Research Association, Chipping Campden, Gloucestershire, GL 55 6LD, England, Technical Commissioning, **11**, 1987**.**
223. Saint-Hilaire, P. and P. Struebi: Tropical Fruit Processing; Alimenta, **21**, 59-65, 1982.

224. Samuelsson, G.: Några erfarenheter av att sterilisera mjölk i Alfa Laval VTIS-anläggning; Svenska Mejeritidningen, **55**, 243-251, 1963.
225. Samuelsson, E.G. et al.: Sediment in Sterile Milk Manufactured by the Steam Injection System; XVI International Dairy Congress, Section 11:2, 3-11
226. Sand, F.E.M.J.: Zur Bakterien-Flora von Erfrischungsgetränken; Brauwelt, **111**, 252-264, 1971.
227. Sanderson, W.B.: Recombination of Milk and Milk Products-Technology and Engineering Aspects; Raw Material Specifications; Dairy Products. International Dairy Federation, Report of Group B, 3-10, 20-23 June 1978.
228. Schaal, E. and F. Nöcker: Untersuchungen über die mikrobiologische Beschaffenheit der im Verkehr befindlichen H-Milch-Erzeugnisse; Archiv für Lebensmittelhygiene, **28**, 56-61, März-April 1977.
229. Schmid H. and K. Guthy: Über Einflüsse auf den Gefrierpunkt der Milch; Deutsche Molkerei-Zeitung, **104**, 1162-1168, 1983.
230. Schoch, W.: Aseptic Packaging; Tappi Journal, **67**, 56-60, September 1984.
231. Schulz, M.: Kombination von Grundlagenforschung und Entwicklung zur Herstellung haltbarer saurer Milchproduktzubereitungen; Milchwissenschaft, **24**, 28-34, 1969.
232. Scott, V.N. and D.T. Bernard: Heat Resistance of Talaromyces flavus and Neosartorya fischeri Isolated from Commercial Fruit Juices; J. Food Protection, **50**, 18-20, 1987.
233. Senften, H.: Vorratshaltung von Wasser: Leitungswasser oder Mineralwasser in Flaschen?; Alimenta, **12**, 207-213, 1973.
234. Shaw, F.B.: Toxicological Considerations in the Selection of Flexible Packaging Materials for Foodstuffs; J. Food Protection, **40**, 65-68, 1977.
235. Sheehan, R.L.: Quality Assurance in the Packaging Industry; J. Pack. Technol., 17-19, (September/October) 1990.
236. Siegenthaler, E.: Sauermilchprodukte mit verlängerter Haltbarkeit; Deutsche Milchwirtschaft, **23**, 409-414, 1972.
237. Sizer, C.E. et al.: Maintaining Flavour and Nutrient Quality of Aseptic Orange Juice; Food Technology, **42**, 152-159, June 1988.
238. Speck, M.L.: Compendium of Methods for the Microbiological Examination of Foods, Chapter 45: Fruit Drinks, Juices, and Concentrates; American Public Health Association, 563-567, 1976.
239. Speck M.L. and D.M. Adams: Impact of Heat Stabile Microbial Enzymes in Food Processing; J. Dairy Science, **59**, 786-789, 1976.
240. Spotti, E. et al.: Capacita delle mueffe di alterare i succhi di frutta con produzione di gas; Industria Conserve, **59**, 8-11, 1984.
241. Stannard, C.J. et al.: Combined Treatment with Hydrogen Peroxide and Ultra-Violet Irradiation to Reduce Microbial Contamination Levels in Pre-Formed Food Packaging Cartons; J. Food Protection, **46**, 1060-1064, 1983.
242. Stevenson, K.E.: Implementing HACCP in the Food Industry; Food Technol., **44**, 179-180, (May) 1990.
243. Stigloher, M.: Erstmals keimfreie Abfüllung von uperisierter Milch in der Schweiz; Deutsche Molkerei-Zeitung, **82**, F.40, 1961.
244. Stranger-Johannessen, M. and T. Röneid: Nachweis von gasdurchlaessigen, Fehlerstellen in Packungen aus Aluminium-Verbundsmaterialen, Verpackungs-Rundschau, **35**, 43-45, 1984.
245. Suhren, G. and W. Heeschen: Zur Impedanz als Messgrösse für die bakteriologische Untersuchung von Milch und Milchprodukten; Deutsche Molkerei-Zeitung, **106**, 1710-1717, 1985.
246. Surak, J.: The ISO 9000 Standards: Establishing a Foundation for Quality; Food Technology, **46**, 74-80, 1992.

247. Swartling, P. and B. Lindgren: The Sterilising Effect against *Bacillus subtilis* Spores of Hydrogen Peroxide at Different Temperatures and Concentrations; J. Dairy Res., **35**, 423-428, 1968.
248. Tamplin, T.G.: Hygienic Design and Operation of Food Plants; Society of Chemical Industry, "CIP, Technology, Detergents, and Sanitisers", London, published by Ellis Horwood Ltd., pp. 183-226, 1980.
249. Tetra Laval: Dairy Handbook
250. Thomé, K.E., et al.: Manufacture of Sterile Milk, Development Work with the Alfa-Laval VTIS; Milk and Dairy Research, Alnarp, Sweden, Report No. **70**, 1964.
251. Toledo, R.T. et al.: Sporicidal Properties of Hydrogen Peroxide Against Food Spoilage Organisms; Appl. Microbiology, **26**, 592-597, 1973.
252. Toledo, R.T.: Chemical Sterilants for Aseptic Packaging; Food Technol., **29**, 164-169, May, 1975.
253. Toledo, R.T.: Postprocessing Changes in Aseptically Packed Beverages: A Review of the Influence of Processing and Packaging Conditions on Quality; J. Agri. Food Chem., **34**, 405-409, 1986.
254. Toledo, T. and S.Y. Chang: Advantages of Aseptic Processing of Fruits and Vegetables; Food Technology, **44**, 72-76, 1990.
255. Toyoda M. et al.: Simple and Rapid Determination of Hydrogen Peroxide in Milk by Use of an Oxygen Electrode; New Zealand J. Dairy Science and Technol., **17**, 41-46, 1982.
256. Toyoda, M. et al.: Rapid Procedure for the Determination of Minute Quantities of Residual Hydrogen Peroxide in Foods by Using a Sensitive Oxygen Electrode; J. Agri. Food Chem. **30**, 346-349, 1982.
257. United Nations: Modern Sterilisation Methods for Milk Processing, United Nations Industrial Development Organisation, Vienna; Food Industry Studies, No 4.
258. US FDA: 21 CFR, 173.310
259. US FDA: 21 CFR, 176.
260. US FDA: 21 CFR 178.1005.
261. Vögele, P.: Mikrobiologische Kontrolle der H-Milch, Welt der Milch, **34**, 3-14, 1980
262. Vögele, P.: Mikrobiologische Kontrolle der H-Milch; Welt der Milch, **34**, 582-590, 1980.
263. Voss, E. and B. Moltzen: Untersuchungen über die Oberflächenkeimzahl extrudierter Kunststoffe für die Lebensmittelverpackung; Milchwissenschaft, **28**, 479-486, 1973.
264. Waes, G. et al.: A Rapid Method for the Detection of Non-Sterile UHT Milk by the Determination of the Bacterial ATP; Milchwissenschaft, **39**, 707-711, 1984.
265. Wasserfall, F.: Die statistische Qualitätskontrolle als Mittel zur Prüfung der bakteriologische Wirksamkeit von UHT-Anlagen; Milchwissenschaft, **28**, 558-766, 1973.
266. Weiss, G.: Erhitzungsnachweis bei Konsummilch: Bedeutung and Analytik; Deutsche Molkerei-Zeitung, **110**, 168-174, 1989.
267. West, F.B. et al.: Inactivation of Heat Resistant Proteases in Normal Ultra-High Temperature Sterilised Skim Milk by a Low Temperature Treatment; J.Dairy Sci. **61**, 1078-1084, 1978.
268. Wiese, W.: Erfahrungen im Rahmen der Qualitätskontrolle bei Milch und Milchprodukten, Deutsche Milchwirtschaft, **36**, 1516-1524, 1985.**102.**
269. Wild, R.: Qualitätssicherung in der Lebensmittel-Wirtschaft; Deutsche Milchwirtschaft, **39**, 1492-1500, 1988.

270. Worthington Biochemical Corporation: Peroxidase (Horseradish); Worthington Enzyme Manual **1.11.1.7**, 42-45.
271. Zadow. J.G.: A Study of Some Variables in the Manufacture of UHT Whipping Cream; 18th International Dairy Congress, A.4.1, 194, 1970.
272. Zadow, J.G.: Recombination of Milk and Milk Products - Technology and Engineering Aspects; Recombined Milks and Creams; International Dairy Federation, Report of Group B, 3-10, 20-23 June 1978.
273. Zadow, J.G.: UHT Milk-Standards and Quality Assurance; Australien J. Dairy Technol., **35**, 140-144, 1980.
274. Zadow, J.G. et al.: The Stability of Goat's Milk to UHT Processing; Australian J. Dairy Technol., **38**, 20-22, March 1983.
275. Zollinger, W. and W. Schimdt-Lorenz: Mikroorganismen-Dichtigkeit der Heissiegelnähte von sterilisierfähigen Behältern aus Aluminium-Kunststoff-Verbundmaterialien; Verpackungs-Rundschau, **36**, 69-79, 1985.

„Wohlbesorgt ist dieses nun
Julchen kann was andres tun".
(Wilhelm Busch, "Die Knopp-Trilogie, Julchen")

"Here the book is at its proper end
To something else we can now attend."