Chemistry and Technology of Soft Drinks and Fruit Juices

Second edition

Edited by Philip R. Ashurst

Blackwell Publishing

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Preface

It is now some six years since the first edition of this book was published. The aim of the volume was to provide an overview of the chemistry and technology of soft drinks and fruit juices for graduates in food science, chemistry or microbiology entering production, quality control, new product development or marketing in the beverage industry, or in companies supplying ingredients or packaging materials to the beverage industry.

Soft drinks and fruit juices are produced in almost every country in the world and their availability is remarkable. From the largest cities to some of the remotest villages, soft drinks are available in a variety of flavours and packaging. The market for these products also continues to show a remarkable potential for growth. For example, the UK market alone showed an increase in consumption of some 7% in 2003 (over 2002) and, in the ten years since 1993, this same market grew from a consumption of around 9 billion litres to around 14 billion litres in a full year.

The variety of products and packaging types continues to expand, and among the more significant developments in recent years has been the increase in diet drinks of very high quality, many of which are based on spring or natural mineral water. The skill and imagination of the product developer and the marketing teams have contributed to an ever-increasing range of flavours and ingredients that relate to products designed as much for lifestyle as for age groups. No less a contribution continues to come from the development of new packaging materials and formats alongside the old favourites.

In this extensively revised second edition, a review of the major markets and their development has been included. This chapter provides a fascinating insight into the shape, size and development of beverage markets in many different countries. The book continues with a new chapter on fruit and juice processing and with a review of beverage ingredients generally. There is a new chapter dealing specifically with the wide range of carbohydrate and intense sweeteners that are, after water, among the most important components of almost all soft drinks.

Further new chapters have been added on non-carbonated products and, in particular, on the special techniques and considerations relevant to the production of carbonated beverages, which constitute the major part of the worldwide soft drinks industry. Recent industrial incidents have raised the profile of the quality of carbon dioxide which, although present in a small mass percentage, is key to the customer impact of carbonated drinks.

A chapter has again been included on soft drink production. The packaging chapter has been revised and some of the more important new packaging options are discussed in detail.

The analysis of soft drinks and fruit juices once again merits a full chapter. This chapter has been updated to cover the increasing range of analytical technology and instrumentation that is available for use in the industry. Concerns over ingredient or product adulteration for commercial gain, extortion or bioterrorism are, sadly, part of the world in which manufacturers now have to operate, and this emphasis on cutting-edge analytical capability is not likely to disappear.

The most likely cause of product deterioration for the manufacturer of soft drinks and particularly fruit juices remains that arising from micro-organisms. Because of the relatively low pH values of these products, the likelihood of any pathogenic organism growth remains small (although not unknown), especially in freshly pressed juices such as apple. The manufacturers' nightmare is a major yeast infection which can cause packed product to explode and, in the worst case scenario, to injure a consumer. Thus the chapter on the microbiology of soft drinks and fruit juices remains of key importance in furthering the understanding of micro-organisms and their control.

Another new chapter deals with herbal ingredients and the roles that they play in functional beverages - a newly classified group of beverages that go beyond normal nutrition to give consumers something extra. Products in this category have actually been available for many years in the form of isotonic and energy beverages, but the concept has now been widened to include many products offering a range of consumer benefits that do not extend into the medicinal arena.

The final chapter addresses a range of miscellaneous issues that are relevant to the products under consideration.

The reader will find some overlap from chapter to chapter, but this has been allowed to remain because of the relevance of the topic within the individual chapter.

As with all books, this book has its limitations and shortcomings which are attributable to the editor alone. We do not, for example, attempt to cover legislative issues in general, although specific references are made where appropriate. Readers concerned with ensuring compliance with their local statutes should make separate enquiries with the proper authority. Water, effluent and other environmental matters are similarly not covered in any detail, as there are volumes dedicated to these topics.

The editor is greatly indebted to the contributing authors, as without them the work would be nothing. All are experienced practitioners, expert in their subject fields, and for most of them the work involved in writing or updating a chapter produces a significant extra burden.

All in all, it is to be hoped that the reader will find this new edition to be a valuable extension of the original edition and, most significantly, that it will meet the needs of those who seek to learn more about this worldwide industry.

The editor is very grateful to Mrs Susan Bate for her help with correspondence and the typing and checking of manuscripts.

Philip Ashurst

1 Introduction

P.R. Ashurst

1.1 Overview

Fruit juices and soft drinks are available in essentially the same form almost anywhere in the world. From polar bases to the tropics, and from the largest developed nations to small and less developed countries, soft drinks and fruit juices are available in bottles, cans, laminated paper packs, pouches, cups and almost every other form of packaging known.

This chapter outlines the various types of products available and sets the scene for the following contributions, which deal with the more specialised aspects of the chemistry and technology of these products.

1.2 Soft drinks

What are soft drinks? There is no single definition available but it is generally accepted that they are sweetened water-based beverages, usually with a balancing acidity. They are flavoured, frequently coloured and often contain an amount of fruit juice, fruit pulp or other natural ingredients. The predominant ingredient is water – often ignored and frequently maligned – and it should be remembered that the primary function of soft drinks is hydration. The sweetness and other characteristics are in some respects secondary and yet they do have importance in the provision of energy and some of the minor essential nutrients needed to meet daily requirements.

It is generally accepted that the description of soft drinks excludes tea, coffee, milk beverages and, until recently, alcohol. However, in many countries the production of 'soft' drinks containing alcohol is growing. Many see this as an undesirable trend because traditionally the taste of alcoholic beverages has been associated with adulthood. The blurring of the edges between the markets and tastes for alcoholic drinks and soft drinks appears to facilitate an easy transition for children and young people to the consumption of alcohol.

There are two basic types of soft drinks: the so-called ready-to-drink (RTD) products that dominate the world market and the concentrated or dilute-to-taste products that are still important in some markets. These include syrups and so-called squashes and cordials.

Whether RTD or dilutable, soft drinks characteristically contain water, a sweetener (usually a carbohydrate, although artificial sweeteners are increasingly important), an acid (citric or malic are the most common), flavouring, colouring and preservatives. There is a large range of additional ingredients that can be used for various effects.

1.2.1 Ready-to-drink products

This sector accounts for the largest volume of soft drinks production and is divided into products that are carbonated, that is, contain carbon dioxide, and those that are still. Carbonated RTD soft drinks dominate the world market and detailed consumption trends appear in Chapter 2 of this volume.

The market for carbonated soft drinks is dominated by two giant brands of cola drinks that, together with their associated brand names, account for just over half the world's consumption of such products.

Non-carbonated RTD beverages have shown some considerable growth in recent years mainly because of the availability of aseptic packaging forms. Still drinks that rely on chemical preservation or hot-pack/in-pack pasteurisation suffer from a number of potential problems, including rapid flavour and colour deterioration.

1.2.2 Concentrated soft drinks

Concentrated soft drinks became very important during, and in the early years following, the Second World War. Many were based on concentrated orange juice, which was widely available as a nutritional supplement, and were packed in flat-walled medicine bottles.

The main markets for concentrated soft drinks developed in the United Kingdom and its former empire. The products became universally known as 'squashes' or 'cordials' and became enshrined as such in UK legislation in the 1960s.

Another very important development was the production of citrus comminutes. These were produced by mixing together, in appropriate proportions, the juice, peel components and essential oils of citrus fruits and comminuting the mixture in a stone mill. The resulting product delivered a more intense flavour and cloud than could be obtained from juice alone and allowed the creation of 'whole fruit drinks', which have dominated the concentrates market in the United Kingdom over the past 40–50 years.

1.2.3 Legislation

It is not the intention of this chapter to cover legislation affecting soft drinks in any detail – not least because it varies from country to country and there is a constant variation of legislation within countries.

Legislation is important from a historical perspective. For example, in the United Kingdom the Soft Drinks Regulations 1964 (as amended) codified the products according to the way in which the industry was then organised and set into law definitions not only of 'soft drinks' but also of many of the product types, such as crushes, squashes and cordials, that subsequently became generic household names in the United Kingdom and many parts of the English-speaking world.

These regulations were probably among the most proscriptive compositional statutes that existed for any food products in the United Kingdom, and for beverages anywhere in the world. As well as defining soft drinks, they laid down the requirement for minimum levels of sugars in certain product types, the maximum levels of saccharin (the only artificial sweetener then permitted) and the minimum levels of comminuted fruit and fruit juices that defined the best-known product categories. These regulations were eventually revoked in 1995.

The current trend is to move away from compositional legislation to a much freer approach in which carbohydrates and other nutritional components can be used at will and additives are taken from 'positive' lists of functional components. Other ingredients are frequently controlled by negative usage (i.e. they must not be present or must not exceed closely defined limits).

This move to remove controls on formulations is backed by informed labelling that contains almost every imaginable kind of information for the consumer. This approach is now used throughout the world with only relatively minor variations from country to country.

At the time of writing, in a European country a soft drink label must contain the following information:

- descriptive name of the product ('appropriate designation');
- list of ingredients;
- declaration of contents;
- name and address of packer or importer in the European Union;
- nutritional data (obligatory in certain circumstances).

The declaration of the quantity of key ingredients (fruit or fruit juice in soft drinks) has become law through quantitative ingredient declaration regulations in Europe, and where artificial sweeteners and carbohydrates are used together, an appropriate statement is necessary. A warning about the product being a source of phenylalanine must be incorporated when aspartame is used as a sweetener.

Because legislation is in most countries a rapidly changing sphere, it is essential for those formulating, producing and marketing soft drinks to update themselves regularly in relation to the legislation of consumer countries.

1.2.4 Product types

1.2.4.1 Ready-to-drink products

Historically, soft drinks were refreshing beverages that copied or extended fruit juices. Fruit juices typically have around 10–12% naturally occurring sugars with a pleasant balancing acidity that varies from about 1% down to 0.1%. It is therefore not surprising that soft drinks were typically formulated around 10–11% sugar content with about 0.3–0.5% of added acid (usually citric). The simplest form of beverage contained such a mix of these basic nutritional components in water with flavouring, colouring and chemical preservatives added as appropriate. With the addition of carbon dioxide to render the product 'sparkling', 'effervescent' or 'fizzy', the manufacturer had a lemonade or similar product. With the addition of fruit juice to a level of 5–10%, a pleasing effect of both taste and appearance could be achieved. Such products were typically described as 'fruit juice drinks', 'fruit drinks' or 'crushes' (a reserved description in the old UK regulations). Various other additions can be made, including vitamins and minerals, clouding agents and foaming agents and plant extracts.

RTD beverages are mostly carbonated (i.e. contain carbon dioxide). This, as well as giving sensory characteristics, provides a very effective antimicrobial effect, especially against yeasts and moulds. Carbon dioxide is effective against yeasts because it tends to suppress the production of more CO_2 as a byproduct of the fermentation of sucrose to ethanol. It deprives moulds of the oxygen that most require for growth. Good hygiene standards are the norm in most soft drinks bottling operations today, and it is possible to produce carbonated drinks without chemical preservatives by flash-pasteurising the syrup before it is mixed with carbonated water. The risk of microbiological spoilage is then low, but where multiserving containers are used the risk is increased because of the potential for subsequent contamination.

Carbon dioxide levels vary widely and are usually expressed as 'volumes of CO_2 gas' (i.e. the volume of carbon dioxide contained in solution in one volume of product). Lightly carbonated products will contain around 2.0–3.0 volumes of the gas; moderate carbonation usually refers to about 3.5–4.0 volumes and high carbonation levels are around 4.5–5.0 volumes. Large bottles that are likely to become part full will be relatively highly carbonated, and mixer drinks contain among the highest carbonation levels because the resultant mixture (e.g. gin and tonic) needs to have a satisfactory residual level of dissolved carbon dioxide.

RTD beverages are also produced in non-carbonated forms. The most popular current form of these is distributed in aseptic card/foil laminate packs, such as Tetra Pak or Combibloc. These drinks are typically unpreserved and come in volumes of 200–330 ml.

An alternative form of non-carbonated beverage comes in form–fill–seal plastic containers, which are typically square or round section cups with foil or plastic laminate lidding. Such products are difficult to produce to a quality that will satisfactorily compete with the shelf life of aseptic foil/laminate packs. Form–fill–seal containers leave their contents vulnerable to oxidative degradation and are especially at risk of mould spoilage. The packs can be produced in aseptic conditions but the products are typically chemically preserved.

Some manufacturers produce RTD products at drinking strength but this is wasteful of plant and requires large-volume production tanks. The usual approach is to manufacture a syrup or concentrated form of the beverage that is then diluted with carbonated water. The syrup, which can be flash-pasteurised, can be dosed into bottles that are then topped up with carbonated water. This is known as the 'post-mix' method. Where the alternative, 'pre-mix', method is employed, syrup and water are mixed in the correct proportions in special equipment prior to bottle filling.

1.2.4.2 Dilutables

As indicated in Section 1.2.2, some markets are substantial consumers of concentrated soft drinks. These products are purchased in concentrate form by the consumer, who then adds water (which can be carbonated if required) to achieve the desired taste.

In the United Kingdom and many parts of the English-speaking world these products are referred to generically as 'squashes' or 'cordials'. Chapter 6 of this volume covers this topic in detail.

Most concentrated beverages contain fruit juice or 'whole fruit', a term that refers to a comminuted form of citrus that includes components of juice, essential oil, peel (flavedo) and pith (albedo). Concentrated soft drinks are usually flash-pasteurised and chemically preserved. Their dilutable form means that they are often held in partially filled bottles for significant lengths of time (often many weeks or even months) and are extremely vulnerable to spoilage by micro-organisms.

Some manufacturers do produce unpreserved concentrates, but such products are invariably pasteurised in the bottle and carry a warning that the contents should be refrigerated after opening and consumed within a short time-span (typically 2 weeks).

Concentrates are normally produced at their packed strength, flash-pasteurised and transferred immediately to their final packaging.

1.2.5 Development trends

Probably the most significant trend in soft drinks manufacture in recent years has been towards the use of non-calorific artificial sweeteners. The best known of these, saccharin, was used in soft drinks during and after the Second World War, when sugar was in short supply. Saccharin in its soluble form is about 450 times sweeter than sugar and can be a significant cost-reducer, but its sweetness is marred by a bitter taste to which many consumers are sensitive.

In more recent years other artificial sweeteners have been developed and it is now possible to produce soft drinks with almost all the characteristics of the sugar taste. Such products are almost free of any energy (calorific) content and lack much of the cariogenic property for which many have criticised sugar-containing beverages.

Almost all soft drinks are now available in ‘low calorie’, ‘diet’ or ‘light’ formulations. These products have a low calorie content and are usually cheaper to manufacture than the corresponding sugar-containing products.

Another, and perhaps more obvious, development area is the constant search for new flavours and unusual ingredients. There is currently a great interest in the use of various botanical extracts such as guarana and ginseng because of their implied qualities, but it is noteworthy that one of the oldest and certainly the most successful flavours, cola, was originally formulated with, and still contains, a natural vegetable extract of cola nut.

The third major area for development is that of soft drinks containing ingredients that enable some special nutritional or physiological claim to be made for the product. This will usually be an energy claim because soft drinks are an ideal vehicle for delivering carbohydrates, some in specially formulated mixtures, in a readily and rapidly assimilable form. Of the other nutrients that can be included, fruit juice, vitamins and minerals are the most common, but some products contain significant levels of protein or even fibre (as non-metabolisable carbohydrate).

1.2.6 Nutrition

The nutritional value of soft drinks is sometimes exaggerated by manufacturers who want consumers to perceive their products to be of special benefit. That said, the value of soft drinks must not be understated, because they are an essential vehicle for hydration. Soft drinks are usually absorbed more readily than water (because of their osmolality), can replace lost salts and energy quickly and are rapidly thirst quenching. Their balance of sweetness and acidity, coupled with pleasant flavours, makes them attractive to all ages of consumers. Products are specially formulated to meet the tastes, nutritional needs and physiological constraints of the whole population, from babies to geriatrics.

The claims that are legally permitted for soft drinks vary from country to country but for the most part are limited to nutritional claims concerning energy, proteins, vitamins and/or minerals. Any form of medicinal claim (i.e. curative or symptomatic relief) will almost always be excluded by corresponding medicines legislation. There is, nevertheless, a growing trend to include natural extracts in many soft drinks (e.g. ginseng or ginkgo) and then rely on the general understanding and folklore that surrounds such ingredients to impart the special values that have been attributed to them.

There are three main areas of particular nutritional significance for soft drinks. The first is energy. Some soft drinks are formulated to deliver a rapidly assimilated energy boost to the consumer. All carbohydrates are important sources of energy but soft drinks generally contain soluble sugars, which are easy to administer. However, because high levels of sugars are often intensely sweet and even sickly, with a cloying sensation in the mouth, energy drinks are formulated around glucose syrup. For a given solid carbohydrate content, this raw material is much less sweet than sucrose. Selection of the method of hydrolysis used for the corn starch allows glucose syrup to be tailored, to some extent, to include mixed carbohydrates, that is, mono-, di-, tri-and oligosaccharides. Such blends are the basis of some very effective products used by athletes and those recovering from illness.

The second area of nutritional significance is that of the so-called isotonic drinks, which are of equivalent osmolality to body fluids. They promote extremely rapid uptake of body salts and water, and are very important products for sportspeople and others requiring almost instant hydration.

Third, soft drinks have been widely formulated to low-calorie forms and these are now available for those who wish to enjoy such beverages and yet minimise their calorific intake.

Other nutritional benefits that are claimed by some producers include the delivery of essential vitamins and minerals, especially to children.

On the negative side, soft drinks have acquired a reputation for being an agent in the development of dental caries. This has been claimed to arise when sugar residues remain in the mouth or when (especially) young children have an acidic drink almost constantly in their mouths. It is perhaps now accepted that the dental caries problem is related more to the misuse, or even abuse, of soft drinks than to the effects of normal consumption of such products.

1.2.7 New product trends

New product development is a constant activity for most soft drinks producers. For the most part there are few really new products; alternative flavours and different forms of packaging are widespread and no doubt will continue to be so.

The development of specialised energy drinks or isotonic beverages is perhaps an example of a truly new product area. As new raw materials become available, for example soluble whey protein, whole ranges of products are likely to be spawned.

A recent trend has been to incorporate alcohol into soft drinks. Whether such products are properly still to be known as ‘soft’ drinks is a separate issue, and the outcry in many countries about their use as a means of introducing children to alcohol has been widespread.

As indicated above, another area of interest is the re-introduction of botanical extracts into soft drinks. It is sometimes overlooked that one of the earliest widely available soft drinks was based on an extract of cola nut.

Packaging developments are likely to offer some exciting new opportunities in the future and soft drinks are likely to remain at the forefront of product innovation in many countries.

1.3 Fruit juices

What is a fruit juice? Various definitions have been suggested, but the one used in the UK Fruit Juice and Fruit Nectars Regulations of 2003 may be helpful. These regulations implement EU Directive 2001/112/EC. Four alternatives are accepted as defining fruit juice (at least within the United Kingdom).

(1) 'Has been prepared from fruit material described in Schedule 2 either with or without any other material listed in that Schedule';
(2) 'Where it contains any additional ingredient, that ingredient is authorised for the food in question by Schedule 3';
(3) 'Being a food bearing the reserved description "fruit juice", "concentrated fruit juice", "fruit juice from concentrate" or "dehydrated or powdered fruit juice", it has not been prepared by adding to it:
   (i) both sugars and lemon juice (whether concentrated or not) or
   (ii) both sugars and acidifying agents as permitted by Directive 95/2/EC';
(4) 'Where it has been prepared by using any treatment or additional substance, that treatment or substance is specified in Schedule 4'.

An additional description refers to fruit nectars. The Schedule referred to above give more precise definitions of fruit juice.

Working from the above definitions there are thus two basic juice product types, and it is these that dominate today's markets.

The majority of fruit juice is made by reconstituting concentrated juice with water to a composition similar to that of the original state. However, since records are not usually kept of the exact quality of the original juice, such reconstitution normally relates to an agreed trade standard. Reconstituted juices are often packed in aseptic long-life containers such as TetraPaks.

There is in many countries a growing market for fresh 'single-strength' juice made by squeezing fruit, subjecting it to some processing, packaging it and selling it within a cold chain distribution system. Such juice is usually referred to as 'not from concentrate' and will have a shelf life that varies from 1 or 2 weeks to 2 or 3 months.

1.3.1 Processing technology

It is not intended to give a detailed description here of juice processing. The subject is covered in Chapter 3 of this volume and in other volumes such as *Fruit*

Processing (Arthey & Ashurst, 2001) or *Production and Packaging of Non-Carbonated Fruit Juices and Fruit Beverages* (Ashurst, 1995), which may help the reader who wishes to obtain more detail.

In general terms, fruits are collected, sorted and washed, and then subjected to a type of mechanical compression appropriate to the fruit concerned. Although there are general fruit presses that can be used for more than one fruit type, fruits such as citrus, pineapple and stone fruits are usually processed in specially designed equipment.

Some fruit types (e.g. pome fruits such as apples and pears) require mechanical treatment (milling) coupled with a biochemical process (involving enzymes) to break down the cellular structure and obtain best yields. It is possible to achieve almost total liquefaction by means of an appropriate enzyme cocktail.

Additionally, a diffusion or extraction process can be used to obtain best yields from certain fruits.

If juice is to be sold as 'not from concentrate' it is usually screened and pasteurised immediately after pressing – an operation with two main objectives. The first is to control the growth of spoilage micro-organisms that live on the fruit surface (mainly yeasts and moulds). The second is to destroy the pectolytic enzymes that occur naturally in fruit that would otherwise break down the cloudy nature of the juice. If, however, a clear juice is required (e.g. apple or raspberry), enzymes can be added to accelerate this natural process.

Juice for concentration is normally subjected to screening to remove cellular debris and then fed to a one- or multi-stage evaporation process to remove most of the water and other volatile material. Evaporators today are highly efficient processing units: up to nine stages are used, sometimes with thermal recompression to obtain maximum efficiency. Increasingly, evaporators also recover the volatile aromatic substances that are partly responsible for giving fruit juices their sensory characteristics. The re-addition of such volatiles is widely practised at the point when concentrates are reconstituted into single-strength juices. The issue of whether this should be obligatory has been clarified. European Council Directive 2001/112/EC (the Fruit Juices Directive) makes the addition of such volatiles at reconstitution obligatory. The UK 2003 Regulations (which are based on this Directive) state that reconstituted fruit juice is the product 'obtained by replacing, in concentrated fruit juice, water extracted from that juice by concentration, and by restoring the flavours'.

After concentration, juices are normally held in storage until they are reconstituted. Some concentrated juices, particularly orange, require freezing at below −10°C for effective preservation. Others, particularly apple, can be held at around 10–15°C without risk of deterioration. The degree of concentration plays an important part in determining storage conditions: in the above examples, orange juice is normally concentrated to about 65°Brix and apple to 70°Brix.

An alternative method of storage is to hold juices under aseptic conditions in drums or other containers. No particular temperature constraints then apply for microbial stability but there is a substantially increased risk of colour browning and taste deterioration if juices are held aseptically at temperatures above about 10°C.

Some juices are held in sulphited conditions (e.g. 1500–2000 ppm sulphur dioxide) but this is suitable only for juices destined for uses other than reconstitution as fruit juice.

1.3.2 Adulteration

The adulteration of fruit juices is widespread. As with any commodity, juice manufacturers, blenders and users can secure considerable financial benefit from adulterating fruit juice. It should be emphasised that food safety issues are not normally an issue in fruit juice adulteration. The issue is simply the fact that traders and consumers are being defrauded: an adulterated fruit juice sold as pure fruit juice is not as it has been labelled.

Although adulteration is becoming increasingly sophisticated, it is normally seen as falling into one of three types:

(1) overdilution of juices with water;
(2) use of cheaper solid ingredients (particularly sugars);
(3) blending of cheaper with more expensive juices.

The issue of too much water being added to juices has largely been addressed through the application of a minimum solids content (measured in degrees Brix). Many countries now have in place a minimum Brix value for various juices. These minima are backed either by legal statute or industry code of practice. They normally apply to juices prepared by adding water to concentrate and not to 'not from concentrate' products.

The second category of adulteration is by far the most common. For example, apple juice will normally contain around 11% by weight of solids. At least 90% of these solids are carbohydrates – sucrose, dextrose and fructose predominating. Considerably cheaper sources of carbohydrates can be found, and the simple addition of a mixture of carbohydrates in roughly the same proportion as those found naturally in apple juice can be used to 'stretch' apple juice by a considerable proportion. In more sophisticated forms of adulteration the added components can be made to carry a similar 'signature' to the juice.

In the third category a cheaper juice can be used to adulterate a more expensive one; for example, elderberry juice can be used to extend strawberry or raspberry juice.

The detection of adulteration and its quantification have spawned some elegant scientific techniques, some borrowed from other fields and some developed specifically for use in fruit juice work.

Detection of overdilution and the presence of sugars of other origin is now carried out largely by measuring key isotope ratios (such as carbon 13 : 12 ratios, deuterium : hydrogen ratios and oxygen 18 : 16 ratios) and comparing them with both those found naturally in fruit and agreed international standards. An important part of the fight against adulteration has been the development of databases examining fruit of different origin and season.

Another elegant method of detecting sugar addition in particular has been the use of high performance liquid chromatography (HPLC) to determine the presence of oligosaccharides that are characteristic of the added sugars but not the fruit. The use of enzymic methods for determining the presence of specific components (e.g. D-malic acid, which does not occur naturally) is also helpful.

The analytical detection and measurement of fruit juice adulterants is a rapidly developing field and the interested reader is directed to works dealing specifically with the subject, such as *Food Authentication* (Ashurst & Dennis, 1996) and *The Analytical Methods of Food Authentication* (Ashurst & Dennis, 1997).

Finally, the addition of cheaper juices to more expensive ones can usually be detected and measured using techniques appropriate to the likely components. For example, the addition of elderberry to strawberry juice can readily be detected by examining the anthocyanins present using HPLC and comparing them with standards.

1.3.3 Other processes

A number of other processes have become commonplace in the manufacture of fruit juices. For example, if oranges of the varieties Navel or Navellina are processed the juice becomes unpleasantly bitter because of the biochemical development of a glycoside, limonin. This substance can be partially or totally removed by the use of appropriate ion-exchange resins to yield a juice of acceptable taste.

There has also been a range of developments leading to the removal of acidity, colour and minerals from clear juices such as apple. The product of such a combination of processes can be a clear, colourless carbohydrate syrup that can be used in a variety of food processes. There seems little doubt that the legal status of such a product is not fruit juice; yet it is often, optimistically, so called.

Enzyme and finishing treatments are widely used in the processing of fruit juices to obtain products of particular specification.

Another contentious issue is the further processing of fruit pulp, and especially citrus pulp. The addition of water to such a pulp can give an extract containing around 5% solids, which can be concentrated to around 65% and used to dilute (more expensive) pure juice. These products are normally described as pulp-wash extracts. Note, however, that 'in-line' pulp-wash extract arises in normal citrus processing and is becoming an acceptable component at levels of addition not exceeding 5% of the juice.

Byproducts of the juice industry are important but are not dealt with here. The interested reader is referred to *Fruit Processing* (Arthey & Ashurst, 2001).

1.3.4 Nutrition

Fruit juice is important in human nutrition far beyond its use as a refreshing source of liquid. Many fruits contain a variety of minor ingredients, particulary vitamins and minerals, as well as carbohydrates, which are the predominant solid component. Although fruit contains small amounts of protein and fat, these are not important ingredients of juices.

Nutrients frequently consumed in sub-optimal concentrations by humans are proteins, calcium, iron, vitamin A, thiamin (vitamin B1), riboflavin (vitamin B2) and ascorbic acid (vitamin C). Some of these nutrients occur in higher concentrations in fruit juices than in other foods. There is experimental evidence that indicates that ascorbic acid of natural origin is apparently superior to that of synthetic origin.

It has been established that the above phenomenon is caused by the presence of certain flavonoid compounds in fruit juice that influence blood circulation, increasing the permeability and elasticity of capillaries. This action is known as vitamin P activity, but the flavonoids showing this property are not classified as vitamins, because there are several substances with this activity and no serious deficiency diseases occur if they are not consumed. There are indications that these flavonoids have a useful protective action, in particular against some respiratory diseases, but they are readily decomposed in the body and it is impossible to maintain an effective concentration in the blood.

Apart from the more obvious benefits of fruit juice, such as being a source of potassium, it contains other substances that have or are claimed to have useful pharmacological activity. For example, limonin and other related limonoid substances present in citrus fruit are believed to have a role in inhibiting certain forms of cancer. Sorbitol, which occurs in many fruit juices, has a laxative effect.

Several components with antioxidant activity are found in fruit juices. These include ascorbic acid, tocopherols (vitamin E), beta-carotene and flavonoids. Beta-carotene has antioxidant activity that can quench the singlet oxygen that can induce precancerous cellular changes.

Whatever the nutritional interest, it should be noted that changes occur during storage, particularly to the minor components of juices and particularly under adverse conditions (e.g. light, increasing temperature, time).

1.4 Packaging

Chapter 9 of this volume deals specifically with packaging, and it is not the intention of this chapter to pre-empt that contribution. However, it is perhaps useful to look briefly at the trends in packaging that are important in the whole area of beverage development.

Traditionally, most beverages were packed in glass. This has many attractive features, not least that it is an excellent protective medium, but its overriding disadvantages are its weight and brittleness. Despite this, high volumes of soft drinks and juices are still packaged in glass, some of it multi-trip packaging.

The development of the board–polymer–aluminium package used to form in-line boxes, which are packed aseptically, has been perhaps the outstanding packaging development for beverages. The pack provides an almost ideal combination of protection, minimal weight and economic size.

Another important packaging development area is plastic. Various plastics have been and continue to be used: high- and low-density polyethylene (HDPE, LDPE), polyvinyl chloride (PVC), polystyrene (PS) and various barrier plastics. These can be formed into bottles of conventional shape or fed into machines producing form–fill–seal packages, typically cups.

By far the most important plastic is polyethylene terephthalate (PET). Bottles of this material are formed in a two-stage process. So-called pre-forms are made by injection moulding and, in a second process, are then stretch–blow-moulded to produce a bottle. PET has properties surprisingly like those of glass, but it does not have the same disadvantages of weight and brittleness.

Perhaps the most exciting developments are yet to feature in fruit juice packaging. PET can be laminated with other plastics, such as nylon and ethylene vinyl alcohol (EVOH), to give extremely good barrier properties, and polyethylene naphthalate (PEN) may enable production of a plastic bottle that can be pasteurised at high temperatures.

1.5 Summary

Soft drinks and fruit juices are widely consumed in ever-increasing quantities and are very important commodities in the trade of most countries. This volume sets out to introduce the reader with a good general science background to the more detailed aspects of these products, and it is hoped by this means to provide a useful reference work that will be widely used by those wishing to learn more about the products.

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2 Trends in beverage markets

G. Roethenbaugh

2.1 Introduction

The need to quantify the size and activity of the world's beverage markets does not arise simply from a fascination with statistics. Beverages are clearly big business. The success or failure of a product launch or an existing brand strategy can have considerable impact on the profitability, and share price, of many a beverage multinational. Embroiled in the new information age, the global beverage industry is awash with news and information that shapes and describes the market's evolution. Interpreting this information effectively is critical. This applies equally to beverage manufacturers and to those organisations and individuals throughout the beverage supply chain that act as retailers, brokers and suppliers to the industry.

The pace of change is relentless and the global beverage industry has witnessed unprecedented levels of market activity. New product development, brand extensions and sustained price depreciation alongside the relentless pace of innovation and industry consolidation have all contributed to intense competition. As the pace of change has not slackened, taking stock of global developments remains tremendously challenging. This chapter describes the context for the global beverage industry. It beings with a general definition of beverage categories, and then considers the latest consumption trends by sector. An analysis of the world's most influential beverage markets is followed by discussion of the fastest growing regions and countries. In keeping with the theme of this book, the chapter examines the soft drinks sector across the globe, particularly those segments that are leading the way for growth. New issues and trends facing beverage industry players are then explored – with a summary of the future growth outlook and those market developments that should transform the beverage industry.

There are four primary sectors of the global commercial beverage market (Figure 2.1): first, hot drinks, such as tea, coffee and hot malt-based products; second, milk drinks, including white drinking milk and flavoured milk products; third, soft drinks, a sector with five main subcategories (bottled water; carbonated soft drinks; dilutables, also known as squash and including powders, cordials and syrups; 100% fruit juice, and nectars with 25–99% juice content; still drinks, including ready-to-drink (RTD) teas, sports drinks and other non-carbonated products with less than 25% fruit juice); fourth, alcoholic drinks, including beer, wine, spirits, cider, sake and flavoured alcoholic beverages – sometimes referred to as pre-mixed spirits.

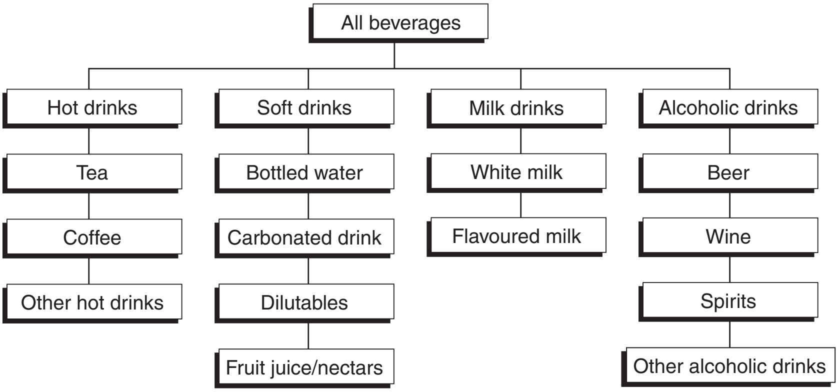

Figure 2.1 Beverage sectors and segments.
Source: Zenith International.

2.2 Beverage consumption trends

Globally, growth in beverage consumption remains solid and steady. In the last 5 years commercial beverage volumes have risen between 2.3 and 2.7% annually. The volume of RTD hot drinks, milk drinks, soft drinks and alcoholic drinks consumed reached an estimated 1.36 trillion (million million) litres in 2003 – a 2.5% increase on 2002 (Figure 2.2). Per capita consumption stood at 217 l in 2003 (Figure 2.3).

Of the main beverage sectors, soft drinks was the fastest growing in 2003, up 4.7% in volume terms and clearly ahead of the global average of 2.5% growth in beverage markets. Boosted by new flavoured milks and offsetting the fairly slow consumption of white milk in recent years, milk drinks generated 1.8% growth in 2003. Alcoholic drinks saw 1.7% growth in 2003, while hot drinks were the slowest growing sector, up 1.2% in volume. Overall, hot drinks represent the largest volume share, accounting for 37% in 2003. Soft drinks, with a 34% share, are next. In third and fourth positions are milk drinks and alcohol, respectively claiming 15 and 14% volume shares.

Breaking down the four main beverage sectors into their composite categories, tea is the world's favourite beverage and claims the largest share of volume consumed. Sometimes referred to as 'share of throat', this particular measure of consumption provides a useful method of comparing the trends in various beverage categories. This is particularly the case if we compare shares of throat over a period of years. In 2003, tea claimed a 25% share of throat (Figure 2.4). However, in 1998 its share was 2% higher, at 27%. White drinking milk has likewise lost favour among beverage consumers, with a 15%

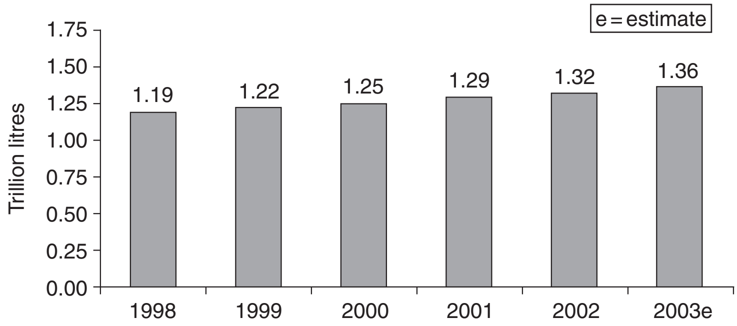

Figure 2.2 Global beverage consumption, 1998–2003.
Source: www.globaldrinks.com (Zenith International).

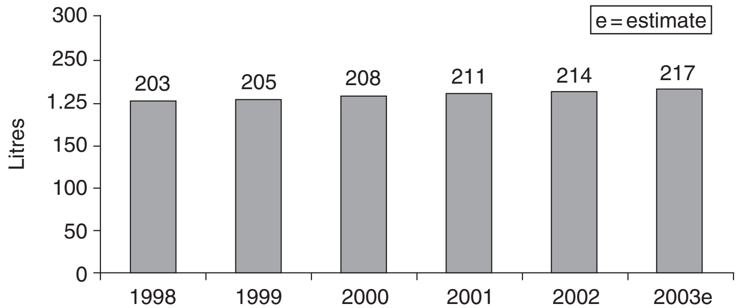

Figure 2.3 Global beverage consumption per person, 1998–2003.
Source: www.globaldrinks.com (Zenith International).

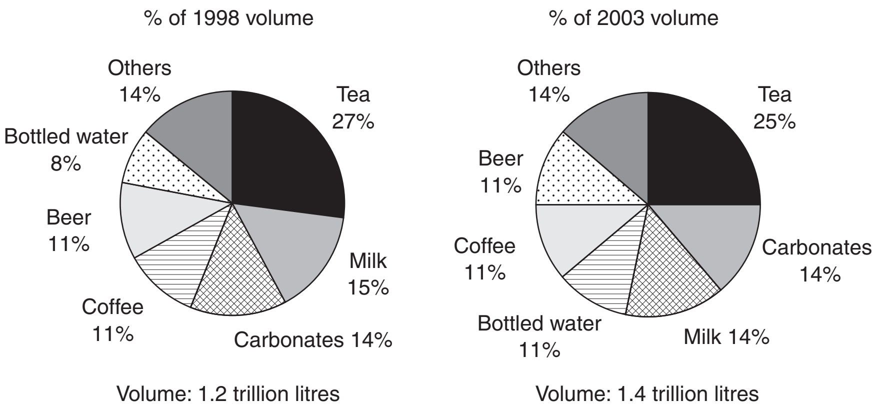

Figure 2.4 Beverage consuption by category, 1998–2003.
Source: www.globaldrinks.com (Zenith International).

worldwide share of throat in 1998 down to 14% in 2003. The main beneficiary of this swing in volume share during 1998–2003 was bottled water. Representing only 8% of global beverage consumption in 1998, bottled water claimed an 11% share of volume consumed in 2003.

The trend towards consumption of bottled water and other soft drinks categories has been a major driver in the global beverage market. As previously mentioned, tea is the world's most popular drink – at 54 l per person per annum. However, by 2003 carbonated soft drinks had overtaken white milk to become the second most popular beverage. Thus the world's most powerful beverage multinationals, such as Coca-Cola, PepsiCo and Cadbury Schweppes, have transformed consumption trends in recent years. With a globally recognised brand, Coca-Cola in particular has embedded itself into the lifestyles of consumers worldwide. Other carbonated beverages have similarly delivered taste and brand values to a multitude of willing beverage consumers.

The consequence of this has been a move towards convenient, RTD products, supported by multi-million dollar marketing budgets and, equally as important, sophisticated distribution systems. White milk, tea and other hot beverages face particular challenges in today's ready-to-imbibe world.

The world's more mature beverage categories face competition for share of throat not only from carbonated drinks but also from an increasingly important bottled water category. Bottled water is now the world's fourth most popular beverage and has grown in stature from being the sixth most popular beverage category in 1998. In this period bottled water has consistently been the world's fastest growing category, recording a 58% gain – compared with the slower performance of milk, tea and coffee, which respectively grew by 5, 6 and 7% in the 1998–2003 period. Bottled water's popularity can be explained by the simple concept of 'convenient hydration'. Like other RTD soft drinks, bottled water is convenient and portable. Coupled with this is an awareness among consumers of the 'eight glasses a day' rule – that drinking eight glasses of water each day replenishes body fluids and aids well-being. A number of other factors have prompted heightened consumption in countries with more established bottled water markets and can be used to explain the uplift of bottled water consumption in more nascent markets. First, consumers have become more aware of their environment, health and individuality. This has been reflected in consumer purchasing behaviour. Second, tap water has become less relied upon for water consumption in more developed markets such as the United States, and in developing markets across the globe mistrust of tap water has remained widespread. Any occasional off-taste or discolouration in tap water has reinforced the contrast with the natural attributes of bottled water. Additionally, the change in emphasis by bottled water companies, from projecting the purity of their product to highlighting its health benefits, is increasingly bringing the 'eight glasses a day' rule to consumers' attention.

With less impressive growth than bottled water, coffee is the world's fifth most popular beverage. It is the world's second most important legally traded commodity by value, after oil, and is the primary export of a number of developing countries. Yet, despite a burgeoning coffee bar culture in the West, growth in coffee consumption globally has been particularly slow in recent years, at around 1–2% p.a. in the years 1998–2003. Coffee suppliers have faced dwindling coffee prices and thus margins. At the same time, they have had to adapt to heightened consumer demand for speciality and premium coffees and the increasing sophistication of Western coffee consumers. Although coffee consumption has a defined role in a great many markets and has well-established occasions on which it is consumed, such as at mealtimes, consumers are increasingly limiting the role of coffee in their daily lives. Alongside tea, coffee is one of the world's more mature beverage categories. Slow and steady growth across the globe is the inevitable consequence of market maturity. Meanwhile, the challenge facing tea and coffee manufacturers is the increasing competition presented by RTD and pre-packed tea- and coffee-based soft drink alternatives. Alongside this is an increasing consumer preference for chilled rather than hot beverages that threatens to edge out hot drink products for share of throat.

Turning to alcoholic beverages, beer is the most widely consumed alcoholic drink and is the world's sixth most popular beverage. The global beer market has proved to be a rapidly changing environment. Traditionally strong markets, such as North America and Western Europe, are seeing the consumption of beers and lagers stabilise, and in several countries consumption is actually falling as consumers turn to other types of alcoholic drinks, such as flavoured alcoholic beverages, or towards healthier options, such as bottled water, fruit juice and other soft drinks targeted at adults.

Alcoholic drinks, possibly more than any other food or drink, have woven themselves into the culture of the countries in which they are sold. Although this makes the sector more resilient in times of economic hardship, it also means that, overall, the main alcohol categories are less inclined to demonstrate rapid growth in consumption levels. Other factors, such as strong governmental legislation, higher taxation and a move towards healthier lifestyles and therefore healthier beverages, all help to restrict the sector's potential for rapid growth. In addition, a more limited number of drinking occasions has held back rates of growth (with consumption throughout the day clearly not socially acceptable in most societies).

This is not to say that there is no dynamism within the global alcoholic drinks sector, for it is one that is showing considerable change. It is just that the changes in the consumption of alcoholic drinks come less from rapid volume growth and more from the changing nature of the type of alcoholic drink that is consumed. Whereas the increase in soft drinks consumption often displaces hot drinks or dairy consumption, the alcoholic drinks sector is more likely to see a transfer of consumption from one alcoholic drink segment to another. For example, the popularity of spirits is decreasing in China but this is being compensated for by increased consumption of beer.

In the three remaining soft drinks categories, still drinks with less than 25% juice content, fruit juices/nectars and dilutables claimed positions seven, eight and nine, respectively, in the most popular beverage ranking. Although on a global scale none of these categories generates annual per capita consumption greater than 10 l, these soft drinks categories are significant. In particular, a wide variety of still drinks, either with or without juice, have become increasingly influential. The increasing consumer demand for a healthy soft drinks option has driven the strong growth in this area. Consumers have embraced products such as juice drinks, bottled tea and sports drinks. Although many of these products might be associated with a specific consumption occasion, such as the use of a sports drink to replenish lost body salts after exercise, the role of still drinks is broadening. There has, for example, been a wider positioning of sports drinks as a more mainstream thirst quencher for active people with busy lifestyles. This in turn has helped sustain high volume sales. Fruit juices with 100% juice content and nectars with 25–99% juice have, like other soft drinks, benefited from a consumer shift towards healthier food and drink products. Fruit juices/nectars have grown at a steady 2–5% p.a. since 1998. Although fruit juice is more confined to particular consumption occasions, such as the breakfast table in many Western households, it has increased in stature due to association with bottled water – purity, naturalness and health. Dilutables have generated more slow and steady growth in recent years. Dilutable squashes and other concentrates are the world's ninth most popular beverage at drinking strength. However, for a number of years, the trend has been away from dilutables towards the convenience of RTD products.

As the tenth most popular beverage, global wine consumption remained fairly stable over the period 1998–2003, especially when looking at the market from a per capita perspective. Globally, annual average wine consumption has remained static at 4 l per capita since 1998. France and Italy continue to dominate the market in terms of consumption, though this dominance has been steadily eroded over recent years. This erosion is expected to continue as consumption levels in France and Italy level out and other countries, such as the United States, become more prominent in the global wine market. The development of wine consumption in the world's emerging markets has come at a time when consumers are seeking alternatives to the predominant alcoholic beverage, beer. Wine consumption has also benefited from the greater availability of branded 'new world' wines. Moreover, increased branding in the wine market has afforded consumers greater accessibility to a segment where naivety about the product had often been a barrier to entry.

2.3 Regions and markets

Over the period 1998–2003, the global tides of volume consumption have seen a shift in favour of the more populous markets in Asia/Australasia (Figure 2.5). Claiming a 34% share of global consumption, Asia/Australasia has increased its share by two percentage points since 1998, and the region's global influence is expected to grow further. This is due predominantly to the influence of two highly populous yet still fundamentally emerging beverage markets – China and India. China and India are two of the world's three largest beverage markets, behind the United States, which claims first place with a 15% share of global beverage consumption. Given their 10 and 9% volume shares, respectively, China and India help ensure that the top three countries claimed an overall one-third share of total beverage consumption in 2003 (Figure 2.6).

These three markets are by no means similar in their composition. The United States is the world's largest soft drinks market by a substantial margin. Thus, soft drinks account for the majority of beverages consumed: a substantial 56% of US beverage consumption. India and China, on the other hand, have thus far reached their ranking through a hot beverage – tea – rather than through the dominance of soft drinks. Overall, hot beverages account for 41% of total beverage consumption in China and more than two-thirds (68%) in India. However, despite the cultural dominance of tea, it is important to note that hot beverages have registered volume share declines in the three largest beverage markets since 1998. The United States witnessed a one percentage point decline for hot beverages over the period 1998–2003. Despite being well established in India, hot beverages also registered a single percentage point decline. Most significantly, tea in China saw a substantial 10% share decline, from 51% of total beverage consumption in 1998 to 41% in 2003. In the same period soft drinks registered a 9% increase in volume share in China.

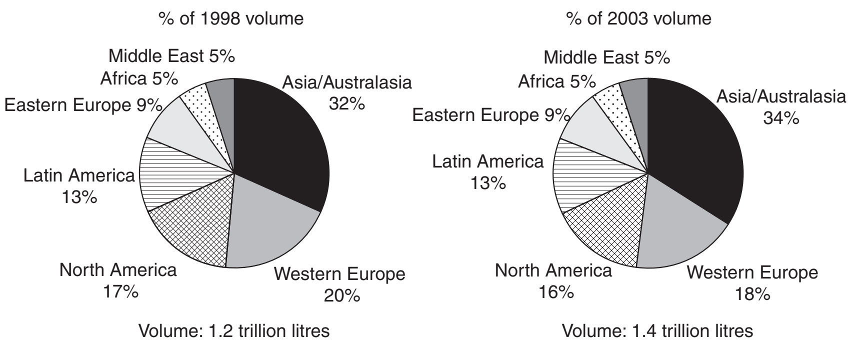

Figure 2.5 Beverage consumption by region, 1998–2003.
Source: www.globaldrinks.com (Zenith International).

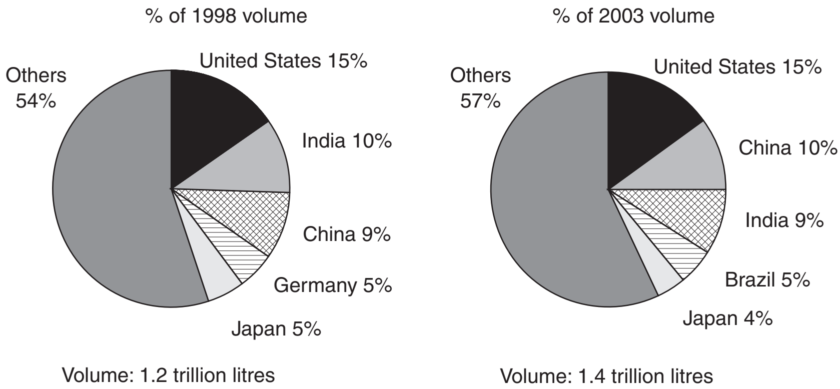

Figure 2.6 Beverage consumption by country, 1998–2003.
Source: www.globaldrinks.com (Zenith International).

This substantial swing away from hot beverages to soft drinks has come about through significant investment by the world's soft drinks multinationals, particularly around the time of China's accession to the World Trade Organisation (WTO). Increasing affluence among Chinese consumers has also brought about a shift in consumption habits, with RTD products gaining favour. Of particular significance has been the meteoric rise of RTD bottled tea. A surge of such products in recent years has helped soft drinks gain share of throat from hot beverages. In addition, a burgeoning bottled water industry has helped carry the tide in soft drinks' favour.

Claiming global pole position for soft drinks consumption in 2003, with a total of 112 billion litres of soft drinks consumed, the United States dominates (Figure 2.7). The United States is some way ahead of the second placed soft drinks market, Mexico, with consumption of 40 billion litres. As previously mentioned, China, in third place in 2003, has witnessed soft drinks grow rapidly in importance in recent years. China is now on track to overtake Mexico and become the second largest soft drinks market. Fourth place in 2003 was claimed by Brazil. The global soft drinks top five was completed by the more mature and established market of Germany, which was overtaken by Brazil in 2003.

Much like in the rest of Europe, soft drinks consumption in Germany was fundamentally boosted by the summer heat wave in 2003. Germany is one of the more mature markets in Europe, and one that is undergoing significant structural change; and the hot summer weather could not have come at a better time for the soft drinks industry. However, in 2003 Germany's soft drinks market was beset by structural upheaval. The main issue was the introduction of a mandatory deposit system on non-reusable packaging. Through the imposition of deposit charges on non-refillable drinks packaging, specifically non-refillable

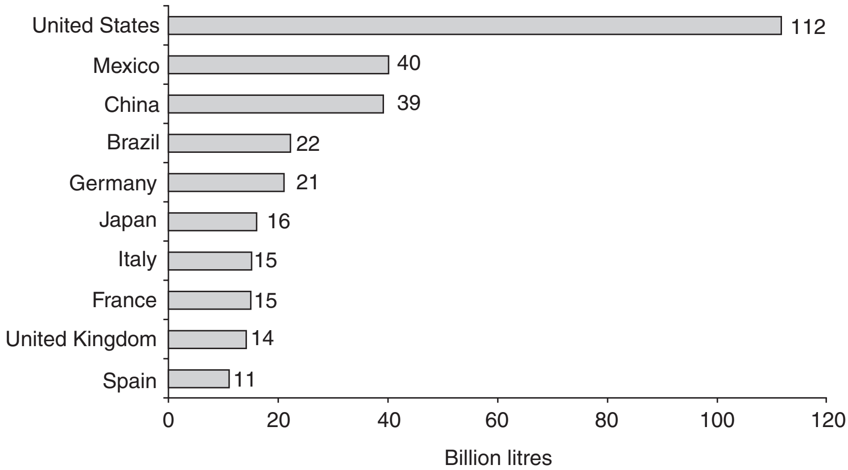

Figure 2.7 Top 10 soft drinks markets, 2003.
Source: www.globaldrinks.com (Zenith International).

polyethylene terephthalate (PET) containers, certain soft drinks categories were thrown into disarray, causing heartache for many in the beverage industry. PET packaging has in recent years edged out glass pack formats due to its convenience for on-the-move consumption and its lower weight, and thus transportation cost. Since the German introduction of the mandatory deposit on one-way packaging, the proportion of drinks sold in reusable packaging has increased substantially. Yet, the irony facing drinks players in 2003 was that volume in single-use packaging was a major driver of market growth in 2002. Those manufacturers investing in reusable PET thus benefited in 2003, while manufacturers of non-reusable PET saw the gains they made in 2002 effectively reversed.

The situation in Germany illustrates that market regulation can have profound effects on the fortunes of drinks manufacturers. Trends witnessed in more mature markets, such as Germany, can have wider repercussions on the global stage – through the formation of new regulatory frameworks, appeals against those frameworks or the readjustment of business practices and international trade procedures. An important additional development in the United States, the world's most developed beverage market, is likewise producing a fundamental impact on international beverage trade. Emphasising a new framework for international beverage distribution, the US government has implemented new regulations under the country's homeland security laws, with the goal of encouraging the development and deployment of anti-terrorism technologies and procedures. The consequence is that food and drink operators wishing to import and distribute in the United States must apply formally to, and are thus regulated by, the US Department of Homeland Security (DHS).

It will take some time before the effect on trade is clearly measurable. What is certain is that the United States is of fundamental importance to the global beverage market as a major importer and exporter of finished beverage products and raw materials. The United States is often referred to as a key benchmark for beverage consumption trends. The steady march of globalisation is often linked with the 'Americanisation' of consumption habits. Thus, beverage trends are often compared through the positioning of a given market against the often more sophisticated and advanced US beverage market. Despite the need to analyse beverage markets in isolation, it is apparent that certain shifts in US beverage consumption have mirrored those found elsewhere around the world. Mirroring total beverage consumption worldwide, US consumption has advanced by around 2% p.a. in recent years. Tea consumption witnessed small volume declines in the 3 years to 2003. Coffee and other hot beverages have made limited annual advances. White milk consumption has been largely static. Flavoured milk products have been more popular. Dilutables, such as powders, have lost ground due to the prevalence of RTD formats. Other types of soft drinks have flourished. Bottled water has been the fastest growing soft drinks category in the United States. In 2003, the gains made by bottled water were substantial, as Coca-Cola took on distribution of Danone's bottled water brands, such as Evian, and forged ahead with its own brands in the United States.

In the United States, beer, wine and spirits have seen fairly modest growth. However, in the other alcoholic drinks category, flavoured alcoholic beverages (FABs), significant gains have been made, albeit from a small volume base. In the United States this category generated 376% volume growth in the 1998–2003 period, thanks predominantly to a glut of malt-based FABs. Globally, the success of FABs, or premixed spirits, has been the driving force behind the other alcoholic drinks segment. The rise of flavoured alcoholic beverages has tended to be concentrated in areas where beer has been the traditional alcoholic beverage of choice, such as in the United States, Western Europe and Australia. The question remains, though, whether this illustrates a permanent consumer shift away from traditional alcoholic offerings or whether the FAB phenomenon is simply a fad that is temporarily appealing to younger drinkers' desire for something different. The long-term success of FABs is also being threatened by criticism that they are contributing to underage drinking. Critics argue not only that their sweeter taste makes them more palatable to younger drinkers, but also that the marketing employed is designed to appeal directly to the more youthful consumer.

Despite the challenge presented by soft drinks and 'healthier' beverage products, alongside the inherent maturity of the alcoholic drinks market, alcoholic drinks consumption has undergone considerable change over the last few years. While some countries are witnessing declines in often traditionally strong alcoholic beverage categories, others are demonstrating real growth potential. Many alcoholic drinks companies in stagnating markets are therefore faced with the choice of either innovating to stimulate demand or expanding their operations into emerging markets where the opportunity for strong growth still exists. Indeed, many of the major brewers have been laying the foundations for expansion into emerging markets, with the potential volume gains an attractive proposition. However, though the potential may be strong, poor distribution systems and underdeveloped market structures in many developing countries have meant that instant volume growth has been hard to achieve. Therefore, though the opportunity for significant growth does exist in developing countries, the real gains may be made only by those willing to invest in markets over the long term.

Worldwide, the major international brewers have tended to move away from their traditional markets and towards developing markets such as China and Russia, countries that have many of the ingredients for strong consumption growth, including positive economic outlooks, low per capita beer consumption and younger demographics. The major international brewers are increasingly underpinning growth within the beer sector as they consolidate their market positions through mergers and seek to enter new and emerging markets through acquisitions.

The world's emerging beverage markets are not just the domain of the world's multinational breweries. Large dairies, hot drinks manufacturers and soft drinks players are seeking to capitalise on the growth opportunities offered by regions such as Asia, Eastern Europe, the Middle East, Latin America and Africa. In the 1998–2003 period, as global beverage consumption advanced overall by 14%, consumption in the Asia/Australasia region grew by 19%, in Eastern Europe by 17% and in the Middle East by 15%. Despite the economic hardship experienced by many Latin American countries, the region kept pace with the global average in 1998–2003. Africa performed just below the global average – perhaps a reflection of the limited degree of investment in the region to date. Emphasising their maturity, and confirming the importance of the world's emerging beverage markets, consumption in both North America and Western Europe grew more slowly than the global average, by 10 and 5% overall, respectively, in the 1998–2003 period.

2.4 Soft drinks dynamics

In soft drinks, it is again interesting to witness the heightened growth of Eastern European, Asian, Middle Eastern, Latin American and African markets. The markets of North America and Western Europe, reaffirming their relative maturity, do not feature at all among the world's top 20 fastest growing countries in terms of soft drinks consumption. The world's major soft drinks players have, of course, been laying the foundations for growth in many emerging markets and the volume gains to be made across the developing world are an attractive proposition for global players and local operators alike. In terms of soft drinks consumption per person, the United States has, reflecting its maturity, held the global lead in recent years. Not far behind are Mexican consumers of soft drinks. By the close of 2003, more rapid growth in Mexican soft drinks consumption placed annual consumption in Mexico and the United States neck-and-neck at around 386 l per head.

For all soft drinks combined, the top 10 fastest growing countries in the 1998–2003 period were exclusively from the emerging regions of Asia, Eastern Europe and the Middle East (Figure 2.8). The fastest growing overall in the period was Indonesia, up 210% – a figure boosted considerably by rapid growth in bottled water consumption in small and larger pack formats. Again with considerable bottled water growth, Pakistan claimed second place – up 125%. In third and fourth place in the soft drinks growth ranking, Turkey and India both achieved volume growth in excess of 100%. China completed the top five, up 92% between 1998 and 2003.

Latin America is regarded as a more advanced emerging soft drinks market, with Mexico and Argentina on a par with North America and Western Europe in terms of per capita consumption. However, many countries in the region fall well below this level of consumption and exhibit substantial potential for future growth, especially once the economic situation within the region improves. Eastern Europe is in a very similar position to Latin America, in that a few countries with close links to developed Western countries have higher per capita consumption, whereas the region's other countries have a lower penetration of soft drink products. As the economies in Eastern Europe develop and consumer purchasing power within the region increases, consumption patterns are expected

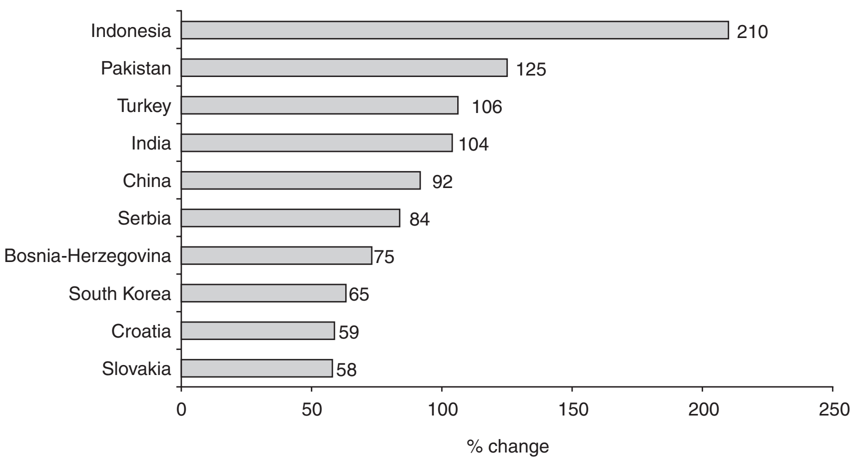

Figure 2.8 Fastest growing soft drinks markets, 1998–2003.
Source: www.globaldrinks.com (Zenith International).

to come more into line with their Western European counterparts. Of all the emerging markets, Asia is the most interesting region in terms of untapped potential for soft drinks growth. The key to this potential is twofold. The first factor is the region's large population coupled with a low per capita consumption, meaning that even a small rise in per capita consumption will result in substantial volume gains. The second is the rising economic status of many of these countries, most notably of the two 'sleeping giants', India and China. The potential of the Chinese soft drinks market in particular has been noted by the Coca-Cola Company, which has stated its intention to make it the company's number one market within the next 10 years.

Emphasising its tremendous importance to the global range of soft drinks products, bottled water claimed a majority 58% share of the volume growth in the 1998–2003 period (Figure 2.9). Carbonated drinks' share of growth was 21%, still drinks claimed a 13% share, followed by fruit juice/nectars on 6% and dilutables on 2%. Although it has yet to reach an equivalent level of actual volume consumed to that of carbonated drinks, bottled water now presents a real challenge to carbonated soft drinks. The increasing consumer demand for a healthy soft drink option and the greater awareness of the need to stay hydrated has driven the strong growth in the bottled water category. The concept of high bottled water consumption, which was previously frowned upon in many countries, is becoming increasingly accepted by consumers, and has been boosted by improved availability through both small pack sizes and water for coolers.

Affirming its importance as the world's most advanced soft drinks market, the United States is by some margin the world's largest bottled water market, with a 16% volume share of global bottled water consumption in 2003,

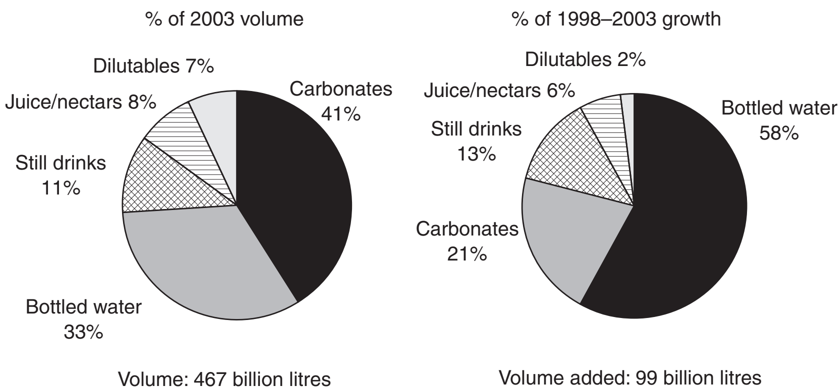

Figure 2.9 Soft drinks sector shares, 1998–2003.
Source: www.globaldrinks.com (Zenith International).

compared with a 9% share for second placed Mexico. However, if we rank bottled water volume growth in the 1998–2003 period across the globe the United States is some way down the ranking table, in 48th place. As with soft drinks overall, emerging markets, growing from a smaller and less well established base, claim leadership in the growth stakes. Pakistan was by some considerable margin the fastest growing bottled water consuming country in the 1998–2003 period, up a staggering 1,805%. The growth was so pronounced because Pakistan has risen from a small volume base, and is thanks to a flooding of bottled water products, notably Nestlé Pure Life, through all distribution channels from roadside stalls to supermarkets. Second placed in the bottled water growth ranking was South Africa, up 428% in the period. India followed in third place on 375%.

In carbonated soft drinks it is not surprising that the United States takes the largest volume share globally. With 30% of total carbonated drinks volume, the United States dominates, with Mexico again claiming second position – albeit some way behind, with a 9% global volume share. As with bottled water, the United States, underlining its maturity, grew at a considerably slower pace than the world's emerging markets in the 1998–2003 period. The United States features in 57th place in the global carbonated drinks volume ranking in this period. Two Eastern European countries were ranked first and second as the fastest growing consuming countries in 1998–2003. Bosnia-Herzegovina was up 160%, and Serbia grew by 88%. In third place, China closely followed Serbia, with 86%. Despite being dented by the SARS (severe acute respiratory syndrome) outbreak in 2003, and its impact on tourism and consumer confidence, Chinese carbonated drinks have over the years been boosted by considerable direct investment from US multinationals such as Coca-Cola and PepsiCo. In dilutables at RTD volume, Mexico led in 2003, with a 17% share of total dilutables volume. Mexico's lead in the dilutables category stems largely from *aguas frescas*, a popular local beverage that utilises fruit essence and that is in turn mixed with bottled water. The United States claimed second place in dilutables in 2003, with a 14% global volume share, largely through a range of powdered soft drinks and cordials. Emphasising that it is a much less dynamic soft drinks segment, the growth of dilutables has been less pronounced in recent years. The three fastest growing dilutables consuming countries in the 1998–2003 period were Serbia, 133%, Lithuania, 100% and Slovakia, 66%. Each of these three Eastern European markets has grown dilutables from a small base, through a range of popular local products. Across the globe, however, dilutables continue to face stiff competition from low-priced RTD formats.

The United States, a substantial juice-producing nation, is again in the top spot in the global fruit juice/nectar ranking, with a majority 27% share of global volume consumed in 2003. A major fruit juice processor and consumer, Germany, takes second place with a 9% share. Other important fruit juice/nectar consuming nations are China and Russia. Both countries have embraced fruit juice in a big way in recent years. China notably ranks as a significant market through its large population, but also through a dramatic spread of product availability. As more Chinese consumers have opted for juice-based drinks as a natural means of obtaining vitamins, a new wave of sophisticated processing equipment has driven product quality and thus consumption within the local market. At the same time, improved means of production have also boosted the opportunities for local Chinese fruit juice producers to sell their products abroad. In growth terms, the increasing strength of Nigeria's local fruit juice manufacturing industry, supported by the government, saw it lead the global fruit juice/nectar growth ranking in 1998–2003, with a 233% uplift over the period. Russia claimed second place in the growth ranking, up 163%, thanks to sustained marketing activity by local players, such as Wimm-Bill-Dann, and a consumer shift towards natural juice products. Kenya followed in third place, up 114% over the period.

Still drinks with less than 25% juice content have become increasingly popular in the United States. The country is a major consumer of juice-based drinks, sports drinks and other still soft drinks products, claiming a 29% volume share of global still drinks consumption. Still drinks are also tremendously popular in Japan, which claimed second position in 2003 with a 20% volume share. China, again benefiting from the juice drinks phenomenon and the surge in bottled tea products, took third place. Turning to growth in still drinks, Kenya claimed pole position, up 200% in the 1998–2003 period. However, here growth has been achieved, for the most part, from a low volume base. Similarly, both Egypt and Ireland, second and third in the growth ranking and respectively up 150 and 136%, have witnessed a new emergence of still drinks products. Yet these markets have some way to go before still drinks reach considerable scale. In a great many markets across the globe, still drinks first emerged as convenient single-serve products, often packed in cartons and delivering low cost through lower fruit juice content. Most markets have since seen still drinks evolve into more sophisticated product offerings, containing juice blends, added vitamins and minerals. Often these products have taken a more premium positioning, capitalising on and delivering to specific consumption occasions.

2.5 Soft drinks industry issues

2.5.1 Obesity

The key issue faced by the soft drinks industry in 2003 and into 2004 was the increasingly heated debate over obesity. A growing burden of Type 2 diabetes and increased levels of cancers and cardiovascular ill health have all been linked with the increased 'fatness' of many populations. According to the International Obesity TaskForce (IOTF) an estimated 300 million people around the world are obese: 'Childhood obesity has reached epidemic proportions; an estimated

22 million children under 5 years are overweight worldwide in [and] millions more older children.' Scientists have warned that intensive marketing by food and drink manufacturers to push products high in fat, sugar and salt is systematically undermining the well-being of children.

The World Health Organisation (WHO) notably sparked the obesity debate in 2002 when it released a draft report recommending the restriction of sugar-rich items, such as soft drinks, particularly those consumed by children. The WHO report stated that.

Food and food products have become commodities produced and traded in a market that has expanded from an essentially local base to an increasingly global one. Changes in the world food economy are reflected in shifting dietary patterns, for example, increased consumption of energy-dense diets high in fat, particularly saturated fat, and low in unrefined carbohydrates. These patterns are combined with a decline in energy expenditure that is associated with a sedentary lifestyle . . . For children and adolescents, prevention of obesity implies the need to . . . restrict the intake of sugars-sweetened soft drinks.

Intensified lobbying of political leaders and increased media attention on the obesity issue have combined to highlight the 'vast societal dimensions' (WHO, 2003) of the obesity problem. Soft drinks manufacturers and trade associations across the globe are locked in a debate with governments and regulators on the issue of obesity and soft drinks. The US National Soft Drinks Association (NSDA), for example, cites a sizeable body of research linking obesity with poor diet quality and insufficient exercise, rather than high consumption of soft drinks. Most researchers agree that a lack of exercise and an overall increase in the sedentary lifestyles of children need to be addressed. Yet, while flurries of scientific papers dispute the relationship between consumption of soft drinks and obesity, the attempt to attribute blame does not resolve the problem. It is all about shared responsibility. The soft drinks industry must deliver good value for consumers, a range of product choices and the necessary information for consumers to make sensible choices. The soft drinks industry cannot be blamed for increasingly sedentary lifestyles. However, alongside governments and regulators, the industry owes it to its customer base to encourage and promote a culture of activity rather than inactivity.

2.5.2 Wellness and functionality

Health issues are already influencing the dynamics of the soft drinks industry, and this influence is likely to become more pronounced as time progresses. Bottled water, fruit-based still drinks and fruit juice/nectars have gained volume on the back of an increased understanding of good health. As consumers, we wish to 'live healthy and die healthy'. At the core of this is the concept of 'wellness' – an increasing awareness of physical well-being and good health. Wellness drinks are those beverages that aid health and well-being. Consequently, a broad range of products can be said to fit the wellness mould: from bottled water to juice- and tea-based drinks as well as those with added ingredients to provide an additional functional benefit.

To feel good and be healthy has high importance for most consumers. Although broader in scope than functional beverages (i.e. those drinks with specifically added functionality), wellness drinks span the naturally functional and the scientifically enhanced. Functional soft drinks are defined as drinks providing a health benefit beyond their basic nutritional value, by virtue of their physiologically active added components. Drinks providing a health benefit based on their inherent ingredients, such as mineral water, cranberry juice and green tea, can be said to be naturally functional, unless they are fortified with vitamins or other functional ingredients. A distinction can therefore be made between products functional by nature (water and orange juice) and those made functional by producers. For functional soft drinks, the term 'health benefit' is used in its widest possible application and includes benefits such as improved sports performance, increased mental alertness and better skin, which would not normally be considered health benefits in the strictest sense. Functional soft drinks can be broken down into four main sectors: enriched beverages (such as juices and waters with added vitamins and minerals); sports drinks; energy drinks (such as Red Bull); and finally nutraceuticals (those products with targeted ingredients considered to provide specific medical or health benefits). Nutraceuticals embrace a multitude of specific claims ranging from digestive benefits and detoxification through to aphrodisiac, nicotine craving relief, cholesterol lowering and anti-ageing declarations.

Volume sales of functional soft drinks across the Western European, US and Japanese markets rose from 7.4 billion litres in 1998 to just under 12.3 billion litres in 2002, an increase of 66%. Functional soft drinks can provide high profit margins since consumers accept the idea of paying more for a product with a functional benefit. By offering a multitude of product dimensions – such as brand identity, design and experimental value – alongside a mass of claimed functional benefits, functional soft drinks also offer the possibility of extending customer satisfaction into customer enthusiasm. The functional trend offers value gains for soft drinks producers, on the condition that the consumer understands and accepts the benefit of the functional ingredients used.

Functional variants, by extending existing soft drinks product lines, provide an opportunity to explore and grow core soft drinks brands. Thus, functional drinks can allow the targeting of diverse niche consumption occasions and demographic groups. Appropriate communication of functional ingredients to the consumer is a key success factor for functional food and drink manufacturers. Legislative issues have limited functional soft drinks' availability in certain instances and must be considered carefully when formulating products for more than one geographic market. At the same time, manufacturers must not clutter the marketplace with a multitude of products utilising a confusing mixture of new ingredients and terms.

2.6 Beverage trends – outlook and conclusion

The growing importance to the consumer of health and wellness should continue to drive the growth of bottled water and still drinks in the coming years. Total world beverage consumption is forecast to approach 1.5 trillion litres in 2007, reflecting continued growth of around 2.5% a year (Figure 2.10). While the overall compound annual growth rate (CAGR) forecast for 2003–2007 stands at 2.5% for all beverages, bottled water is forecast to generate a CAGR of 6.7% over the same period, with still drinks producing a CAGR of 6.4%. Soft drinks have increased in importance in recent years against other beverage categories, and this trend is expected to continue.

Global beverage developments remain fundamentally complex. Although certain similarities in market augmentation and growth patterns can be seen in consumption dynamics across the globe, like leading soft drinks player Coca-Cola, we must 'think local, act local'. We must observe the idiosyncrasies of individual markets rather than attribute overall trends to those distinct markets. As consumers we are fundamentally different, with varying beverage needs. This applies just as much within a market as it does in the multitude of vibrant beverage markets across the globe.

Emerging markets are expected to gain in importance in the coming years. Asia contains over half the world's population, but soft drinks consumption per person remains considerably less than in North America. Asia thus holds huge growth potential for those companies willing to invest in and develop the drinks market there. Meanwhile, Eastern Europe is set to achieve strong per capita consumption growth in soft drinks during 2003–2007, boosted by sustained

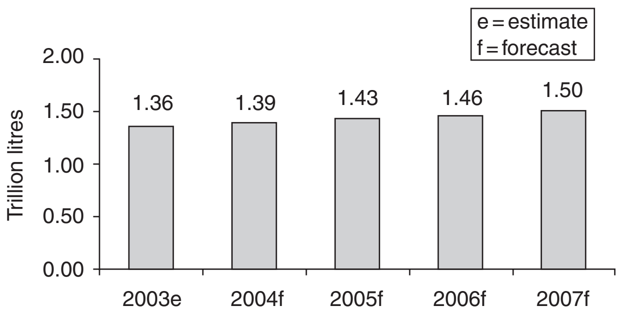

Figure 2.10 Projected global beverage consumption, 2003–2007.
Source: www.globaldrinks.com (Zenith International).

direct foreign investment as witnessed across Asia. At an early stage in the soft drinks product lifecycle, the world's developing regions clearly present significant growth opportunities.

One of the main challenges facing the soft drinks industry is not only the need to deliver optimum volume growth and adapt to changing consumer demands, but also to stimulate demand itself through greater product innovation. Innovation, whether it be in terms of packaging or product formulation, has been widespread in the soft drinks industry over the last few years. Much innovation does not actually impact upon volumes in a big way, but instead provides an opportunity for large and small soft drinks players alike to differentiate themselves and, most importantly, to positively impact the bottom line. Innovative premium priced soft drinks, such as energy drinks and other functional products, are witnessing rapid growth in the mature soft drinks markets of Western Europe and North America. These products are also generating substantial gains in the developing world. Such innovation can also provide the 'tired' soft drinks sectors and segments with an opportunity to gain new life. This has been witnessed in the case of carbonated drinks, where new pack formats, new products such as energy drinks and new low-sugar and innovative flavour variants have helped maintain momentum for the sector.

In an environment of innovation and new product development, tremendous pricing pressures remain. From the most developed markets to those that are classified among the most emerging, the squeeze on prices is being felt. Competition among soft drinks companies and a clamp-down on pricing by retailers and other outlets throughout the distribution chain are taking their toll. In this environment of 'hyper-competition' the pressures on margins are intense. Production and supply chain efficiencies must therefore be maximised if companies are to survive in today's aggressively price sensitive and highly competitive markets.

The global soft drinks market has demonstrated a tremendous amount of dynamism over the last 5 years. High levels of volume growth have been achieved by sectors such as bottled water and still drinks and by a number of countries in the developing world. Though some areas have been more spectacular in their development than others, opportunities remain in all soft drinks sectors. There are several factors that could represent potential difficulties for the global soft drinks industry, such as uncertainty in the global economy, the influence of the health debate and the ever-variable global weather. However, despite the possible pitfalls that could beset the industry, it is aided by the simple fact that consumers genuinely enjoy soft drinks products. As long as the industry can adapt itself to market and regulatory change, acknowledge the changing needs of consumers and keep them excited with innovation, the growth experienced in recent years can surely be sustained, and even bettered.

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3 Fruit and juice processing

Barry Taylor

3.1 Introduction

It could be said that freshly pressed fruit juice provides the truly natural answer to all the requirements of a soft drink: thirst quenching, fresh, healthy, flavoursome, nutritional and, of course, natural. So why should it have been necessary to look any further towards the creation of different flavour types and the myriad of drink varieties that has appeared over the years in beverage markets around the world? Necessity, being the mother of invention, has been the driving force in all this. In the early years of the industry there was a real necessity for soft drinks manufacturers, in order to stay in business, to control a major threat to their trade, that of microbial fermentation and spoilage of the bottled product.

A freshly squeezed orange or fresh pulped and strained apple would supply a fruit juice drink for immediate consumption, but to expect it to maintain its quality for even a day or two was tempting providence. Nowadays, with the benefit of ultra-high temperature (UHT) pasteurisation, aseptic packaging techniques and systems, pressed juices can be stored for extended periods with very little deterioration in quality. Previously, reliance had to be places upon the use of preservatives and 'classical' pasteurisation at lower temperatures (70ºC+) and longer holding times. As businesses grew and production and filling lines enlarged and developed, so did understanding of the need for correct sanitisation of the plant items. The progressive nature of the soft drinks industry has meant that throughout its history there have been many innovative developments, and in the early years these centred on the filling and packaging, or containerising, of beverages. During the second half of the twentieth century, apart from the continuous move towards more efficient means of production and marketing of bottled or canned soft drink products, there was much progress in our knowledge of the constituents responsible for perceived flavour notes. Advances in instrumental analytical techniques have made it possible to identify those chemicals in natural extracts (whether of fruit or botanical origin) that provide the characteristic flavour profile.

The advent of gas chromatography in the 1950s, its coupling with the diagnostic strength of mass spectroscopy (GC–MS) and the remarkable improvements in column sensitivity have no doubt been critical factors in the generation of the large numbers of beverage flavours and drink types available on today's market. This analytical knowledge, apart from leading the way to 'designer drinks', has also served to maintain and standardise the quality of a range of beverage types that still base their success upon traditional fruit juice systems. The majority of the active flavour components of most fruit types have been identified (TNO-CIVO) and provide the beverage technologist with a basis for the addition of certain characters in the development of a new product.

It is the emphasis placed upon certain flavour characters that can provide the drink with its identity in the marketplace. This can be achieved by the use of both natural and synthetic or nature-identical flavourings to create the desired top-note effects. A fruit-based drink will always declare as part of its list of ingredients the juice or juices (if mixed) used, at natural or ‘single-strength’ status. Usually appearing fairly high in order of concentration, the fruit juices will provide the generic character of the drink, with its specific identity being given by the flavouring materials chosen for a particular formulation (preferably in synergy with the flavour contribution of the juice ingredient).

The beverage technologist has a wide range of fruit types to choose from, and this chapter will investigate some of the procedures associated with the processing of these to produce fruit juices commercially.

3.2 Fruit types

3.2.1 Botanical aspects, classification of fruit types

The term ‘fruit’ is applied to a critical stage in the reproduction of botanical species throughout the plant kingdom: it is the structure that encloses, protects or harbours the seeds until they are ripe, and it often assists in their dispersal. Broadly speaking, fruits can be categorised into two groups according to their physical condition when ripe: *dry fruits* and *succulent* or *fleshy fruits*.

The widest diversity in the manner of seed dispersal is exhibited by the dry fruits. These include the windborne types such as dandelion ‘parachutes’ or sycamore ‘keys’. Mechanical scattering is exhibited by many of the legumes, whose seed pods, when fully ripened and dried out, can split with explosive force to scatter their contents in readiness for a follow-on crop. Another type is made up of those fruits, such as ‘cleavers’ or ‘dock-burrs’, that possess small hooks whereby the fruit is caught up in the fur of animals for transportation.

It is the second group, however, that will be the focus of this chapter, the succulent or fleshy fruits, where the seeds are ripened or supported within a soft or fleshy mass containing food materials that may tempt animals to eat the fruit. After being eaten by animals the fruit is digested, but the seeds, protected by a hard shell-like coating, will progress through the alimentary canal of the animal to be passed out in the faeces. This rather primitive method of propagation has been Nature’s way almost since the dawn of time, having the advantage of a ‘built-in’ seedbed growing system rich in nitrogenous compounds and often essential trace elements. The *Prunus aves* or bird cherry gained its name in recognition of this manner of propagation.

The process of evolution and natural selection ensured that the more successful fruit forms survived in harmony with their surrounding environment.

The appearance of humans on the planet and their own evolution to the status of hunter–gatherer brought a new meaning to the term 'selection', and our ancestors would have taken the first steps in categorising the available fruits according to flavour character.

Many of the commercial fruit varieties popular in the Western world have been developed from specimens whose origins can be traced back to regions east of the Mediterranean, where stone fruits (such as peach, apricot and cherry) and pome fruits (such as apple and pear) grew in fertile surroundings and became part of the staple diet of the inhabitants.

Figures 3.1–3.3 show the structures of various succulent or fleshy fruits.

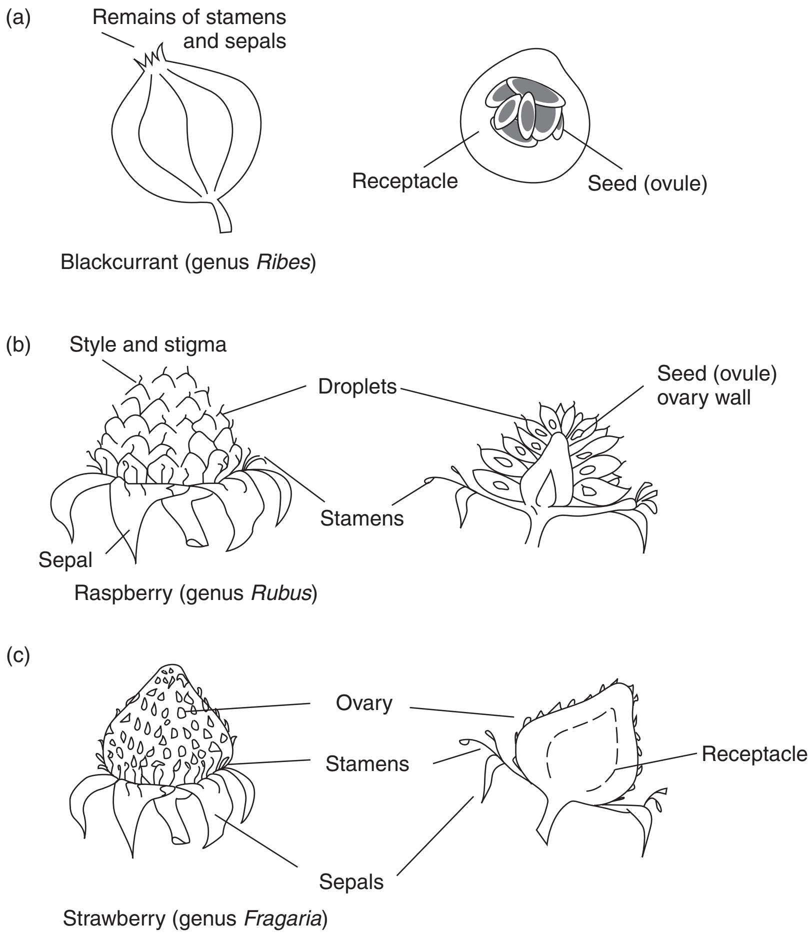

Figure 3.1 The structure of the soft fruits. (a) Currants, e.g. blackcurrant (*Ribes*); (b) raspberry (*Rubus*); (c) achenes, e.g. strawberry (*Fragaria*).

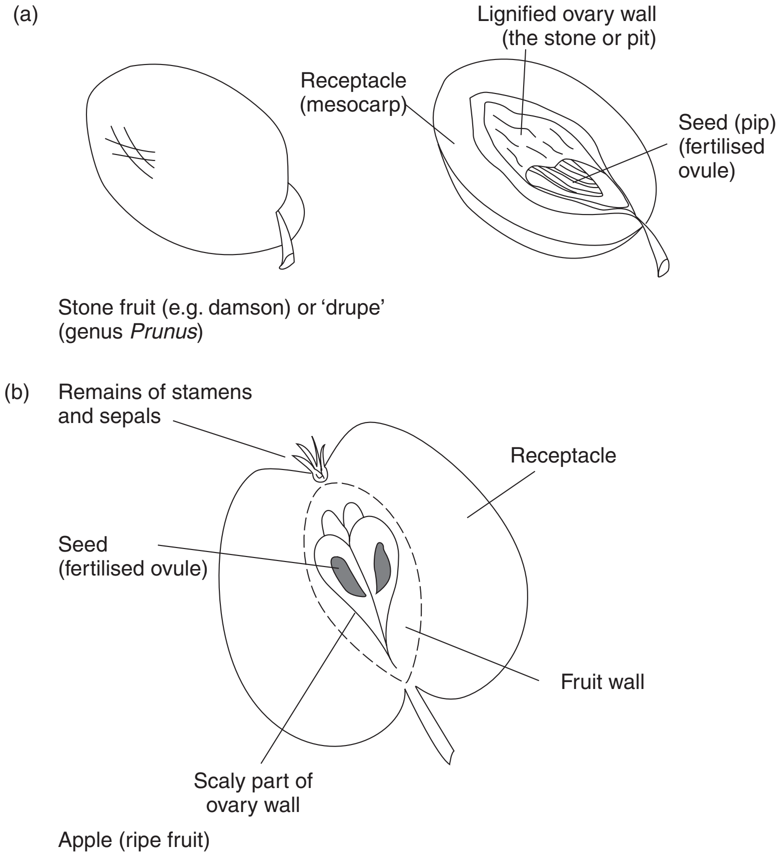

Figure 3.2 (a) A typical stone fruit or drupe, e.g. the damson (*Prunus*) and (b) structure of a ripe apple (*Malus*).

3.2.1.1 The basics of plant reproduction and fruit formation

The flower is the reproductive centre of a plant, and the series of changes resulting in the formation of a fruit starts here with the process of pollination, whereby pollen is transferred from anthers to stigma by a series of mechanisms dependent upon the plant species. In essence, these mechanisms can be initiated by an insect visiting a flower and becoming dusted with pollen from the ripe stamens (carrying the pollen-bearing anther) and then visiting another flower where the pollen on its body will adhere to the stigma of the second flower. At this stage the pollen grain, containing the male nucleus, fuses with the stigma, absorbing nutrient and sending out a growing tube that eventually reaches an ovule contained in the plant ovary. The male nucleus passes down the tube during this period and is ideally positioned to fuse with the female nucleus

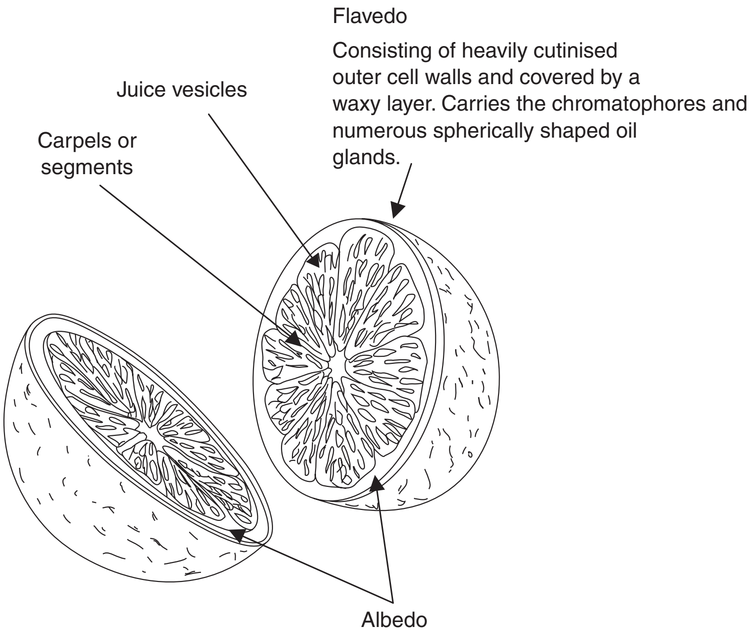

Figure 3.3 Component parts of a typical citrus fruit.

contained in the ovule. From here on rapid changes occur, resulting in the fruit, or receptacle, and seed formation.

3.2.1.2 Respiration climacteric

During their ripening or maturation stages some, but not all, fruit varieties undergo a phase of upsurge in metabolic activity known as the climacteric, a term coined by Kidd and West (1922) to describe the increase in respiration rate and heat evolution as the fruit softens and develops flavour and aroma. Fruits such as apples, pears, bananas and most stone fruit have stored reserves of starch, and during the climacteric these are converted to sugars by starch-degrading enzymes. The carboxylic acid content of the fruit also takes part in the conversion, so that acidity levels are consequently reduced as the maturation takes place.

3.2.2 Harvesting considerations for berry, citrus, pome, stone and exotic fruits

The harvesting of fruit purely for juicing purposes is a relatively minor issue compared with the size of the world market for fresh fruit for direct consumption. Ease of transportation and distribution enables an almost continuous supply of fruit from around the world as harvest time limits are compensated for in the growing areas of both the Northern and Southern hemispheres.

In the Western world, where marketing has become a major influencing factor, fresh fruit for direct consumption is produced to meet very exacting standards. We will have all seen, on the supermarket shelves, high-class displays of, for example, regulation sized Granny Smiths or Red Delicious apples, uniform in shape and appearance, presented at optimum ripeness. The main criteria for juice processing, on the other hand, are more straightforwardly that the fruit should be sound, of good quality, and of correct maturity. In order to achieve these objectives, a highly efficient international quality management system is required to operate throughout the whole procedure of growing, harvesting, ripening, storing and handling of the fruit. Apples and pears can be stored under prescribed temperature and environmental conditions in order to ripen gradually. When they are graded to meet requirements for direct sale, those fruits falling outside size limits may be sent for juicing purposes. Careful handling of the fruit during harvesting is an essential requirement, as also is the post-harvest management. If the fruit is already destined for juicing then it is more usual for mechanically harvested fruit to be transferred direct from the orchard to the processing plant to meet the demands of a tight production programme during the season. (In Europe the season usually runs from the end of June to mid-December).

However, the decision to be taken on exactly what degree of maturity should be reached by the fruit before harvesting is not an easy one, and it is here that the whole operation can encounter success or failure. The soluble solids content of the fruit is a reliable indicator in the case of non-climacteric fruit, whose composition will show little change following harvest and storage under suitable conditions. The suitability for harvesting of citrus fruit and grapes, both non-climacteric fruits, can be assessed successfully, and the ratio of soluble solids to titratable acidity is frequently used in establishing maturity criteria for these fruit. Citrus fruits are harvested direct for commercial sale or juice processing, undergoing washing, grading, and thereafter packaging or juicing. Fruits that undergo climacteric change are more difficult to deal with because the full potential of the fruits will not be known until they are fully ripened, but the important commercial decisions on harvesting cannot be left until then.

Unlike pome and citrus fruits, soft fruits are subject to rapid deterioration when even the slightest ‘bruising’ takes place, and unless they are harvested for direct use in the press it is best to subject them to rapid freezing and hold them in the temperature range of -18 to -26°C. Two forms of grading are employed for soft fruit. Selected top-quality berries will be individually quick-frozen (IQF) to be used as later whole fruit pieces in jams, yoghurts and culinary uses, whereas fruit for conversion and use as fruit pulp may be cleaned by washing it free from leaves and twigs and ‘block-frozen’. Berry fruit intended for juice production will be block-frozen as harvested, complete with any incidental stray foliage. In the case of blackcurrants, redcurrants and the like, this comprises the stalks to which the individual berries are attached; it is known as 'strig' and serves a useful function as a natural filter-aid during the pressing stage to release juice from the fruit.

Although freezing will disrupt the cell structure of soft fruit and render it 'pulpy' upon thawing out, any adverse changes to juice quality will be minimal and flavour and colour can be easily preserved by this treatment

3.3 Fruit types for processing

3.3.1 Pome fruits

Pome fruits include the apple, pear, medlar and quince. The latter two fruits are of little commercial importance in an age in which ease of harvesting and crop yields rule supreme in terms of cost considerations, but they are occasionally seen in speciality outlets. Medlar fruits are brown-skinned and apple-shaped and are best eaten after 'bletting', a process whereby the flesh softens and sweetens during storage. This can take up to 2–3 months from harvest and explains why in the Middle Ages, in Europe, the medlar was a useful fruit to store for winter consumption. Likewise the quince, similar to the pear in shape, although at one time a very popular fruit is now something of a speciality. It is ripe when the fruit turns a bright yellow. It is high in natural pectin and finds its main use in jams and jellies.

The apple and pear are of major commercial importance and are grown in most temperate regions of the world. Argentina, Australia, Bulgaria, Canada, China, France, Germany, Hungary, Italy, Japan, the Netherlands, New Zealand, Poland, South Africa, Spain, the United Kingdom and the United States are among the foremost countries growing pome fruit on a considerable scale for both home and export use. Although local varieties are found to have a following in their own regions, the world markets are dominated by perhaps no more than 20 dessert and culinary varieties, which have been selectively bred for such characteristics as disease resistance, winter hardiness, appearance (colour and shape) and texture together with high average yield. Among these are Bramley's Seedling, Brayburn, Cox's Orange Pippin, Red Delicious, Golden Delicious, Discovery, Granny Smith, Jonathon and Newtown Pippin. The main varieties of pear to be found in the market are the Bartlett or Williams Bon Chrétien, the Comice (Doyenné du Comice) and the Conference.

3.3.2 Citrus fruits

Citrus fruit varieties are grown for commercial use in many parts of the world. Originating in the southern and eastern regions of Asia, China and Cochin China, and the Malay Archipelago, the citron (*Citrus medica*) is said to have first arrived in Europe during the third century BC, when Alexander the Great conquered western Asia. Later, the orange and lemon were introduced into the Mediterranean region in the days of the Roman Empire, when trade routes from the Red Sea to India became established. Cultivation of citrus fruits has since spread worldwide to all regions where the climate is not too severe during the winter months and suitable soil conditions are available. In the United States, the notable growing areas are in Florida and California and in South America, Brazil has taken over the largest share of the world market for oranges and orange juice products. Morocco, South Africa and parts of Australia have shown increased output during recent years, although within the latter two areas yields are frequently affected by variable weather conditions. China is the largest producer after the United States and Brazil, but over 90% of its output is for the home market.

In the area of citrus juice production, Brazil, California and Florida are the major players, with Spain and Israel being notable producers of specialised concentrates. Israel enjoyed a thriving citrus industry in the 1980s, but has since lost much ground due to strong competition from South America with the emergence of the Brazilian market. In recent years a spate of drought conditions, labour shortages and political instability has done nothing to improve the situation.

The main citrus varieties for juice processing are the orange, lemon, lime and grapefruit.

3.3.2.1 Orange

The most important of all citrus fruits is the sweet orange (*C. sinensis*), and this is widely grown in those regions of the world suited to citrus. Each region tends to have its own characteristic varieties. Common varieties to be found growing in various parts are Navel, Valencia, Shamouti, Hamlin and Parson Brown. The mandarin orange (*C. reticulate*) is representative of the ‘soft citrus’ loose-skinned oranges, ‘easy peelers’, hitherto of primary importance to the Far East and now popular in other parts, including the United States and Europe. The group includes satsumas, an important crop in Japan, and the clementine, an important cultivar to be found in Mediterranean areas. Other cultivars of note are the tangor, a hybrid of mandarin and orange, and the tangelo, a hybrid of mandarin and grapefruit. A third distinctive variety of orange is the bitter orange (*C. aurantium*), chiefly represented by the Seville orange, which is grown commercially in southern Europe mainly for such products as marmalade. Compared with other citrus crops its yield is small and of little use in the juice market.

3.3.2.2 Lemon (C. limon)

An important crop in Italy and some other Mediterranean countries, the lemon is also grown commercially in the United States. The characteristic oval-shaped, yellow fruits, apart from their culinary use, are an important source of juice and flavouring for the soft drinks industry.

3.3.2.3 Grapefruit (C. paradisi)

A large round citrus fruit with a thick yellow skin and somewhat bitter pulp, the grapefruit is generally accepted to be a hybrid between the pummelo and the orange. The pummelo (*C. grandis*) originated in Asia and is grown in many eastern countries including China, Japan, India, Fiji and Malaysia. It was introduced to the West Indies during the seventeenth century by Captain Shaddock, and hence in that region it is sometimes referred to as a 'Shaddock'. Today the commercially important grapefruit is grown in many parts of the world. Notable producing countries are Argentina, Cuba, Cyprus, the Dominican Republic, Egypt, Honduras, Israel, Mexico, Mozambique, Pakistan, South Africa, Spain, Turkey and the United States. The most predominant cultivar to be seen in the market is the Marsh Seedless, followed by a red, pigmented version, the Star Ruby.

3.3.2.4 Lime (C. aurantifolia)

Limes require warm and humid weather conditions in order to thrive on a commercial scale. India, Egypt, Africa, Mexico and the West Indies are therefore prime growing areas. Mexico and the West Indies together produce a large percentage of the world's lime crop. Relative to the other citrus fruits, limes are a small round fruit; they are green or greenish yellow in colour, and not more than 8–10 cm in diameter, with a sharp, fresh and characteristic flavour.

3.4 General comments on fruit juice processing

The various types of fruit, because of their nature, shape, size, harvesting characteristics and so on, may require specialised treatment during processing. In all instances, however, the operation involves a number of stages: obtaining the fruit supply in a correct state of maturity, expressing the juice in the most efficient manner possible, and then, if required, treating the juice with enzymes (e.g. pectolases and cellulases) for clarification, followed by a suitable filtration stage before concentration and eventual packaging or storage.

In citrus fruits, where the outer skin or epicarp is a composite structure containing certain flavouring substances, it would be detrimental to juice quality if the fruit were subjected to direct pressure as is the case with the fleshy fruits, that is, soft fruits, pome fruits and stone fruits. Stone fruits, before being processed for juice separation, must first be separated from their stones, or pits, in order to facilitate ease of handling and to avoid unwanted notes in the finished product. The pits can be further processed to yield both fixed oils for application in the cosmetics industry and glycosides from which may be sourced other natural flavouring ingredients, such as benzaldehyde, a characteristic of marzipan, almond flavourings and the like.

3.4.1 Processing of 'fleshy' fruits

In the separation of juice from its fruit, the traditional method has been to apply pressure to the mashed, or pulped, fruit in order to force the liquid portion through a cloth or some form of screen. There are several styles of separator available, for both batch and continuous production, and a few of these are referred to in the following.

3.4.1.1 Pack press

The pack press is based on the traditional 'rustic' press widely used in the cider industry. The press comprises a set of frames for containing the fruit. These are loaded, in stages, by placing a loose weave cloth over each rectangular frame and adding an appropriate quantity of the fruit mash from a hopper feed above the assembly. The mash is then smoothed, or trowelled, over the frame by the operator, who then folds the cloth across to cover it over. Another frame is then placed on top and the process repeated until several filled layers are formed. The stack is built up inside a rectangular tray, or bed, which serves as both a collecting device and a platform to be raised by the action of a vertical hydraulic ram in bringing the top of the stack into contact with a fixed frame. In this way pressure is created to express the juice, which runs down into the tray for collection.

Earlier versions of the pack press were made of hardwood. Some still are, but modern pack presses are usually constructed from stainless steel and are frequently designed to accommodate two stacks for improved efficiency. These are assembled in sequence in their respective collecting trays before moving across the hydraulic ram, so that while one pressing operation is under way the next stack is being prepared and charged in readiness to follow on. Thus an almost continuous, albeit labour-intensive, pressing operation can be carried out.

The pack press is ideally suited to a relatively small production output for specialised fruit varieties, but for large-scale production fully mechanised systems are necessary.

3.4.1.2 The horizontal rotary press

Perhaps the most successful of the mechanised systems, to date, has been the horizontal rotary press designed and developed by a Swiss company – Bucher-Guyer AG, of Niederweningen/Zurich. This design carries a horizontal hydraulic piston (HP) operating within the cylindrical hollow body of the press. Between the specially designed endplate and the piston faceplate run a large number of flexible drainage cores, with well defined ribbing along their lengths to act as juice channels. Each line throughout its length is covered with a coarsely woven filter sock. The press will work, self-optimised, to operate in strict sequence with pressing periods appropriate to the fruit in process.

Mash is pumped into the press to partly fill the cylinder space. The piston then moves forward under hydraulic pressure to express the juice by consequently forcing it through the filter sleeves and along the channels in the drainage cores, to be fed through outlets in the specially designed plates for collection in a juice tank. The piston then withdraws whilst a second mash charge is received and the process repeats. During the pressing operation the endplate of the unit rotates with consequent meshing of the filter-lines, so that at the end of a pressing cycle the lines are loosely meshed together within the press-cake, or fruit pomace. As the piston moves back in conjunction with incoming mash, reverse rotation occurs, disentangling the lines and redistributing the pomace amongst the fresh charge of mash. Hence there is no build up of press-cake and maximum efficiency in terms of juice removal. Depending upon the fruit type there will be several charges of mash to feed the pressing sequence until a ‘full load’ has been achieved.

At the end of a complete pressing sequence the pomace residue is discharged by rotating the whole press body with the piston fully retracted and moving the cylinder flanges away from the endplate to create an opening through which the dry pomace is released for collection; usually along a screw conveyer with a suitable storage hopper. The standard Bucher-Guyer press, the HPX 5005i (similar to the one shown in Figure 3.4), is designed to comfortably accept a 10 tonne loading, although with suitable selection and pre-treatment of the fruit, quantities in excess of this can be handled quite easily. Good quality fruit (e.g. apples) will yield 85–95% by weight of expressed juice (depending on pre-treatment and degree of post-extraction). It is quite feasible therefore for a press of this size to handle in excess of ten tonnes of fruit mash, in stages, leaving around one tonne of pomace to discharge.

3.4.1.3 The use of centrifuges in processing

Although direct pressure has previously been an obvious choice of processing method, in comparatively recent times there has been a move towards employing centrifugal separation of juice from a continuous fruit mash stream. The modern decanter centrifuge can be used in conjunction with a pressing system as a preliminary step to increase throughput efficiency, or, when two units are used, as a complete separation system providing a coarse primary stage, followed by a final clarification stage.

Figure 3.4 Horizontal rotary press: Universal Fruit Press HP5000 (Bucher-Guyer).

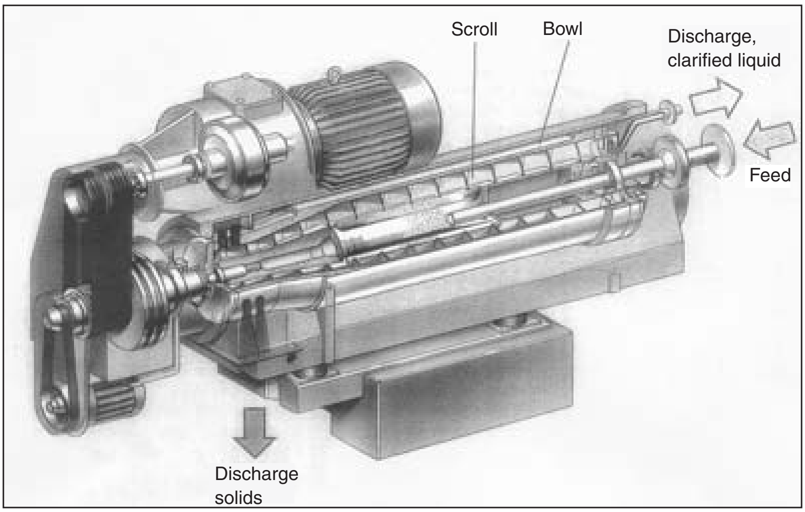

Figure 3.5 Clarifying decanter (horizontal scroll centrifuge).
Source: Courtesy of Westfalia Separator Ltd.

The decanter is a horizontal scroll centrifuge with a cylindrical-conical solid-wall bowl for the continuous separation of solids out of suspensions (see Figure 3.5). Centrifugation has a particular advantage when producing single-strength cloudy juices for direct consumption since a better definition in terms of particle size distribution can be attained. Typically, with decanter juice, 60% of the particles in suspension are smaller than 1 μM, whereas this figure is reduced to around 20% for pressed juice. Hence there is greater likelihood in the latter for instability and sedimentation. It should be noted that the major factor in the production of 'naturally cloudy' juices is the rate of processing and that to ensure stability the juicing stage should be followed immediately by pasteurisation in order to deactivate the enzymes naturally present in the fruit.

Decanters are also of use in the production of fruit purée, where the aim is to remove only the undesired particles such as pips, stalk fragments, skin fragments and coarse tissue material, leaving the crushed fruit flesh evenly distributed throughout the juice. By setting the machine parameters accordingly, the undesired components can be selectively removed from the liquid stream output of purée.

Decanters are frequently used in conjunction with disc-stack-type centrifuges in the pre-preparation of clear juices and juice concentrates, where the initial decanter treatment results in a partially clarified juice with a low level of suspended solids. This is followed by a clarification stage using a disc-stack whereby the solids are thrown outwards from the through-flow juice stream into a solids-holding space and automatically discharged therefrom as and when an optimum level of solids is reached (see Figure 3.6).

3.4.2 The use of enzymes in fruit juice processing

Pectin is an essential structural component of fruits, where in combination with hemi-cellulose it binds single cells to form the fruit tissues. Pectins are chains formed almost exclusively of D-galacturonic acid units, partially esterified with methanol. These chains are often referred to as 'polygalacturonic acid' or its synonym 'homogalacturonane'. In the immature fruit the pectins are mainly insoluble, but as the fruit ripens there is a gradual breakdown of some of the pectic substances in the skin and flesh cell walls, resulting in the formation of polysaccharide component materials. The general term 'pectic substances' covers not just pectins but just about everything resulting from the degradation processes involving pectin that take place as the fruit maturates, soluble forms included.

As fruit becomes softer, less acidic and sweeter and heads towards its optimum state of maturity, such changes need to be taken into account by the juice processor. Apples, in particular, are best processed prior to their fully ripened state, as solubilised pectin and softened fruit tissues will seriously affect the

1 Feed
2 Discharge
3 Centripetal pump
4 Sensing liquid clarifying discs
5 Disc stack
6 Separating disc for sensing liquid
7 Solids-holding space
8 Solids ejection ports
9 Annular valve
10 Drain nozzle
11 Storage chamber
12 Nozzle
13 Opening water
14 Piston
15 Closing chamber
16 Closing water
17 Sensing-liquid pump
18 To the control unit
19 Flowmeter
20 Regulating valve

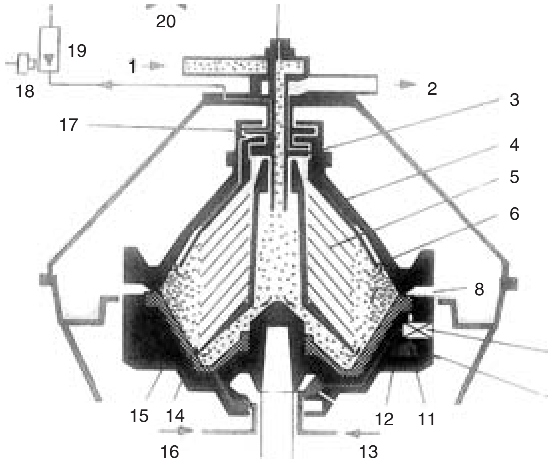

Figure 3.6 Self-cleaning clarifier (disc-stack centrifuge).
Source: Courtesy of Westfalia Separator Ltd.

efficiency of separation of the juice and low yields will result. Other fruits, such as the berry fruits, need to be fully ripened in order to optimise flavour. Broadly speaking, therefore, if the resulting juice is to be clarified, enzyme treatment is required at some stage in order to break down the pectin and to enable precipitation or sedimentation of the resulting pectic substances. Particularly important when the final product is to be a juice concentrate is that no pectin should be left available to 'jam up' the operation.

As a general rule it is the juice of the apple, not the fruit, that is to be treated with pectolytic enzymes (i.e. after pressing) whereas the soft fruit varieties are 'de-pectinised' at the mash stage, before pressing to facilitate processing. There are, of course, natural enzymes in the proximity of the fruit, borne within any traces of surface moulds, and the pre-washing stage is designed partly to reduce the effect of these. Also, the milling of fruit is violently disruptive and, apart from accessing juice for ease of separation, will initiate a host of interactions as the integral enzyme systems natural to the fruit are introduced to suitable substrate materials. Thus the presence of pectin esterase will, during the milling, pressing and clarification stages, prior to any pasteurisation or heat treatment, result in the de-methoxylation of the pectin chain, liberating methyl alcohol, which will appear later as a trace contaminant in the aroma volatiles fraction. In some processes, therefore, by passing the milled fruit pulp through a tubular heat exchanger, these rogue enzymes are denatured by a pasteurisation stage prior to juice separation.

Where the juice is destined for concentration it is essential for the pectin to be destroyed, or degraded, as already mentioned. Pectinase or poly(1,4-α-D-galacturonide) glycanohydrolase, in its commercially available form, is produced from fungal sources (i.e. *Aspergillus* sp., *Rhizopus* sp.). and possesses a wide variety of component activities. It operates comfortably between pH 2.5 and 6.0 and subject to supplier type can function well at specified temperatures between 30 and 60°C. Activities include

(1) Esterase (polymethylgalacturonase esterase), where the action is to de-esterify pectins, with the removal of methoxy groups, to form pectic acid.
(2) Depolymerases (polymethylgalacturonases with either endo- or exo-activity). Several different mechanisms take place by which the polymer chain is completely disrupted into fragments. The term 'endo-' refers to those polygalacturonases which act at random within the chain, and 'exo-' to those where the attack is sequential along the length of the chain starting at one end.
(3) Another form of depolymerase activity is given by pectinlyases, which operate at glycosidic linkages either side of which carries an esterified, or methoxylated, group.

Amylases. In the case of pome fruits other enzyme activities are sometimes required. When fruit has been picked before maturity and then ripened under controlled atmospheric conditions in a cool store, there is a likelihood of starch retention originating from the unripe fruit. This starch can become gelatinised during juice processing and can give rise to precipitation and haze effects in the final product. Amylases are used here to break down any residual starch and overcome such problems.

Cellulases. These enzymes may be used to facilitate the rapid removal of colour during fruit processing. Such enzymes have also been employed to good effect in recent years in the 'total' liquefaction of plant tissues during processing, obviating the need to use a press, yet increasing yields.

The usual way of employing enzymes, in the case of soft fruits, is to dose at the recommended level (e.g. 0.1%) into the pre-warmed mash, which is well mixed and left to stand for a recommended period and constant (optimum) temperature. For a typical pectinase, the standing period for blackcurrants and other soft fruits will be around 1.5 h. The exact time is determined by taking a series of samples of the mash during the enzymation stage, pressing or filtering off some of the juice and treating this with an excess of alcohol (e.g. 40 ml single-strength juices +60 ml alcohol in a 100 ml measuring cylinder). Dissolved pectin, if present, will be thrown out of solution as an insoluble gel, whereas if it is fully degraded, it will form a flocculent precipitate of pectic substances settling to the bottom of the measuring cylinder.

Although this is a purely empirical test, it will give an experienced operator correct information on the required process parameters.

3.4.3 Extraction of citrus juices

As already discussed, fleshy fruits yield juice upon pressing. A pre-treatment is necessary, but effectively the whole fruit (or de-pitted fruit in the case of drupe fruit) is used for the resulting extraction. Citrus fruits, however, are handled in an entirely different manner because of their structure.

3.4.3.1 The Fruit Machinery Corporation extractor

The epicarp or outer peel of the citrus fruit contains a rich source of the essential oil in oblate, spherically shaped oil glands situated in that part of the flavedo just below the waxy surface layer. Citrus oils are of great importance in the flavour industries, being widely used, as might be expected, in beverage formulations. They command a strong place in the market. Many different processes have been employed worldwide across the range of citrus types for separation of both the oil and the juice. Typically, the fruit will be passed over a rasping device, for example, an abrasive roller, to pierce and disrupt the oil glands within the flavedo layers, thus releasing the oil which is washed away for collection by a water spray. Thereafter it can be recovered by centrifugation and dried. The rasped fruit is moved into an extractor, where the juice is expressed and recovered, to leave the albedo (pith) and flavedo (outer peel).

The expressed juice is subjected to screening before being further processed. A rotary brush sieve, followed by a centrifugal separator, may be used to bring the suspended particles into the stability range of below 1 μm diameter, at which point the juice can be pasteurised and aseptically packed for direct consumption as single-strength juice or concentrated as required.

Lemons and limes are processed, in a similar manner, by direct pressure on the washed fruit.

Perhaps the most frequently encountered processing sequence is that provided by an extractor manufactured by the Fruit Machinery Corporation (FMC), generally employed for orange types. The extraction unit is designed to process individual fruits in rapid succession. In the processing hall these units are usually set up in banks of 8–10 to accommodate a continuous stream of washed and clean fruit separated into size bands. The fruit passes along feed channels to the appropriate size of extractor.

Each extractor, constructed of stainless steel, comprises two cups, one inverted above the other. During operation, fruit is received into the lower cup and the upper cup descends to press down upon it. Simultaneously, a perforated stainless steel tube is forced up through a channel in the lower cup, cutting a plug out of the bottom part of the fruit. As pressure continues to be applied, the juice is forced out through the perforations in the wall of the tube, which also acts as a screen to retain the plug, seeds and any pulp debris. The solids are later ejected at the end of the process cycle in readiness for the next pressing operation. As the two cups move together to enclose the fruit, the oil vesicles in the outer peel are ruptured to release the oil. While the juice is being expressed from inside the fruit, the oil is removed, together with some skin debris, by water spray, from the outside surface of the fruit. It passes via a conveyer to a cold-pressed oil recovery system.

The expressed juice flows into a manifold attached to the line of extractors and thence to the so-called finishing stages, wherein the juice is progressively screened to remove excess pulp and to bring it into the range set by plant quality standards, effectively minimising the level of insoluble solid material and rendering the juice suitable for further processing.

The pulp recovered during screening may be transferred to a pulp-wash operation to yield further soluble solids by counter-current extraction with water. The washed pulp may be held for further processing or included with the bulk of ejected peel material from the extractors. This is milled, treated with lime (calcium hydroxide or calcium oxide) to break down pectin and reduce water retention, pressed, dried to *c*.10–12% moisture content and finally converted to pellets. Being high in carbohydrates these are used as 'filler' in livestock feed blends.

3.5 Juice processing following extraction, 'cleaning' and clarification

The requirements for further processing will depend to some extent on the juice type. In the case of cloudy juices, heat treatment via pasteurisation is used to denature any residual enzymes released from the fruit during processing and also to eliminate spoilage organisms such as yeasts and moulds that arise also arising from the natural fruit source. The stability of a cloudy juice will very much depend upon the 'cleaning' stages after pressing, which are timed at rapid removal of pulpy and sedimentary material. Brush screens, decanter and disc-stack centrifuges may be used online to remove potentially unstable suspended solid material in order to provide a juice of uniform consistency and cloud, in readiness for pasteurisation. Pasteurisation of citrus juice is carried out at temperatures above 95°C to eliminate the undesirable effects of pectolytic enzymes, particularly pectin esterase, whose action in demethoxylation of pectin will give rise to cross-linking between resulting polygalacturonic acid molecules, gelation effects and loss of cloud stability.

Natural-strength juices for direct consumption are pasteurised and processed under aseptic conditions, where the product is then packed without contamination into sterile containers and hermetically sealed.

There are a number of well-established systems for the aseptic packaging of liquids. Notable among these are those packs constructed, box form, *in situ* on the filling line from a cardboard, aluminium, plastic laminate sheet, such as TetraPak or Combi-box. In the TetraPak system, the packing material enters the filling machine from a feed roll; the sheet contact surface is sterilised with warm hydrogen peroxide solution; it is formed into a tube, and its lower end is heat-sealed across the width; the tube is filled, sealed at the upper end, cut and then folded into a box shape. This produces a continuous output of filled cartons with premium utilisation of bulk storage capacity.

A second, and highly efficient, aseptic packaging system uses a plastic laminated bag with a novel filling and sterile sealing facility. There are several designs available but in general the bags are supplied in a sterilised condition (having been γ-irradiated for that purpose). The neck of the bag is sealed with a plastic membrane that, during the filling action, is automatically steam-sterilised and then ruptured upon the introduction of the pasteurised juice stream. When the filling operation is complete, a non-return valve enables removal of the inlet feed tube without loss of liquid and the neck is capped and sealed. The bags are available in a wide range of sizes and are designed for packing into boxes from 5 to 25 l capacity, into 200 l drums, and into rigid outer containers of up to 1000-l capacity.

Clarified juices, where pectin has been actively removed by enzyme treatment as an integral part of the process, are filtered bright and then either pasteurised or rendered sterile by the use of membrane filters to eliminate yeasts and moulds directly. Ultra-filtration techniques are used in which the juice feed flows transversely, under pressure, across a membrane support tube to avoid 'blinding' of the filter surfaces. For sterile filtrations such membranes provide a porosity value of, typically, 0.02 μm, ensuring the removal of spoilage organisms. Clarified, sterile juice passes from within the tube for storage under sterile conditions. Periodically, the circulating liquors on the feed-side of the filter are run off and replaced with fresh juice input.

3.5.1 Juice concentration, by evaporation

When the final juice product is a concentrate, the clear filtered or 'cleaned' cloudy juices are automatically subjected to heat treatment during the course of their concentration. (Figure 3.7 provides on overview of the concentration process.) Heat treatment of juices is an area where the design of process requires careful consideration in order to avoid any detrimental effects on flavour and appearance of the product. Early evaporators had demonstrated that high-vacuum–low-temperature processing produced concentrate of good flavour quality, but it was soon discovered that there was a drawback in that the heat treatment was insufficient to deactivate pectin methylesterase, which gave rise

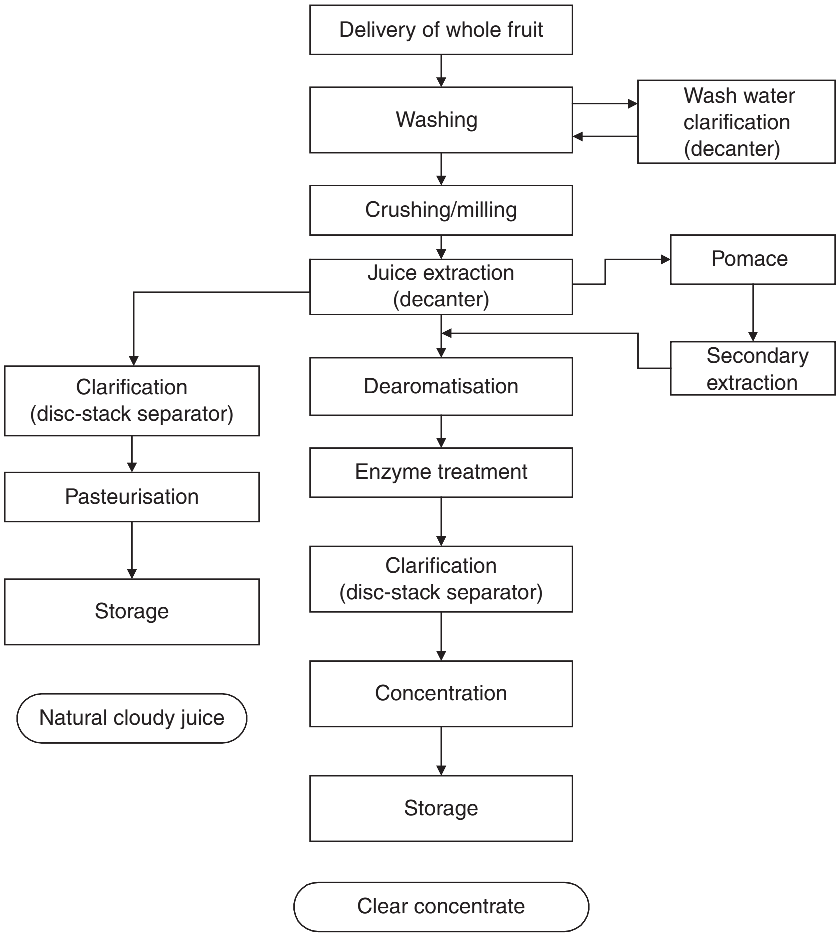

Figure 3.7 Flow diagram of the fruit juice concentration process.

to gelation in the final product. The effect was not immediately apparent, but was seen to occur after a few weeks' storage, when the contents of a filled drum of, say, orange concentrate might be found to have gelled. The introduction of short-term high-temperature pasteurisation into the concentration process, bringing the juice temperature to around 95°C with sufficient 'holding' time to eliminate microorganisms and to denature the enzyme, was used to offset the effect. Early evaporators operated by recycling the juice feed until the desired level of concentration was reached; this also increased the heating effect on the juice. The industry has since progressed to evaporators of a multi-effect, multi-stage, single-pass design, which as well as being highly efficient in heat utilisation, have the added advantage of being easier to control when there are Brix variations in the incoming single-strength juice feed. At the present time, thermally accelerated short-time evaporators (TASTE) are in worldwide use across the processing industry.

Although a combination of product quality and cost considerations will dictate the methods used for bulk processing of fruit juices, there are instances where the flavour components present in the juice are vulnerable to any form of heating during concentration. Strawberry juice is perhaps the best example of this, being one of the most heat sensitive of fruits, and it works well with alternative processes for concentration such as freeze-concentration and hyperfiltration.

3.5.2 Freeze-concentration

In general, the advantage of freeze-concentration is that there is no loss of volatile flavour components as in the evaporation procedures. Freeze-concentration is carried out by crystallisation of water from the juice. Depectinised juice is cooled to low temperature within a scraped-surface heat exchanger, to form a slurry of ripening water crystals as the heat of crystallisation is withdrawn from a well-mixed suspension of crystals. The crystals are removed by screening or centrifugation. On an industrial scale, freeze concentration is generally carried out as a multi-stage operation where the overall crystallisation rate, which strongly decreases with increasing concentration, is higher than in a single-stage operation. Viscosity is a determining value of the degree of concentration attainable. An upper limit of 55% refractometric solids (RS) is claimed, below that of standard evaporative processes. In this process the majority of the volatile flavour components are retained, although some traces (<10 ppm) are lost during the removal of the ice crystals.

3.5.3 Hyper- and ultrafiltration

By use of selective membranes, water can be removed by filtration from the juice in order to effect its concentration. Depending upon the molecular size of the compounds and the cut-off value of the membrane used, there is likely to be some loss of flavour components. These may be recovered from the permeate by distillation and returned to the juice concentrate. Concentration by these methods is less effective in terms of ‘folding’ than other methods but can provide advantages in specific cases; for example, capital costs associated with hyperfiltration are around 10–30% less than for evaporative systems with aroma recovery equipment.

Table 3.1 shows the degree of concentration that can be achieved using these different processes.

Table 3.1 Range of product concentrations technically attainable from depectinised juices

Equipment type	Juice concentrate (% m/m)
Scraped film evaporators	75–85
Plate evaporators (recycling effect)	65–75
Falling film evaporators	65–75
Freeze-concentration	45–55
Hyperfiltration	15–25

3.6 Volatile components

The characterisation of a fruit type or variety will be reflected in the flavour profile of its volatile components. Analytical techniques can produce an accurate peak profile using gas chromatography, but in simpler terms the sensory receptors of most individuals can quickly differentiate between fruit varieties. We have four basic taste senses, sometimes described as sweet, sour, acid and bitter, and these are identified by taste receptors situated mainly on the tongue. The key component of flavour differentiation, so-called top-notes and the like, is detected not so much by taste as by aroma in the nasal cavity. Thus, during the process of eating and drinking, the release of aroma volatiles can be identified and an assessment of their value arrived at.

It is not always necessary to return aroma volatiles to the juice concentrate, as this is dependent upon the intended application of the latter. However, if the concentrate is to be reconstituted for use in a soft drink formulation, then the addition of flavouring top-notes will almost certainly be a necessity. Aroma volatiles recovered during evaporative concentration of juices provide a prime source of natural flavouring. However, as already detailed, from the moment the fruit is milled in readiness for processing changes will occur due to the release of natural enzymes and the initiation of biosynthetic pathways, resulting in the formation of some atypical flavour components. Hence, to create a more representative extraction of flavour it is preferable to remove volatiles at the earliest possible stage in the process. The term 'pulp-stripping' applies to the technique of inline removal of volatiles from the pulped fruit before its depectinisation in readiness for the pressing stage. This may be effected by passing the pulped fruit through a tubular or scraped-surface pre-heater/pasteuriser into a suitably sized evaporator to remove something in the region of a 5% strip, carrying the larger part of the volatiles fraction, before cooling and returning back to process (Figure 3.8). The volatiles may be further concentrated by distillation or stored frozen at the strength produced.

The condition of incoming fruit is always of the greatest importance and there will inevitably have been a certain amount of fermentation due to natural yeasts

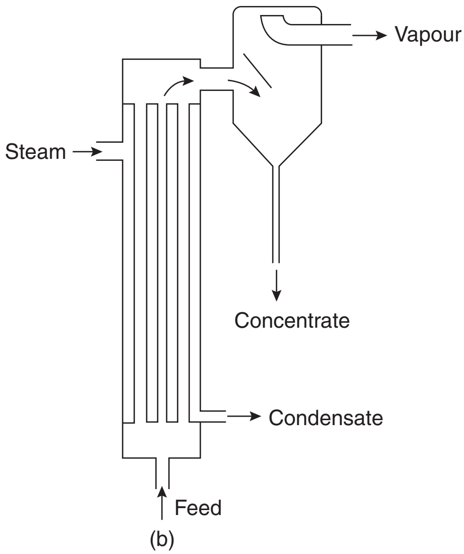

Figure 3.8 Rising film evaporator.

present on the fruit. Rapid processing minimises the effect, but there will almost certainly be a certain amount of ethanol present in the stripped volatiles fraction. Levels of ethanol up to 5 or 6% are not uncommon, and they are often deemed acceptable, but above this the balance of flavour may be seriously affected. Design of plant is critical, and the rate of heat input is carefully controlled to enable a steady-state and optimum level of volatiles strip for a particular fruit system. Separator designs vary from the simple cylindrical chamber with side entry and steady input of pre-heated pulp to high-speed tangential entry units creating a thin-film effect and larger surface area for more efficient removal of volatiles, or to the more technically efficient counter-current system producing multi-stage effects, of which the spinning cone column is a prime example.

3.6.1 Spinning cone column

Although conventional separators are successful in removing the volatiles from juice streams, the resulting strip will usually require further rectification and thereby concentration in order to render it a flavour component in its own right.

Additional heat treatment inevitably involves losses in efficiency as there are likely to be subtle changes in quality and yields due to heat degradation of some of the components. The spinning cone column (Figure 3.9) is designed to selectively isolate a volatiles fraction in one operation. It is in effect a

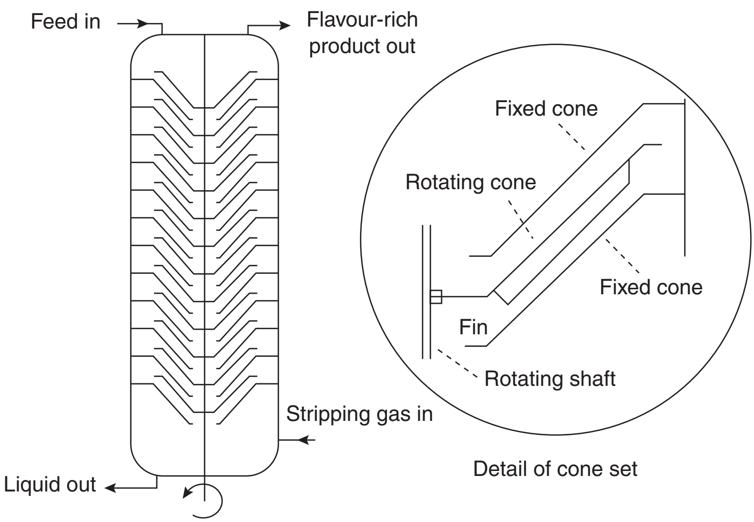

Figure 3.9 Spinning cone column.

counter-current extractor, incorporating a central, vertically placed, rotating shaft, along the length of which are a series of upturned cone-shaped cups. In operation, the cups rotate smoothly within a similar series of cone-shaped stators attached to the inner wall of the column.

The juice feed is introduced at the top of the column, on to a distributor disc, and runs down the column in a thinly layered stream between the stators and spinning cone assembly. Counter to its flow and moving up the column is a gas phase, which may be non-intrusive, such as nitrogen, or more commonly a 'live' steam feed. The multiple-stage effect produces a highly efficient separation of the component volatiles from the juice stream and an enriched fraction is produced.

3.6.2 Fruit juice volatiles composition

As already mentioned, it is the volatile constituents that serve to identify fruit type and variety. Broadly speaking, qualitative analysis will identify the principal substances present in the volatiles fraction as representative of a particular fruit type, but it is the relative proportions of these substances that will reflect the variety. Alcohols, volatile acids, esters, carbonyl compounds, and low-boiling hydrocarbons are the principal groups represented. Analysis by GC–MS (gas chromatography coupled with mass spectroscopy) can be used to provide quantification and identification of the various constituents.

3.7 Legislative concerns

Fruit juices, whether of natural strength or concentrated, are materials of commerce, to be sold direct or for use in a variety of food and drink applications. It is essential that they conform to legislative requirements for authenticity and purity, whether for labelling purposes (in avoidance of misleading statements), nutritional standards or in respect of food safety in the final product.

On a global scale, there is good correlation between quality standards for fruit and fruit juice processed in different regions. In the European Union, legislative controls are set up, or modified, by the central European Council of Ministers, usually following discussion with and between trade organisations from the EU member countries. A new directive, once agreed and approved, is translated into the statutory laws of the member countries concerned.

3.7.1 European fruit juice and nectars directive

In the European Union, Council Directive 2001/112/EC relating to fruit juices and certain other products intended for human consumption was published in the *European Official Journal* on 12 January 2002, thereby becoming law and revoking several earlier regulations (as amended) dating from 1977. The new directive, effectively an update on a range of previous amendments, invokes various provisions from the Food Safety Act 1990, Council Directive 95/2/EC (Miscellaneous additives) and Council Directive 2001/111/EC (Sugars). Definitions and descriptions are given (Schedule 1) for fruit juice and concentrated fruit juice and also for fruit juice from concentrate. It is this latter category that has involved some difficulty in the interpretation of the regulations.

The commercial importance of juice concentration lies, of course, in the area of transportation. The removal of a large volatile fraction including both water and flavour top-note components from the natural strength juice will reduce both weight and volume of product and result in worthwhile cost savings on transport. At the same time, there are benefits over natural strength juice in that the higher concentration of dissolved solids (usually 66–72% m/m RS) will exert an osmotic effect upon microorganisms, if present, and inhibit their growth, hence the greater stability and extended shelf-life under cool storage conditions of the so-called commercially sterile juice concentrates. Some difficulty is likely to occur, however, when the concentrated juice is reconstituted.

In an ideal situation, both water and flavour components removed during concentration should be returned when reproducing the diluted form. This approach would seem to be commercially untenable, yet is included in part of the legislation. Recent guidelines (UK Food Standards Authority) provide an extension of the description of ‘fruit juice from concentrate’ that enables a more practical approach. The following designation is given:

The product obtained by replacing, in concentrated fruit juice, water extracted from that juice during concentration, and by restoring the flavours and, if appropriate, pulp and cells lost from the juice but recovered during the process of producing the fruit juice in question or fruit juice of the same kind; in which the water added must display such chemical, microbiological, organoleptic and, if appropriate, other characteristics as will guarantee the essential qualities of the juice; and the product must display organoleptic and analytical characteristics at least equivalent to those of an average type of fruit obtained from fruit or fruits of the same kind.

3.7.2 AIJN guidelines

Central to the processing of fruits and juice products within the member countries of the European Union is the Association of the Industry of Juices and Nectars from Fruits and Vegetables of the European Union, familiarly termed AIJN. This body has, through its technical committee, issued guidelines detailing standards for the range of juice products manufactured in the European Union, and also on issues of good manufacturing practice (GMP). During its draft stage, the 2001/112/EC Directive led to much discussion on the subject of the restoration of flavour to fruit juice reconstituted from concentrate. AIJN guidelines have been published in clarification of this part of the directive. Add-backs, restoration aromas and flavours are focused upon in an effort to clarify any controversy over their use. Section 5 of the guidelines is perhaps the most important as it refers to permitted solvents to be used in the preparation of the restoration aromas:

'. . . Additives & Solvents permitted in the manufacture of Natural Restoration Aromas': The natural status of these aromas should not be jeopardised by the use of materials not from the named fruit. Water, food grade CO_2, and ethanol from non-GMO foodstuffs may be used as solvents, and also in extraction. Other solvents and additives, even those allowed by the EU Flavouring Directive (88/388/EU) and subsequent amendments, are not permitted.'

3.7.3 Labelling regulations and authenticity

All things should be laid bare, so that the buyer may not be in any way ignorant of anything the seller knows. (Cicero)

This has always been an objective of fair trade, but the temptation to enhance profitability too frequently intervenes and it is necessary for the buyer at times to carry out testing to ensure the authenticity of a purchased raw material so that the declaration subsequently given can meet the requirements of labelling. Fruit juices have often, over the years, been subjected to sophistry. As analytical techniques have become more objective and accurate in discovering fraud, so the methods used by the fraudsters have also become more technically devious. The most frequent method of deception is to dilute the concentrated juice product with sugar syrup and to adjust flavour levels and colour levels as necessary. Thirty years ago the 'stretching' of juices was carried out in a fairly basic fashion, using sugars that did not match the fruit, booster flavourings likewise and sometimes colours that might have been more at home on an artist's palette. Nowadays, such subterfuges would be easily recognised. Periodically, there have been attempts at using analytical profiling of certain of the juice components. Amino acid profiles were at one time thought to be an ideal route to proving authenticity, particularly in the case of citrus juice concentrate, until it was discovered that a carefully selected blend of amino acids could be used to restore balance, at least on an analytical scale, and so such testing went out of vogue. However, the testing of a wide range of parameters was for many years still the only real method of arriving at a sensible assessment of authenticity.

Perhaps the most detailed system was that developed in the early 1970s under the auspices of the Association of the German Fruit Juice Industry. A special working group of experts from backgrounds in research, industry and food control was set up with the objective of devising a method of evaluation of fruit juices. The initial results were published in 1977 under the title 'Richwerte und Schwankungbreiten bestimmter Kennzahlen für Apfel-, Trauben- und Orangensäfte' (Guide values and ranges of specific reference numbers for apple juice, grape juice and orange juice). The system of RSK-values (named from the title of this report) was officially recognised within the Federal Republic of Germany in 1980. RSK-values for other fruit juices were published in following years. The appraisal relies upon the degree of correlation within a full set of analytical values, and the process can be quite time consuming as something of the order of 30 values can be included in the assessment.

Although AIJN and RSK-value guidelines are of great use in the evaluation of fruit juices, there is always the risk of fraud. Today, more sophisticated techniques involving the analysis of stable isotope ratios of the sugars present in juices are a more reliable means of assessing authenticity.

3.8 Quality issues

Quality can be regarded as a measure of the suitability of a fruit juice, fruit juice concentrate or fruit juice extract for an intended application. In general, whatever the application, it will be the consistency in performance of the product, from batch to batch and season to season, that is the prime concern. In order to meet quality targets, therefore, it becomes critical that processing is carried out in the correct manner using fruit of an optimum level of maturity, and that the product is stored under suitable conditions to limit effects of degradation during a required shelf-life.

3.8.1 Absolute requirements

As previously indicated, the evaluation of fruit juice will, as a rule, require an expert appraisal of the entire analysis. In practice there will be key parameters to be noted and adhered to during the manufacture of fruit juices and related products. Of these, the soluble solids content and titratable acidity are the major indicators to be taken into account when identifying the status and suitability of a juice product for use in an application.

3.8.1.1 Soluble solids

The soluble solids content will relate directly to both the sugars and fruit acids, as these are the main contributors. Pectins, glycosidic materials and the salts of metals (sodium, potassium, magnesium, calcium etc.), when present, will also register a small but insignificant influence on the solids figure.

Although the soluble solids can be determined gravimetrically from a juice sample (filtered clear if necessary from any suspended solids), it is usual to refer to the more accessible determination of Brix value. Since it is dissolved solids that influence the measurement, there is a direct relationship between the Brix value and the specific gravity of the solution. Although the measurement is most accurately determined using a Brix hydrometer, which reads the percentage of sucrose directly, for the higher viscosities of concentrated fruit juices it is more convenient to use a Brix-calibrated optical refractometer, thereby providing a direct reading of % w/w sucrose. Although the term '% w/w RS' (i.e. refractometric solids) would be more appropriate to fruit juices when measured in this way than 'degrees Brix', it is the latter that is in general use in the fruit juice industry to indicate the degree of concentration or 'folding'.

Refractometer readings can be affected by the presence of other dissolved solids. The presence of fruit acids in particular can influence the refractometric Brix reading and should strictly be taken into account when calculating juice concentration. In most cases the effect is not significant; however, when there are appreciable levels of acid in the juice, for example, lemon and lime juices, there will be a need to apply the correction.

Stevens and Baier (1939) produced a table that gives a correction for obtaining Brix from refractometer readings from juices or other acid-containing sugar solutions. Based on the citric acid content of juices, the corresponding correction is to be added to the refractometer reading.

Tables 3.2 and 3.3 provide Brix values and corresponding specific gravities and sucrose contents, and Brix values for a range of fruit juices.

3.8.1.2 Titratable acidity

The acidic character of a juice contributes to its flavour type and is taken into consideration when assessing the value of the juice for inclusion into new

Table 3.2 Brix table

Degrees Brix or % w/w sucrose	Apparent SG at 20/20°C	Grams of sucrose per 1,000 ml (in air)	Degrees Brix or % w/w sucrose	Apparent SG at 20/20°C	Grams of sucrose per 1,000 ml (in air)	Degrees Brix or % w/w sucrose	Apparent SG at 20/20°C	Grams of sucrose per 1,000 ml (in air)
1.0	1.003	10.8	31.0	1.134	340.5	61.0	1.295	787.7
2.0	1.008	21.7	32.0	1.139	363.4	62.0	1.301	804.3
3.0	1.012	32.8	33.0	1.144	376.4	63.0	1.307	821.1
4.0	1.016	44.0	34.0	1.149	389.4	64.0	1.313	838.0
5.0	1.020	50.8	35.0	1.154	402.6	65.0	1.319	855.0
6.0	1.024	61.3	36.0	1.159	415.9	66.0	1.325	872.1
7.0	1.028	71.7	37.0	1.164	429.3	67.0	1.331	889.5
8.0	1.032	82.3	38.0	1.169	442.8	68.0	1.338	907.0
9.0	1.036	93.0	39.0	1.174	456.4	69.0	1.344	924.5
10.0	1.040	103.7	40.0	1.179	470.2	70.0	1.350	942.3
11.0	1.044	114.5	41.0	1.184	484.0	71.0	1.356	960.3
12.0	1.048	125.5	42.0	1.189	498.0	72.0	1.363	978.3
13.0	1.053	136.5	43.0	1.194	512.1	73.0	1.369	996.5
14.0	1.057	147.7	44.0	1.120	526.3	74.0	1.375	1014.9
15.0	1.061	158.7	45.0	1.205	540.7	75.0	1.382	1033.5
16.0	1.065	170.0	46.0	1.210	555.2	76.0	1.388	1052.2
17.0	1.070	181.4	47.0	1.216	569.8	77.0	1.395	1071.1
18.0	1.074	192.8	48.0	1.221	584.5	78.0	1.401	1090.0
19.0	1.079	204.3	49.0	1.227	599.3	79.0	1.408	1109.3
20.0	1.083	216.0	50.0	1.232	614.3	80.0	1.415	1128.6
21.0	1.088	227.7	51.0	1.238	629.4	81.0	1.421	1148.7
22.0	1.092	239.6	52.0	1.243	644.6	82.0	1.428	1168.5
23.0	1.097	251.5	53.0	1.249	660.0	83.0	1.435	1189.6
24.0	1.101	263.5	54.0	1.254	675.5	84.0	1.441	1208.2
25.0	1.106	275.6	55.0	1.260	691.1	85.0	1.448	1228.3
26.0	1.110	287.9	56.0	1.266	706.9			
27.0	1.115	300.2	57.0	1.272	722.8			
28.0	1.120	312.6	58.0	1.277	738.8			
29.0	1.124	325.2	59.0	1.283	755.0			
30.0	1.129	337.8	60.0	1.289	771.3			

beverage product formulations. Acid content (% w/w) is determined using a pH meter by direct titration against standardised alkali solution (e.g. 0.1 M sodium hydroxide) to an end-point at pH 8.1. When the juice is naturally clear, or has been clarified, and is of low colour intensity, the end-point may be accurately found using phenolphthalein as indicator.

Although there are other acids present in fruit juices (e.g. oxalic, iso-citric, tartaric), it is usual to record acidity in terms of citric acid, both for citrus fruit

Table 3.3 List of current Brix values for single-strength juices direct and from concentrate

Fruit juice	Direct juice Rel. density 20/20°C	Single-strength minimum Brix (direct juice)	From concentrate Rel. density 20/20°C	Single-strength minimum Brix
Orange	1.040	10.000	1.045	11.200
Grapefruit	1.038	9.500	1.040	10.000
Apple	1.040	10.000	1.045	11.200
Grape	1.055	13.500	1.065	15.900
Pineapple	1.055	11.200	1.052	12.800
Lemon	1.028	7.000	1.032	8.000
Passion Fruit	1.050	12.400	1.055	13.500
Mandarin	1.042	10.50	1.045	11.200
Pear	1.044	11.000	1.048	11.900
Apricot	1.041	10.200	1.045	11.200
Tomato	1.015	4.000	1.012	5.000
Blackcurrant	1.040	10.000	1.046	11.400
Sour cherry	1.050	12.400	1.055	13.500
Raspberry	1.025	6.300	1.028	7.000
Strawberry	1.025	6.300	1.028	7.000
Peach	1.036	9.000	1.040	10.000
Mango	1.057	14.000	1.061	15.000
Guava	1.034	8.50	1.038	9.500
Banana	1.083	20.00	1.088	21.000

Source: AIJN reference guidelines for the evaluation of fruit and vegetable juices. (Revised complition 2001.)

juices and for the majority of soft fruit juices. Where apple and other pome fruit juices are concerned, the major organic acids are malic and citric, although usually malic predominates. In some varieties of pears, the two acids can occur in approximately equal proportions. Nevertheless, it is general practice to quote titratable acidity for pome fruit juices as % w/w malic acid, and the adjustments required, if the juice is to be used in the production of apple-based drinks, will be effected by the addition of malic acid. Both acids are usually measured in % w/w terms in their anhydrous form, although it is sometimes convenient to determine titratable acidity for citric acid in terms of its monohydrate form, as this form of the acid may be used in the formulation of certain beverages.

As a general rule, the acidity of juices will decrease with increasing maturity of the fruit source, or with increasing levels of sugars in the resulting juice. Hence the ratio of soluble solids (e.g. Brix values) to acidity is an important value in the assessment of juice quality. The Brix/acid ratio is frequently used to establish standard sensory, or taste, qualities for incoming juice supplies and to minimise the effect of seasonal variation. The higher the Brix value in relation to the acid content of the juice, the higher the ratio and the 'sweeter' the taste.

3.8.1.3 Other quality considerations

In order to create a quality standard for a particular juice, natural strength or concentrate, there are a number of fairly routine analytical test parameters to be considered. These include

- *Specific gravity* (20°C/20°C).
- *Acidity* (% w/w) as anhydrous citric acid (or malic acid, as appropriate).
- *Colour measurements*. For clarified juices, strawberry, blackcurrant and like this will be a spectrophotometric determination, recording absorption of monochromatic light of a specified wavelength passing through a 1 cm pathway. Cloudy juices may be assessed directly by visual comparison against a recognised standard using a colorimeter.
- *Pulp content* (cloudy juices only; screened and suspended pulp determinations). In the citrus juice industry, the natural juice cells, or sacs, present in citrus juice are described as ‘screened’ or ‘floating’ pulp. Such pulp is normally removed during processing and can be added back as desired to meet a required specification. ‘Suspended’ pulp is centrifugable to a degree and may be determined thus, or by certain specialised tests where a juice concentrate is diluted into a measured volume of water and allowed to stand and settle over a specified time. These are generally empirical measurements that serve to provide comparisons within agreed parameters – for example, the Imhoff cone test, in which the sedimentable pulp is allowed to settle to the base of a calibrated (inverted) conical vessel, where its apparent volume can be read off.
- *Oil content*. Citrus juices are unique among fruit juices, where normally no oil is present, in containing residual oil after processing, or oil that has been added back to concentrates. It is essential to standardise oil levels, since much of the flavour character is established this way. The oil content is determined using either the Clevenger or Cocking–Middleton procedures. These methods make use of an oil trap apparatus in distilling off, and collecting, the volatile oil fraction of the juice. Both sets of apparatus include a calibrated vertical glass tube in which the steam-distilled volatile oil collects and where it can be easily measured and quantified with respect to the weight of juice taken.
- *Ascorbic acid*. Many juices contain ascorbic acid or vitamin C, which is quantitatively the most important vitamin in soft fruits, ranging from a negligible level in some whortleberries to around 200 mg/100 g in blackcurrants. Ascorbic acid performs a valuable function as an antioxidant in minimising degradation of certain flavour principles, and it is often important for it to be included in the processed juice or in a soft drink formulation. Levels in the range 200–400 mg/kg are typical. It should be noted that ascorbic acid can be added to natural strength juice only if it is

intended for direct use; otherwise, it should be added to the juice concentrate. Addition to natural strength juice before its concentration will result in its own degradation during the heating process and ultimate spoilage of the product when an intense browning reaction takes place.

- *Preservatives*. Modern aseptic processing techniques and packing of juices will largely obviate the need to use preservatives, but there are instances when they are required and will need to be specified. Preservatives are strictly controlled by legislation; upper limits for sorbic and benzoic acids used singly in soft drinks are 300 and 150 mg/l (drinking strength), respectively. When used at higher levels for juice concentrates, it is important to recognise the statutory levels for the final application. The use of sulphur dioxide has been severely limited in recent years, and in soft drink formulations it is now permitted, under European legislation, at just 10 ppm 'carry-over' from the use and addition of juice concentrates. High-performance liquid chromatography (HPLC) techniques for the analysis of sorbic and benzoic acids have largely replaced the earlier well-used method whereby the acid was isolated from its sample by steam-distillation and the determination carried out spectrophotometrically on the resulting distillate.
- *Yeasts, moulds and bacteria*. Because of their low pH, fruit juices will present less than ideal conditions for pathogenic bacteria species, and these therefore are generally of no major concern for the juice producer who is operating under good manufacturing practices. There are acid-tolerant bacteria, however, whose presence can give rise to off-flavours, and this effect can be encountered with citrus juices, where it is thought that both diacetyl and acetylmethylcarbinol may be metabolic products of the growth of acid-tolerant bacteria. Diacetyl will introduce a mild cheesy or buttery note to the juice, whereas acetylmethylcarbinol produces no off-flavour although its presence may indicate bacterial growth. Where appropriate, it is customary to determine the diacetyl value, and this is done using a colorimetric method involving the reaction of diacetyl isolated from a sample with α-naphthol and the comparison of the optical density of the resulting colour formation against a standard scale. The standard plate count (selected agar media, 29–31°C, 3 days) giving the total viable count (TVC) and the pour plate yeast and mould count methods (OGY agar, 20–24°C, 3–5 days) are generally used for microbiological evaluation to ensure results will be within acceptable limits. Complications can arise with certain spoilage types, where specialised control procedures will need to be applied – in particular for osmophilic yeasts, if present in concentrates, and also for spore-forming yeast or mould species, which in their inactive state can withstand the effects of pasteurisation.

3.9 Conclusions

Fruit juices are of great commercial importance in their own right as well as for direct use as ingredients in food and beverage products, and, as we have seen, they form the basis of a worldwide industry. Although production of natural strength juices can be sustained locally for direct consumption, it is the concentrates that offer advantages in transworld trade in view of savings in terms of bulk transportation. Modern methods of processing are aimed at optimising all quality factors by use of highly efficient, short-time processing followed by pasteurisation and aseptic filling. Aseptic filling techniques and sanitisation of plant facilities have now reached a very high standard. Improved conditions of storage incorporating refrigeration are used more and more to offset colour degradation effects and to maximise product shelf-life.

This chapter has touched upon some of the mechanical techniques used to express juice from the fruit source, but it should be noted that there are process variations on those listed that are subject to exclusivity within parts of the industry and the details of which are not publicly available.

Good improvements in the concentration of fruit juices have been achieved in recent years. As it is the case that the majority of concentrates are to be reconstituted in application, the quality achievable for the natural strength remake will be an important issue.

Commercial fruit juice represents the end of a carefully orchestrated chain of events starting with the selection and cultivation of certain fruit-bearing botanical species. Harvesting yields, seasonal changes and maturation, among other factors, have to be taken into account before the processing and juice production take place, and so it is not surprising that we encounter subtle variations in the taste profile of the final product. We can anticipate that in future more focus and research effort will be directed to the recovery and treatment of the aroma volatiles and other natural flavour ingredients and the manner in which their reintroduction into the diluted concentrate is carried out. It is here, in the area of taste, that the consumer finally puts product quality to the test and a resulting guarantee of satisfaction will be essential to future growth and commercial viability throughout the industry.

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4 Carbohydrate and intense sweeteners

K. O'Donnell

4.1 Overview

The profile of bulk and intense sweetener use in soft drinks has changed significantly over the last 10–15 years. Several reasons have caused this change in formulations:

(1) technical improvements in the manufacture of bulk sweeteners with different carbohydrate profiles that deliver equivalent sweetness and mouthfeel properties to sugar at lower cost;
(2) deregulation of some soft drinks markets, allowing combinations of bulk and intense sweeteners to be used in non-diet products;
(3) sweetener development – improvement of taste quality and number of intense sweeteners available for soft drinks formulations;
(4) increasing consumer awareness of healthy eating and concern about the growing incidence of obesity and Type 2 diabetes in the Western world, leading to an increased number of low- and reduced-sugar formulations.

The use of carbohydrate sweeteners in juices and drinks has increased ever since the times of Captain Cook, when sugar was used to preserve juices. Sugar (sucrose) is still regarded as the 'gold' standard for taste delivery and mouthfeel. Carbohydrate-based sweeteners still represent the largest share of the global sweetener market and currently account for 81% of sweetener usage (Cosgrove, 2003). Figures 4.1 and 4.2 show the current split globally and in the United States of various sweeteners.

High-fructose corn syrups dominate the carbohydrate-sweetened soft drinks sector in some markets – notably in the United States. However, in other markets, for example, Europe, the use of high-fructose glucose syrups (HFGS) is restricted by production quotas, and a variety of carbohydrate products including sucrose, glucose syrups, fructose and fructose syrups are used.

4.2 Carbohydrate sweeteners

A number of carbohydrate sweeteners are used in soft drinks and they provide different attributes, including sweetness, mouthfeel, stability and, in some cases, colour. Table 4.1 summarises the properties of some carbohydrate sweeteners that are, or could be, used in soft drinks.

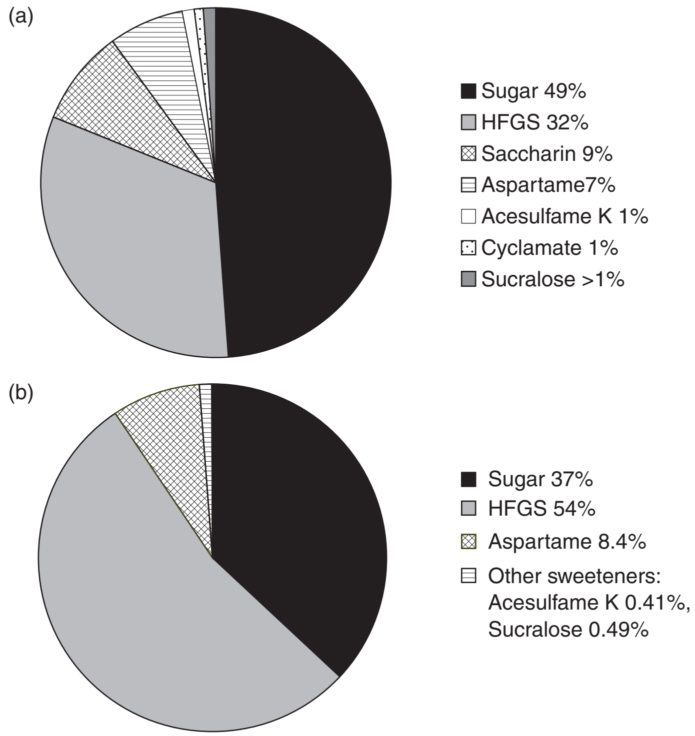

Figure 4.1 Estimated sweetener market (equivalent sweetness basis): (a) global (b) United States. *Source*: Ajinomoto.

4.2.1 Sucrose

Sucrose is regarded as the 'gold' standard for a sweet taste. It is manufactured from cane or beet and available in crystalline or liquid form. Sucrose is a disaccharide with a molecular weight of 342.31. It is available in a very pure state and in a variety of physical forms.

4.2.1.1 Manufacture

Juice extracted from cane or beet undergoes further purification steps, including precipitation, absorption, crystallisation and evaporation, which remove non-sugars and progressively concentrate the sucrose solution. The final step is crystallisation of sucrose from the syrup. This mixture of sucrose and liquor, known as the 'massecuite', is then centrifuged, and the crystals are washed and dried to a moisture content of 0.02% w/w and stored (Beesley, 1990).

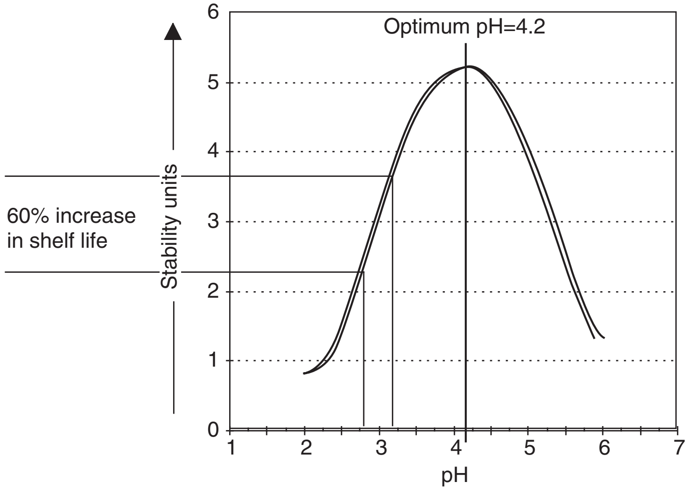

Figure 4.2 Managing pH for shelf-life: Aspartame stability as a function of pH range 1–7. *Source*: Ajinomoto Aspartame Technical Bulletin (2003).

Table 4.1 Properties of bulk sweeteners

Sweetener/ carbohydrate	Relative sweetness (Sucrose = 1)	Solubility (g/100 g water at 20°C)	Insulin-dependent metabolism	Caloric value (kcal/g)	Approval for use in soft drinks		Dietary fibre approval	
					EU	USA	EU	USA
Sucrose	1	200	Yes	4	N/A	N/A	×	×
Glucose syrup	0.6	High[a]	Yes	4	N/A	N/A	×	×
HFGS	0.9–1.0	High[a]	Yes	4	N/A	N/A	×	×
Fructose	1.2–1.8	374.83	No	4	N/A	N/A	×	×
FOS	0.3–0.6	75	No	2	N/A	N/A	✓	✓
Inulin	0	10	No	1	N/A	N/A	✓	✓
Polydextrose	0	80	No	1	Yes	Yes	✓[b]	✓
Trehalose	0.45	40.8	Yes[c]	4	Yes	Yes	×	×
Tagatose	0.92	62.0	No	1.5	No	Yes	×	×

[a] Solubility good, but varies with products of different specifications.
[b] Approved as dietary fibre in Belgium and Finland.
[c] Glucose and insulin responses lower than glucose.

Liquid sugar is readily available as an aqueous solution, usually at 67% w/w (67°Brix) at 20°C. It is manufactured by dissolving granulated sugar in water at an elevated temperature. The product may then be further refined by carbon filtration and de-ionisation. It may then be further treated using ultraviolet (UV) light to reduce microbial contamination.

4.2.2 Glucose syrups/high-fructose glucose syrups

Glucose syrups, also known as corn syrups in the United States, are defined by the European Commission (EC) as 'a refined, concentrated aqueous solution of D(+)-glucose, maltose and other polymers of D-glucose obtained by the controlled partial hydrolysis of starch' (Howling, 1984). Glucose syrups were first manufactured industrially in the nineteenth century by acid hydrolysis of starch. Hydrochloric acid was normally used, because sulphuric acid caused haze in syrups due to insoluble sulphates. The source of starch can vary; in the United States corn is widely used, whereas in other parts of the world wheat, potato and cassava starch are also employed. Acid hydrolysis of starch is still used today. The method is non-specific, but if conditions are tightly controlled, it is possible to make products with a reasonably consistent carbohydrate profile.

The degree of hydrolysis is defined by the 'DE' value or 'dextrose equivalent'. Starch (with no hydrolysis) has a DE of 0. Glucose or dextrose, which is starch that has been totally hydrolysed, has a DE of 100. The DE value is the total reducing sugar content of the syrup, compared to D(+)-glucose on a dry matter basis. As the DE increases, the viscosity of glucose syrups decreases, due to the proportion of shorter chain molecules increasing relative to the longer polysaccharide chains. In the soft drinks industry glucose syrups with DE values in the range 42–63 are commonly used.

Enzymes are also used to hydrolyse starch to glucose syrups, and these give a greater degree of control over the sugar profile of the resulting syrup. The availability of commercial isomerase enzymes in the 1970s, which are capable of converting glucose to fructose, allowed significant development of the production of high-fructose corn syrups with fructose levels of 42% and a sweetness level equivalent to sucrose. Use of separation technology allowed further refinement of these products to give 55% fructose syrups. These types of syrups are used extensively in the soft drinks industry, particularly in the United States. It is now possible to tailor-make syrups with a given carbohydrate profile to optimise the combination of specific properties of the different carbohydrate fractions.

In soft drinks, glucose syrups are used to provide sweetness and mouthfeel to products and occasionally specific physiological properties in sports and energy drinks. Glucose syrups are significantly less sweet than corresponding sucrose solutions (glucose has a relative sweetness of 0.6), unless they have a high fructose content.

4.2.3 Fructose (levulose)

Fructose can also be used as a sugar substitute in crystalline or syrup form. It is present naturally in many fruits and in honey, but commercially it is manufactured using sucrose as a starting material. Sucrose is first hydrolysed to a glucose–fructose mixture. The monosaccharides glucose and fructose are separated using chromatography and the fructose is then crystallised.

Fructose is unique among known sugars in being sweeter than sucrose. In solution, fructose can exist as four or five isomers, and the relative sweetness of a solution is dependent upon the equilibrium between the sweeter pyranose isomers and the less sweet furanose isomers, which is in turn dependent on such conditions as pH and temperature. In cold conditions the pyranose form predominates and, therefore, fructose solutions are sweeter (Danisco Sweeteners, 2003). Fructose has a clean, sweet taste; it is also synergistic with many bulk and intense sweeteners and is often used at low levels to improve the taste profile of some intense sweeteners. It is very soluble and also relatively hygroscopic, compared with sucrose (Danisco Sweeteners, 2003).

Fructose has some interesting physiological properties. It is a monosaccharide sugar with an energy content of 4 kcals/g (17 kJ/g) but due to its increased sweetness can be used at lower levels than sucrose. Fructose is slowly absorbed and metabolised by the body, independent of insulin production, and does not cause rapid rises in blood glucose after ingestion. It is, therefore, suitable for diabetics and also for use in drinks intended to act as a slower, more sustained energy source. Owing to its limited effect on blood glucose, it is a low glycaemic index sweetener (compared with glucose). This is an area of increased nutritional interest and may be a stimulus to the greater use of fructose in drinks. Fructose has also been shown to have an increased satiety effect, compared with other sweeteners (Spitzer and Rodin, 1987). Mineral absorption (iron and calcium) has also been shown to be positively affected by the incorporation of fructose into the diet (Holbrook *et al.*, 1989).

Chemically, fructose is very active and it readily takes part in maillard reactions, which may cause browning in some products. It is available in crystalline anhydrous form and also in high-concentration syrups.

4.3 Overview – intense sweeteners

The use of intense sweeteners in soft drinks has increased dramatically over the period 1985–2004. Saccharin was the first high-intensity sweetener to be marketed and its usage increased during the First World War as a result of sugar scarcity. Cyclamate entered the UK market during the 1960s, but was controversially banned in many countries as it was thought to be a potential carcinogen.

The 1970 cyclamate ban ended the use of saccharin/cyclamate blends in many soft drinks markets. The effect of this was that the low-calorie soft drinks market remained small and static owing to the poor taste quality of products available. The introduction in 1982–83 of aspartame in particular, and acesulfame K to a lesser extent, into the global soft drinks market dramatically improved the taste quality of sugar-free soft drinks formulations. There followed a period of rapid growth in the low-calorie sector. The use of intense sweeteners in soft drinks was given a further boost in the UK market when, in 1995, the requirement for a minimum carbohydrate level of 4.5°Brix in non-low-calorie products was removed. Products were reformulated to incorporate blends of intense sweeteners and low levels of carbohydrate sweeteners (around 0.5–3.0°Brix) to deliver cost savings without compromising taste quality. Over time, and as the use of intense sweeteners expanded, optimisation of the sweetener blends continued to deliver excellent tasting products. Currently, in the UK market, 50% of all beverages contain intense sweeteners, even though the diet market is only 25% of the total (Cosgrove, 2003). In the United Kingdom, Diet Coke now outsells regular Coke (*The Grocer*, 2003).

Harmonisation of the EU Sweetener Regulations in 1994 (94/35/EC; as amended in 1996 – 96/83/EC) saw approvals for aspartame and acesulfame K extend across the European Union and for the reintroduction of cyclamate into the UK market. In 2002, sucralose was approved for use in the United Kingdom – its first introduction into a European market. In 2004, sucralose approval was extended across the EU.

Formulators in most markets now have a wide range of sweeteners available to use either alone or in combination. As Figures 4.1 and 4.2 show, the main intense sweeteners in use in soft drinks today are acesulfame K, aspartame, saccharin and cyclamate. Currently of less importance commercially (either because they are new to the market or because they have not found significant use in soft drinks), but still approved for use in soft drinks in some markets, are thaumatin, neohesperidin dihydrochalcones, alitame, stevioside, sucralose and neotame.

4.3.1 Sweetener approval

The main intense sweeteners currently permitted for use in the major markets of Europe and the United States are not natural and have had to go through a food additive approval procedure. Within the European Union, approval is controlled by the EU Commission, with the aim of achieving harmonisation across member states. The current system allows for temporary national approval (and this was the mechanism by which sucralose was approved in the United Kingdom). This in turn allows the other EU countries time to review the data and either approve or reject each product within a specified period. Within the European Union, approved sweeteners are assigned an 'E' number and can also be assigned a maximum use level within a specific application (e.g. soft drinks). The maximum use levels for sweeteners in soft drinks in the European Union are given in Table 4.2.

When assigning maximum use levels, the regulators take into account a sweetener's likely intake across different population groups and its 'acceptable

Table 4.2 Physiochemical and regulatory properties of intense sweeteners

Sweetener	EU E-Number	Solubility (g/l)	Caloric value (kcal/g)	Nutritive	ADI (mg/kg bw)	Approval in EU	Approval in USA	Max. use level (ppm)[a]	Relative sweetness[b]
Acesulfame K	950	270	0	No	9	Yes	Yes GMP	350	200
Aspartame	951	10	4	Yes	40	Yes	Yes GMP	600	200
Cyclamate	952	200	0	No	7	Yes	No	250	35
Saccharin (sodium salt)	954	3,700	0	No	2.5	Yes	Yes GMP	80	400
Stevioside	Not approved	1.2	0	No	N/A	No	No	N/A	70–300
Sucralose	955	280	0	No	15	In UK	Yes GMP	300	450
Alitame	N/A	131	1.4	Yes	0.1	No	No	N/A	2000
Neotame	N/A	12.6	0	Yes	Not assigned	No	Yes GMP	N/A	8000
NeoDHC	959	0.5	0	No	5	Yes	No	30	300–600
Salt of aspartame–acesulfame	951,950	23	2.56	Yes	—[c]	In UK and NL	—[d]	—[e]	350

[a] In soft drinks in the European Union.
[b] 5% equivalent; sucrose = 1.
[c] ADI covered by ADI for constituent parts (i.e. aspartame and acesulfame).
[d] Not required as aspartame and acesulfame are approved.
[e] Maximum level should not exceed maximum levels for constituent parts.

daily intake' (ADI). This ADI of sweeteners (and other food additives) is the estimated amount of the product expressed as milligrams per kilogram of body weight that can be consumed every day throughout a lifetime without any harmful effects. ADI levels are set by the Joint FAO/WHO Expert Committee on Food Additives (JECFA) after reviewing the toxicology data generated by feeding trials. Generally, the ADI is set at one-hundredth of the intake level that shows no harmful effects. The ADI levels for the intense sweeteners are given in Table 4.2.

In the United States, the Food and Drug Administration (FDA) assesses the safety and suitability of potential new sweeteners, giving GRAS (generally recognised as safe) status to products that are viewed as being safe and suitable for food use. In addition, the FDA can give 'good manufacturing practice' (GMP) status, which means that there are no upper limits for use of these ingredients.

4.3.2 Labelling

In the United Kingdom, intense sweeteners can be described on ingredient listings as 'Sweetener (name)' (e.g. 'Sweetener aspartame') or 'sweetener E-number' (e.g. 'Sweetener E951'). The label should make it clear to the consumer that the product contains sweetener and have 'with sweetener' close to the sales denomination. Blends of intense sweetener and sugar should have 'with sugar and sweeteners' close to the sales denomination. Products containing aspartame should have the additional notice 'contains a source of phenylalanine' (EU Food Labelling Regulations, 1996).

4.3.3 Main intense sweeteners in use in soft drinks

Physiochemical and regulatory properties of some intense sweeteners are given in Table 4.2.

4.3.3.1 Acesulfame K

Acesulfame K is the generic name for the potassium salt of 6-methyl-1,2,3-oxathiazine-4(3H)-one-2,2,dioxide. Manufacture is by chemical derivation from acetoacetic acid. It is a white, non-hygroscopic crystalline product. Its solubility in water is good. The relative sweetness of acesulfame K varies from 100 to 200, depending on concentration and application (Von Rymon Lipinsky, 1985).

This ingredient has a fast-onset sweet taste with a marked bitter aftertaste that is more noticeable at high concentrations. Combinations of acesulfame K and aspartame are synergistic with respect to sweetness intensity and sweetness quality when compared with acesulfame K alone. Synergy has also been reported with cyclamate, glucose, fructose, sucrose and sucralose (Nutrinova, 2003; Von Rymon Lipinsky & Huddard, 1983). Negative synergy (i.e. suppression) occurs with acesulfame K/saccharin combinations.

The stability of acesulfame K is very good under most food processing and storage conditions. In general, it does not appear to be reactive with other soft drink ingredients. However, the inclusion of potassium ions through the addition of acesulfame K should be taken into account when selecting clouding agents (Von Rymon Lipinsky, I.C. personal communication 1988).

The maximum use level in soft drinks within the European Union is 350 mg/l and, therefore, it must be combined with other sweeteners to reach a sweetness level of 10°Brix equivalent. Acesulfame K is excreted unchanged from the body, primarily in the urine. It is non-cariogenic and has an ADI of 9 mg/kg bw (Renwick, 1983).

4.3.3.1.1 Analysis. HPLC is the preferred method for analysis of acesulfame K with detection in the UV range (Von Rymon Lipinsky, 1985).

4.3.3.1.2 Use in soft drinks. Use of acesulfame K in soft drinks is largely in combination with one or more other intense sweeteners, in particular aspartame.

There is no commonly accepted optimum blend; maximum synergy occurs with a 50 : 50 blend, but use generally appears to have shifted from 50 : 50 blends to aspartame-rich blends in the range 30 : 70 to 10 : 90 acesulfame K : aspartame. This may be to take advantage of improved stability of these blends (see Section 4.3.3.2.3 on managing the stability of blends).

4.3.3.1.3 Regulatory. Acesulfame K is widely permitted across the globe.

4.3.3.2 Aspartame

Aspartame is the generic name for *N*-α-aspartyl-L-phenylalanine methyl ester. It is composed of two amino acids, L-aspartic acid and L-phenylalanine, joined by a methyl ester link. It was discovered in 1965 by J. Schlatter at the G.D. Searle Laboratories. It is a white crystalline product and its solubility in water is 10 g/l at 20°C; this figure increases at elevated temperatures and in acidic conditions (*Ajinomoto Aspartame Technical Bulletin*, 2003). It is sparingly soluble in other solvents.

The taste profile of aspartame is similar to sucrose sweetness (Ripper *et al.*, 1985). It is approximately 200 times as sweet as sucrose. It is synergistic with saccharin, cyclamate, stevioside, acesulfame K and many sugars, in particular fructose, but has little sweetness intensity synergy with sucralose.

The maximum use level in soft drinks within the European Union is 600 mg/l, which means that, unlike most other intense sweeteners, it can be used as the sole sweetener in soft drinks.

4.3.3.2.1 Stability. Aspartame is made up of amino acids and, therefore, unsurprisingly degrades under conditions of elevated temperature and extremes of pH. This results in a corresponding loss of sweetness. The critical factors that dictate the rate of degradation of aspartame in soft drinks are pH, temperature, moisture and time.

4.3.3.2.2 Managing stability of aspartame-containing beverages

pH The optimum pH for aspartame stability is 4.3. The closer the soft drink formulator can get to this level the better. In practice, many beverages containing aspartame have a pH in the 3.0–3.7. Small changes in pH level to get closer to the pH optimum can have a very significant impact on stability, as can be seen from Figure 4.2.

Temperature The effect of ultra-high temperature (UHT) treatments on aspartame stability is minimal, with losses in the range 0.5–5% (Shazer *et al.*, 1988).

4.3.3.2.3 Optimising stability of aspartame/acesulfame *K* blends. Drinks that use aspartame blends can often achieve excellent stability, if formulated

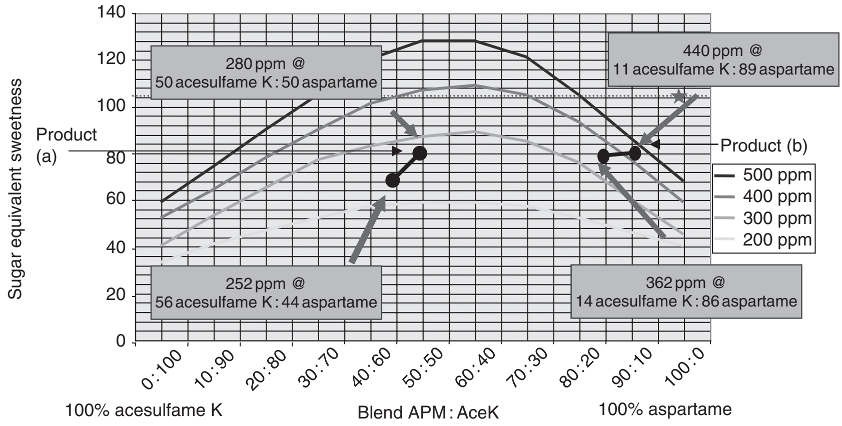

Figure 4.3 Optimisation of an aspartame/acesulfame K blend.

correctly. The data plotted in Figure 4.3 shows the synergy between aspartame and acesulfame K. Take two products:

Product (a) is a 50 : 50 blend of aspartame : acesulfame K to take maximum advantage of the synergy between the two sweeteners. The total sweetener level is 280 ppm.

Product (b) is a 90 : 10 blend of aspartame : acesulfame K to give equivalent sweetness to (a). The total sweetener level is 440 ppm.

Over time, aspartame degrades, and when the products are analysed again product (a) now contains a blend of 141 ppm acesulfame K and 111 ppm aspartame and will have lost a significant amount of sweetness due to

(1) loss of aspartame sweetness through degradation;
(2) loss of synergy, as we have moved down the synergy curve.

Product (b) is also re-analysed. Again, it has lost some aspartame and contains 50 ppm acesulfame K and 311 ppm aspartame, but the impact on perceived sweetness is lower because, although sweetness has been lost due to aspartame degradation, this has been compensated for by greater synergy between acesulfame K and aspartame. In this instance, we are moving up the synergy curve as aspartame degrades.

In summary, to maximize the stability of aspartame/acesulfame K blends in soft drinks, formulate on the right-hand side of the synergy curve. This is also an example of how the analysis of sweetener levels in a soft drink may not always give an accurate reflection of the perceived sweetness/acceptability of a drink.

In dry beverages aspartame is stable for several years. Analysis of aspartame is generally by HPLC (MacArthur *et al.*, 2002).

4.3.3.2.4 Metabolism. Aspartame is metabolised by the body into its two constituent amino acids and methanol. These hydrolysis products are handled by the body in the same way as the aspartic acid, L-phenylalanine and methanol from other commonly consumed foods. It adds nothing new to the diet.

4.3.3.2.5 Phenylketonuria. Phenylketonuria (PKU) is a genetic condition whose sufferers have an inability to metabolise the essential amino acid L-phenylalanine. Their intake of this amino acid from any source (e.g. milk, vegetables, meat and aspartame) must be strictly controlled from birth to adulthood. It is for this reason that an aspartame-containing product requires the statement that it 'contains a source of phenylalanine' on the pack.

Aspartame is non-cariogenic and, like all amino acids and protein products, has a caloric value of 4 kcal/g. However, as the level at which it is used is so low, its contribution to the caloric value of soft drinks is negligible.

4.3.3.2.6 Regulatory. Aspartame is permitted across the world in all major markets. It has an ADI of 40 mg/kg bw, which is significantly higher than other sweeteners. It is ironic that, historically, this sweetener has been the subject of allegations about adverse health effects. Evidence put forward has been largely anecdotal and picked up and promoted via the internet or the lay press. Scientifically controlled peer-reviewed studies have consistently failed to link aspartame consumption, even of abuse levels, to adverse health effects. The most recent review, of over 500 studies and reports about aspartame conducted by the Scientific Committee on Food (SCF), concluded that

Aspartame is unique among the intense sweeteners in that the intake of its component parts can be compared with intakes of the same substances from natural foods. It is clear that the consumption of aspartame represents only a minor source of aspartic acid, phenylalanine or methanol in the diet. (SCF, 2002)

4.3.3.2.7 Salt of aspartame and acesulfame. A salt of aspartame and acesulfame is now available. The product is a chemical combination of aspartame and acesulfame in a ratio of 64 : 36 on a weight basis. This product was given 2 years' temporary national approval in the United Kingdom (Statutory Instrument 2003 number 1182). It also has temporary approval in The Netherlands (Staatscourant, 17 July 2002), and it can be used in the United States, Canada, China, Mexico and Russia. In 2004, amendment of the EU Sweetener Regulation saw extension of the approval to all EU markets. In solution, the salt breaks up to form aspartame and acesulfame. The relative sweetness is 350 (HSC, 2003).

4.3.3.3 Alitame

Alitame is the generic name for L-α-aspartyl-N-(2,2,4,4-tetramethyl-3-thetanyl)-D-alaninamide hydrate. Pfizer patented the sweetener in 1980 (Pfizer, 1980). It is a white, non-hygroscopic crystalline powder with good solubility in water. Alitame is approximately 2000 times as sweet as sucrose (10% equivalent). The taste quality is similar to that of sucrose and aspartame.

Alitame is an amino acid derivative and, therefore, not completely stable. It does hydrolyse in acid conditions, but is more stable than aspartame under certain conditions (Pfizer, 1987). Alitame is partially metabolised and absorbed in humans and is excreted as a mixture of its metabolites and unchanged alitame (Pfizer, 1987).

The caloric value of alitame is 1.4 cal/g. JECFA have assigned an ADI of 0.1 mg/kg bw. The Committee on Toxicology (COT) ADI is 0.3 mg/kg bw. Alitame is currently permitted for food use in China and Australia. Submissions for approval have been made to the FDA. In Europe, a submission will be made to the EU Commission and also temporary approval will be sought in the United Kingdom via the Food Standards Agency (Koivistoinen, 2003).

4.3.3.4 Cyclamate

With the general name of cyclohexylsulphamate, this sweetener was discovered in 1937 by Michael Sveda at the University of Illinois. The sodium salt is the most commonly used form. It is a white crystalline salt with good solubility. The relative sweetness of cyclamate is comparatively low, at approximately 35, in most food systems (Bakal, 1983). The taste quality of cyclamate as a sole sweetener has a slow onset time and can have a sweet/sour aftertaste at high concentrations (Franta *et al.*, 1986). Sweetness quality is greatly improved in combination with other sweeteners. Cyclamate is synergistic with acesulfame K (Von Rymon Lipinsky, 1985), aspartame (Searle, 1971), saccharin (Von Rymon Lipinsky, 1987) and sucralose (Tate & Lyle Plc, 2002).

Cyclamate is stable under conditions likely to be encountered in soft drinks, that is, pH range 2–7, pasteurisation and UHT treatments. Analysis is usually using HPLC. Owing to differences in chemistry between cyclamate and other intense sweeteners, cyclamate requires derivatisation before analysis by HPLC (MacArthur *et al.*, 2002).

Cyclamates are non-cariogenic and non-caloric (Bakal, 1983). The majority of people metabolise less than 10% of cyclamate intake. However, approximately 47% of the population have the ability to metabolise 20–85% cyclamate (via the gut microflora) into cyclohexylamine, in which form it is excreted (Kasperson & Primack, 1985; Renwick, 1985; TNO BIBRA, 2000).

Historically, cyclamate was used in soft drinks in the United States from 1958 and in the United Kingdom from 1964, in combination with saccharin. In 1969, it was banned in the United States for use in general purpose foods on the basis of studies suggesting it may cause bladder tumours in laboratory animals. Several other countries, including the United Kingdom, followed suit. The ban on cyclamates was controversial and the original rat study has been heavily criticised. Subsequent studies on safety have implicated cyclohexylamine (the principal metabolite of cyclamate) in high blood pressure (Eichelbaum *et al.*, 1974), testicular atrophy in rats (Mason & Thompson, 1977) and cancer promotion (Hicks, *et al.*,1975).

The FDA has, so far, refused to lift the ban on cyclamate, despite several petitions from Abbot Laboratories. Cyclamate is permitted in the European Union and came back into use in the United Kingdom via the harmonised EU Sweetener Regulations 1991. Use of cyclamate in the UK soft drinks industry is almost non-existent 10 years after its re-approval.

Cyclamate is permitted for use in over 25 countries. JECFA tripled the ADI of cyclamate to 0–11 mg/kg bw in 1982. In 2001, this was reduced to 0–7 mg/kg bw due to concerns regarding overconsumption by some population groups. The EU maximum use level in soft drinks is 250 mg/l. Therefore, cyclamate can contribute only a relatively small proportion of the total sweetness of a soft drink.

Cyclamate is used extensively in some European countries, usually at low levels as part of three- or four-way blends in combination with aspartame, acesulfame K and saccharin.

4.3.3.5 Neohesperidin Dihydrochalcone

Neohesperidin dihydrochalcone (NeoDHC) is a phenolic compound prepared from the bitter citrus flavanones naringin and neohesperidin (Horowitz & Gentili, 1985). NeoDHC is a white solid with solubility in water of 0.5 g/l, which increases with temperature, but as use level is low, sufficient for most applications.

The relative sweetness of NeoDHC is quoted at 250–1300 depending on concentration (Horowitz & Gentili, 1985). Its taste is characterised by a slow onset time and a lingering liquorice aftertaste. This is a major limitation on its use in soft drinks applications. However, NeoDHC has some interesting taste-masking properties and has been used at very low levels (6–12 ppm) to mask bitterness in fruit juices (Horowitz and Gentili, 1985).

The stability of NeoDHC is good under many of the process and storage conditions that exist in soft drinks production. In the European Union it is permitted via the 1994 Sweetener in Food Regulation, with a maximum use level in soft drinks of 30 mg/l. It is assigned E-number 959. It is not permitted for use in the United States. Use in soft drinks is limited by its taste profile.

4.3.3.6 Sucralose

Sucralose is the general name for 4,1′,6′-trichloro-4,1′,6′-trideoxygalactosucrose, a chemical derivative of sucrose. It is a white crystalline powder with good solubility and shows very good stability in wet and dry forms across a wide range of processing and storage conditions. At elevated temperatures, slow decomposition can occur, resulting in a colour change from white to brown (Jenner, 1988).

The sweetness quality of sucralose is similar to that of sucrose. Sucralose exhibits synergism with acesulfame K, cyclamate, saccharin and stevioside (Tate & Lyle Plc, 1985a, 1986). It is not synergistic with sucrose and shows little sweetness intensity synergy with aspartame. However, the sweetness quality of sucralose can be improved in cola by blending with aspartame (Tate & Lyle Plc, 1985b).

Sucralose is not metabolised by mammalian species and is poorly absorbed by the body. JECFA has assigned an ADI of 15 mg/kg bw. Sucralose was approved in the United States in April 1998 for use in a variety of food applications, including soft drinks. In August 1999, this approval was extended to full-category 'GMP' approval. In the United Kingdom, it was granted a temporary approval on 15 March 2002 (UK Statutory Instrument 379). In 2004, sucralose was added to the EU list of permitted sweeteners.

To date, the use of sucralose in soft drinks in the markets in which it is approved has been relatively limited. There have been a number of launches in the United States involving small- to medium-sized brands. In the United Kingdom, at the time of writing, there are only a handful of relatively minor soft drinks that have converted to sucralose.

4.3.3.7 Neotame

Neotame is the generic name for *N*-[*N*-(3,3-dimethylbutyl)-L-α-aspartyl]-L-phenylalanine-1-methyl ester. It is a derivative of aspartame and is a white powder. It is approximately 8000 times sweeter than sucrose and is, therefore, used at extremely low levels in soft drinks (e.g. 6 ppm in cola) (Prakash *et al.,* 2002).

Neotame is characterised by an intensely sweet taste with a lingering liquorice back-taste, which is more noticeable when it is used as a sole sweetener or at high levels. Combinations with other bulk and intense sweeteners improve the taste quality. Generally, acceptable products can be made without major reformulation using up to 25% of sweetener provided by neotame (The NutraSweet Company, 2003).

Neotame is an amino acid derivative and is, therefore, hydrolysed under conditions of low or high pH. Its stability will be a function of pH, temperature and time. The optimum pH range is similar to that for aspartame: pH 3.2–4.5. In dry form neotame is stable. Products containing neotame and processed by high-temperature short-time (HTST) do not show significant losses to degradation of neotame (The NutraSweet Company, 2003).

Neotame is reported to have some flavour-enhancing properties, for example, of mint. It is rapidly metabolised by the body, yielding de-esterified neotame and small amounts of methanol. It does not accumulate in the body and is eliminated via the urine and faeces. Owing to its structure, L-phenylalanine is not a metabolite and, therefore, a PKU (phenylketonuria) statement is not required. No ADI has been assigned (The NutraSweet Company, 2003).

In the United States the FDA granted general use approval for neotame as a sweetener and flavour enhancer in July 2002. At the time of writing it is also approved in Australia, New Zealand, China, Mexico, Costa Rica and Puerto Rico. Neotame was submitted to the EU SCF in 2001 for evaluation and to date no evaluation has been published. Poland has granted temporary approval for neotame and it is also approved in the Czech Republic and Romania.

4.3.3.8 Saccharin

Saccharin is the generic name for 1,2-benzisothiazolin-3-one-1,1-dioxide and has been used for over 100 years, since its discovery by Fahlberg and Remsen in 1879 and the first production patent granted in 1885. Saccharin is a white crystalline product; the sodium salt of saccharin is the commonly used form in the soft drinks industry. Solubility is excellent and stability under food and drink processing conditions is also excellent.

Saccharin has a relative sweetness of 300–500. The taste profile is marred by a bitter metallic aftertaste, which is more pronounced at high concentrations. As with the bitter back-taste of acesulfame K, some individuals appear to be more sensitive to this than others (Bartoshuk, 1979). Several products have found use as masking agents for saccharin's bitter taste, including fructose and gluconates (US Patent, 1979), tartarates (British Patent, 1975), ribonucleotides (Bakal, 1987), sugars, sugar alcohols (Hyvonen *et al.*, 1978) and other intense sweeteners.

Synergism occurs with fructose (Hyvonen *et al.*, 1978), aspartame, cyclamate (Bakal, 1987) and sucralose (Tate & Lyle Plc, 1986). Negative synergy (i.e. suppression) occurs with acesulfame K blends. Analysis of saccharin is usually done using HPLC (Hahn & Gilikson, 1987) or spectrophotometric methods (Ramappa & Nayak, 1983).

Saccharin is excreted from the body unchanged in the urine (Renwick, 1985).

Saccharin is approved widely throughout the world for food use. However, there have been several attempts to ban it due to clinical evidence based on laboratory animals indicating that high doses may cause certain cancers (Berbanic, 1986; SCF, 1984; Taylo & Weinberger, 1980) For this reason, in the United States, products containing saccharin used to have a warning printed on the packaging that saccharin had been shown to cause cancer in laboratory animals. In 2002, the requirement to put this warning on packs was withdrawn by the FDA. In Europe, saccharin has been assigned an ADI of 2.5 mg/kg bw and has a maximum use level of 80 mg/l in soft drinks. It is permitted via the EU Sweetener in Food Regulations 1996.

4.3.3.9 Stevioside

Stevioside is used as a sweetener in Far Eastern markets and in some South American countries. It is extracted from the leaves of Stevia Rebaudiana

Bertoni, a plant native to Paraguay and now commercially cultivated in Asia and South America. Several extracts from the Stevia plant are available which contain different levels of stevioside and also other sweet compounds (rebaudiosides, dulcosides). This inconsistency of extracts is probably the reason much variation exists in the data about stevioside.

Pure stevioside is a white hygroscopic powder (The Merck Index, 1976) and commercial extracts vary from cream to tan powders. The solubility of pure stevioside in water is 1.2 g/l (The Merck Index, 1976). Commercial extracts have solubilities that range from 300 to 800 g/l (Stevia Corporation, 1986). The relative sweetness of stevia extracts varies from 15 to 300 (O'Donnell, 1983; Richard, 2002; Tunaley *et al.*, 1987) The taste of stevioside is characterised by a lingering sweetness and liquorice, bitter off-taste (O'Donnell, 1983). This limits its commercial application and it is generally not used as a sole sweetener in most applications.

Various other compounds have been shown to improve the taste profile of stevia extracts, including aspartame (Chang & Cook, 1983), fructose (Pilgrim & Schultz, 1959), histidine and sucralose (Tate & Lyle Plc, 1986). The stability of stevioside extracts is generally good. Long-term stability tests in carbonated beverages indicate no degradation over 5 months at 22°C (Chang & Cook, 1983). Analysis is generally done using HPLC (Chang & Cook, 1983).

The metabolism of stevia and stevia extracts has been the subject of much discussion. The available data are inconsistent and it is unclear whether steviol, the aglycone portion of stevioside, is generated in the gut. Steviol produces a mutagen (Phillips, 1987). The generation of steviol has been demonstrated *in vitro* and *in vivo* in rats (Phillips, 1987).

The regulatory position of stevioside varies in different regions of the world. Japan is the main market for stevioside and consumes 90% of the world's supply of stevia leaves (Richard, 2002). Stevioside is used in Japan in a variety of applications, including soft drinks. In other markets, the use of stevioside, if permitted at all, is limited to supplements. In the United States, the FDA issued an import alert in May 1991 blocking the import of and sale of stevia products, following the results of a preliminary mutagenicity study. In 1995, the FDA revised the import alert to allow the sale of stevia and its extracts as a food supplement, but not as a sweetener. It currently does not have GRAS status and is considered to be an 'unsafe food additive' (Richard, 2002).

In the European Union there have been several petitions to approve stevia and its products. In 2000, the EU Commission refused marketing authorisation for Stevia rebaudiana Bertoni plants and dried leaves as a novel food or novel food ingredient (*Official Journal of the European Communities*, 2000). In October 2003, the SCF rejected a request to re-examine the restrictions on the uses of extracts of stevia. Its comment, after throroughly examining the evidence, was that 'the committee has serious doubts about the safety of stevioside and does not consider it acceptable for use in food' (European Parliament, 2003).

JECFA reviewed stevioside in 1998 but could not quantify an ADI because of inadequate data on its safety and composition (WHO, 1998). Approval in the major soft drinks markets of the United States and the European Union looks unlikely until more data that would reassure regulators that it is a safe substance are made available.

4.4 New sweeteners/bulking agents used in soft drinks

The increased nutritional awareness of consumers, together with the desire to make soft drinks more nutritionally dense and, therefore, have a healthier consumer image, has given rise to the development and use in soft drinks of other carbohydrates with specific physiological attributes as partial sugar replacers. These physiological attributes include beneficial effects on gut health and increased levels of dietary fibre. There follows a brief review of some of the potential new ingredients. General properties are summarised in Table 4.1.

4.4.1 Inulin

Inulin is extracted commercially from chicory root, which has a high inulin content (15%). Extraction procedures are parallel to those used to extract sucrose from sugar beet. Inulin is a linear β2-2 fructans, a mixture of oligo-saccharides and polymers in which the number of monomers (fructose) refers to the degree of polymerisation (DP), which varies from 2 to approximately 60 units. When hydrolysed, inulin produces fructo-oligosaccharides (FOS) with different degrees of polymerisation. Generally, fructo-oligosaccharides have DP values from 3 to 5 and inulin an average DP value of 10, although it may be much higher (Roberfroid, 2002). The DP does affect the physiochemical and physiological attributes of this product.

Inulin is soluble in water (maximum 10% at room temperature) and forms a gel-type structure. It does hydrolyse in acid conditions over time to produce fructose. It is heat stable. In soft drinks it can produce similar mouthfeel and technical properties to glucose syrup.

Inulin has no sweetness and possesses a bland taste. Physiologically, inulin behaves as a dietary fibre. At relatively high dose levels (15–40 g/day) it can have a prebiotic effect (i.e. it can selectively promote the growth of beneficial bacteria in the colon) and at high dose levels it may also have a laxative effect (Kolida *et al.*, 2002). This is dependent on the specific composition of the product and the degree of polymerisation, which can vary. The caloric value for inulin is 1 kcal/g. Its use in soft drinks is as a fibre source, prebiotic and partial sugar replacer.

4.4.2 Fructo-oligosaccharides/oligofructose

FOS and oligofuctose are fructose oligomers that are either produced by enzymic conversion of sugar or extracted from chicory, as inulin, and then hydrolysed. These products behave as soluble fibres and prebiotics. In acid conditions, they can hydrolyse, but are usually sufficiently stable for short-shelf-life juices, near-water products with low acid levels or powdered soft drinks. Prebiotic activity varies with preparation and required daily dose can be as low as 2.5–5.0 g/day for shorter chain FOS preparations (DP 2–4). Some positive effects on magnesium absorption and calcium absorption (in some populations) have also been shown (Beghin Meiji, 2001).

Products are available in dry or syrup form. They have a lower sweetness than sucrose, RS = 0.3–0.6. The caloric value in the EU is 2 kcal/g. They are relatively hygroscopic and have good solubility. Use in soft drinks and juice products is as a sugar replacer, soluble fibre and prebiotic.

4.4.3 Polydextrose

Polydextrose was the first of the 'new generation', healthier speciality carbohydrates to be used in soft drinks. In the 1980s, Otsuka in Japan launched Fibermini, which was in effect a flavoured polydextrose solution aimed at the health drink market as a fibre supplement.

Polydextrose was developed by Pfizer and is now marketed by Danisco under the Litesse brand. Polydextrose is produced from glucose, sorbitol and citric acid and, under tightly controlled processing conditions, a randomised glucose polymer is produced. It is soluble in water, neutral in taste and is available in liquid (70% solution) or dry format, as an amorphous powder or agglomerated granule. It is extremely stable to extremes of pH and temperature.

Due to its viscosity, polydextrose gives good mouthfeel in soft drinks, but does not provide any sweetness (RS = 0). The caloric level is 1 cal/g. It is partially metabolised in the large intestine (and, therefore, independently of insulin) and so is suitable for diabetic and low glycaemic index products. It does not promote tooth decay, as it is not metabolised by oral bacteria.

Polydextrose behaves as a dietary fibre and is approved as a fibre in the United States, Japan and Belgium. Its status as a dietary fibre in the rest of the European Union is currently a little unclear. It is permitted for food use in the United Kingdom under The Miscellaneous Additives Regulations and has the E-number E400.

Prebiotic dose–response studies have indicated 10–12 g polydextrose are required to give a prebiotic response (Zhong *et al.*, 2000).

It is currently used to improve mouthfeel and as a soluble fibre, prebiotic and partial sugar replacer in soft drinks.

4.4.4 Trehalose

Trehalose is a relatively new bulk sweetener with potential for use in soft drinks. It is a di-glucose sugar and it occurs in nature in shellfish and mushrooms, where it confers a degree of protection to plant and animal cells in conditions causing dehydration. This led to its use as a cryoprotectant in freeze-drying systems in the pharmaceutical industry. In food markets, its potential use is as a bulk sweetener. It is manufactured using the Hayashibara patented process using starch as a raw material. The process involves enzymatic conversion and crystallisation to the trehalose dehydrate crystal (LFRA, 2001).

In the European Union, trehalose was approved via the Novel Foods process in 2001. In the United States, it was given GRAS status in 2002. It is also approved for use in Japan.

The trehalose molecule, which is two glucose units joined by an $\alpha,\alpha,1,1$ link, gives a stable non-reducing sugar. It has low hygroscopicity and this may be useful in powdered soft drinks to improve the flowability of dry mixes. It is stable in acid conditions, unlike many sugars, and has been reported to enhance flavour profiles in some systems. Trehalose has a very high glass transition temperature and it is thought that this may be the reason for the interesting protective properties for biological cells under extreme conditions of low temperature and dryness.

Trehalose is metabolised in a similar manner to other sugars, being broken down into glucose in the small intestine and then absorbed like other sugars. Its energy value is 4 kcal/g (17 kJ/g). Absorption of glucose from larger doses of trehalose is slower than from a glucose dose, producing a lower insulin response (LFRA, 2001). This may lead to application in diabetic products and sports drinks. The relative sweetness of trehalose is 0.45 and it may, therefore, it may find application where the technical functionality of sugar is required but not the sweetness. It is less cariogenic than sucrose (Cargill, 2003).

Current use of trehalose in soft drinks is limited.

4.4.5 Tagatose

Tagatose is another potential new addition to the range of sucrose alternatives for soft drinks formulators. At the time of writing, it is not currently permitted in the European Union (approval is expected in 2005) (Eriknauer, 2003). In the United States it was granted GRAS approval in 2001 for use in a range of foods and drinks. Approval is also being sought in Asia and Australasia.

Tagatose is manufactured from lactose using technology patented by Biospherics. Lactose is enzymically converted to glucose and galactose; the galactose is then isomerised to tagatose, which is then purifed and crystallised. It is a low-calorie ketohexose reducing sugar which occurs naturally in a polysaccharide gum, from *Sterculia setigera*.

The solubility and hygroscopicity of tagatose are similar to those of sucrose, but the viscosity of solutions is lower. As a reducing sugar it is less stable in acid conditions and liable to take part in Maillard reactions.

It has been reported to be synergistic with intense sweeteners such as aspartame and acesulfame K and, when used at low levels (0.2%), improves certain flavour profiles (Eriknauer, 2003; LFRA, 2001). The relative sweetness of tagatose is 0.92. On ingestion, 20% of tagatose is absorbed in the small intestine and the rest is metabolised by the microflora of the colon. Dose–response studies indicate a prebiotic effect at 10 g/day (Eriknauer, 2003).

Consumption of tagatose does not lead to sharp rises in blood glucose or insulin levels and it is, therefore, suitable for diabetic or low glycaemic index foods and drinks. The calorie level is 1.5 g/day and it is also non-cariogenic (oral bacteria only very slowly metabolise tagatose). Despite the fact that it is a sugar, and, therefore, should fall outside the FDA definition of tooth-friendly ingredients, the FDA somewhat controversially approved it for use in tooth-friendly products (FDA, 2003).

The year 2003 saw a couple of relatively minor launches of tagatose-containing frozen 'Slurpee' type products in the United States.

JECFA has set a temporary ADI of 125 mg/kg bw (Eriknauer, 2003). This is scheduled for review by JECFA in June 2004 (Eriknauer, 2003).

4.5 The future

Soft drinks formulators today have a greater choice of bulk and intense sweeteners than ever before. Optimisation of the taste profile of drinks containing sweeteners has improved significantly over the last 20 years. This may limit the opportunities for some of the newer sweeteners, as additional taste improvement will be incremental, compared with the substantial taste improvements seen in the early 1980s, when several new sweeteners were approved.

However, as global concern regarding obesity and high sugar consumption increases, the market for lower-sugar, lower-glycaemic-load and calorie-controlled products will increase, and it follows that the use of sugar substitutes will increase. The need to optimise the taste and physiological profile of soft drinks to satisfy this market trend will present new challenges to the soft drinks formulator.

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5 Other beverage ingredients

Barry Taylor

5.1 Introduction

The wide variety of products in today's beverage market reflects a background of intense creativity where the characterisation of drink types are concerned. We have already considered, in Chapters 3 and 4, what might be termed the two major base ingredients to be found in beverage formulation, namely fruit juices and sweeteners. This chapter will look at those other ingredients whose individual contributions serve both to stabilise the drink throughout its prescribed shelf life and to ensure that it will exhibit a characteristic identity in its organoleptic performance throughout.

It is useful first to consider the background development of the soft drinks industry and how the word 'additive' found its way into the language.

5.2 Factors influencing the development of the industry

Soft drinks, or to quote their early designation, 'mineral waters' or 'table waters', appeared as a commercial prospect in the mid-eighteenth century. Consequently, their development towards full-scale production followed hot on the heels of the Industrial Revolution taking place at that time throughout Europe and the Western world. Prior to this, although freshly pressed or squeezed juices were available for direct consumption to comply with the description 'soft drink', these were inherently unstable if stored, quickly succumbing to yeast attack and resulting fermentation.

It is not surprising, therefore, that in the Middle Ages (and before) the brewing operation was the only reliable method of producing a stable beverage, as the alcoholic content produced by the fermentation of natural sugars inhibited further yeast or microbial activity. Brewing became an art, and most country estates of any substance held their own supplies of alcoholic beverages. In northern Europe these were fruit wines, produced mainly from hedgerow stock, and, in the case of those countries adjacent to the Mediterranean, wines from the ubiquitous grape. Also, beers, ciders, perrys and such like were widespread, according to the locality.

It was not until the discovery of carbon dioxide that a means of stabilising a non-alcoholic drink became attainable. During the eighteenth century there had been great developments in the discovery of gases and the composition of the air we breathe. Effervescing spa waters and natural mineral waters, with their health-benefit connotations, had been taken for some time, and great scientific interest was shown in the constitution of these waters. In 1741, the identity of carbonic acid gas (fixed air, *gas sylvestre*, carbon dioxide) in naturally occurring 'waters' was demonstrated by Dr Brownrigg, who is also credited as being the first to accomplish the artificial aeration of mineral water.

Later in the century, Dr Joseph Priestley, FRS, was able to claim the discovery – and its practical implications – of the principle and practice of charging, or saturating water with carbon dioxide by methods similar to those with which we are familiar today. In 1772, Priestly invented 'an apparatus for making aerated water' which he exhibited to the Royal College of Physicians, and upon which the college reported favourably. In 1794 Priestly and his wife moved to Northumberland, Pennsylvania. Although he was acquainted with John Adams and Thomas Jefferson, it seems he did not take up American nationality and certainly kept well away from the political affairs of the nation. Priestly may well, however, have maintained his interest in aerated waters for at about that time the seltzer fountains caught on in the United States and a new industry was about to emerge. Fuelled mainly by the Temperance movement, seltzer waters, with their healthy image, soon incorporated flavoured varieties by including such ingredients as botanical extracts and fruit juices. Soda (seltzer) fountains were in vogue in America well into the nineteenth century, each fed by a stream of carbonated water to dilute flavoured syrups. The more sophisticated designs would supply a choice of flavours, with drinks dispensed for direct consumption.

Among the earliest soda water manufacturers in Europe were Jacob Schweppes and Nicholas Paul, who were business partners in Geneva before going their separate ways and moving to England. Schweppes set up in London in 1792, making his artificial carbonated waters on what was effectively a factory scale. Paul was also operating commercially in London by 1800, and it is in this period that we find 'soda water' coming into use as a term for a product; the term seems to have been accepted by the turn of that century. Meanwhile, attention was being given to the problem of keeping the gas in the bottle, for corks, unless wet and in contact with the liquid contents, fell well short of requirements. Different designs of bottle and bottle closure resulted in order to retain the essential preserving capability of carbon dioxide.

The Victorian era was one of invention, and the new industry in aerated waters posed several challenges. Corks, being porous, had to be kept moist in order to prevent gas loss. What better way than to construct a bottle that could not stand vertically? The egg-shaped side-lying Hamilton bottle (*c*.1809) met requirements in keeping the cork permanently in contact with the liquid and was successfully used for a time, but it had disadvantages in filling and quantity storage. Glass and earthenware stoppers, and then screw stoppers made of earthenware, were tried (*c*.1843) with varying degrees of success. Hiram Codd, a Londoner, followed in 1870 with his patent globe-stoppered bottle, so constructed that the pressure of carbon dioxide gas from the charged liquid forced a glass ball against a rubber ring inserted into a groove in the bottle's neck. This formed a secure closure in operation, but needed only a slight downward pressure on the ball to release the pressure and allow the contents to be poured. A ridge of glass at the base of the bottle's neck prevented the ball from falling into the liquid contents (Figure 5.1).

Figure 5.1 Effective in retaining carbon dioxide, the Codd's bottle was widely acclaimed, but the drink components it contained proved, at times, cause for concern.

About this time, the screw (vulcanite)-stoppered bottle and the swing (ceramic) stopper, operated by a wire spring, were also in widespread use. Then towards the end of the nineteenth century there appeared an entirely new closure concept, the crown cork first devised in Baltimore in 1889 and then later awarded a patent in the United Kingdom during April 1892. This closure consisted of a thin metal disc crimped at the edges and originally lined with cork which was clamped around the bottle opening using external mechanical force. This closure is still in use today, although the cork liner has now been replaced with a soft plastic coating. When introduced the crown cork had the advantages of both saving cost and, because of its one-off usage, being more hygienic than other types of closure.

The preoccupation with aerated waters was, of course, due to the stabilising effect of the dissolved carbon dioxide gas, the first additive of major importance. While developments were taking place around the filling and containerising of the new drinks, there were other changes in formulation that would relate further to the term 'additive'.

5.3 The move towards standardisation

Flavourings, colourings, acidulants and new preservatives were tried, at times with disastrous results. There is ample evidence that with lack of statutory controls in the early stages some of the bottled products would have been lethal to the consumer. An early edition of *Skuse's Complete Confectioner*, published around 1890, contained information on cordials and other beverages, in a section on flavours and colours it seriously warned against the use of chrome yellow (lead chromate) as it had been known for certain confectioners to use a little chrome yellow to create stripes in sweets. Much of the hazard was created by the presence of impurities in some of the materials used in the manufacture of certain additives. Frequently, the sulphuric acid or vitriol used in the generation of carbon dioxide from whiting, or more effectively from bicarbonate of soda, was contaminated with metallic impurities, including arsenic and nitrogen compounds, and care had to be exercised to select the best grades.

A recurring problem, again centred on impurities, was the risk of contamination due to lead and copper coming into contact with materials used in the preparation of soft drinks. *The Mineral Waters Trade Review and Guardian* (January issue, 1875) dwells on the subject at some length, and a particular case is cited where lemon oils imported from Messina, Italy, were found to be heavily contaminated with lead following a period of storage in copper cans. The contact surfaces had been 'tinned' and it was established that the solder used for the purpose contained relatively high levels of lead.

Today the limits on impurities are well defined in legislation. Under European regulations non-alcoholic beverages for consumption without dilution are given maximum limits for lead (0.2 mg/kg), arsenic (0.1 mg/kg), copper (2 mg/kg) and zinc (5 mg/kg). In terms of food and drink additives, custom and use eventually indicated what dosage levels were acceptable, but there was no rigid system of assessment for these. In England, the Mineral Water and Food and Drugs Acts entered the statute books in 1875. These instruments indicated very clearly, but nevertheless with some generality, what the industry should not do but omitted to indicate what might be done without fear of the consequences. For instance, Section 3 of the Act (38 & 39 Vic.c.63) of 1875 stated that 'No person shall mix colour, stain, or powder . . . any article of food, with any ingredient or material *so as to render the article injurious to health* with the intent that the same may be sold in that state.' There was no restriction of colouring material of any kind other than that nothing 'injurious to health' could be used.

The absence of standards of quality or composition, apart from those relating to pharmaceutical products (the reference here was the British Pharmacopoeia), caused some confusion in the early years. Toxicology was in its infancy as a science and many of the ingredients used in the manufacture of beverages to stabilise and standardise the drink with apparent safety were subsequently found to be injurious to health at the levels used. It has been said that in the area of food and drink everything is a poison, it just depends on the dosage or intake!

The process of assessment and control has continued to the present day, such that, by and large, all food ingredients are controlled by legislation. When and where appropriate these are removed from the permitted list or limited to an acceptable daily intake (ADI).

Across the globe, countries employ their own legislative controls for food ingredients, but there are two main regions that exert great influence upon world opinion on this issue: the European system controlled by the European Parliament with designated E-numbers for permitted food additives, and the system used in the United States where at the federal level the Federal Food, Drug and Cosmetic Act (FFDCA) lays down the framework for food safety.

5.4 The constituents of a soft drink

The term 'soft drink' applies to beverages containing flavourings and/or fruit juices together with other constituents of technological or nutritional value designed to enhance the appearance and stability of the product and to ensure its organoleptic properties remain intact during a reasonable shelf life. These factors are taken into consideration in all development work, and in order to meet current stringent quality and legislative controls a new beverage is subjected to extensive trials to assess the suitability and performance of all components in its makeup. It becomes essential to arrive at the correct ingredient formulation to achieve a reproducible product.

Table 5.1 lists the functional constituents of soft drinks and their typical usage levels. Each category of ingredient, other than fruit juices and carbohydrate/intense sweeteners, is discussed in more detail in the following sections.

5.5 Water

Water, as the main component of a soft drink, usually accounts for between 85 and 95% of the product and acts as a carrier for the other ingredients. Its quality must conform to rigid requirements and not interfere with the taste, appearance, carbonation or other properties of the drink. Subject to the location of the bottling plant, the source of water and product specifications, it may be necessary to carry out treatment to improve the quality of the water used in the manufacture of soft drinks.

In most urbanised areas of the world, public water supplies can meet consumer requirements of potability, but for the soft drinks manufacturer this is not always a suitable qualification for use of the water as a raw material. Most soft drinks factories will carry out their own treatments to counteract the likelihood of a possible change in quality. This is most important in areas where variations are introduced as a result of the use of a national grid system for water supply.

In less developed countries, water treatment becomes an essential prerequisite where microbial loading could provide cause for concern. It is necessary for a full water treatment to be effective and to ensure the wholesomeness of water supplies for boiling purposes.

5.5.1 Requirements

Water should comply with the following quality requirements. It should be free from

- high levels of elements and mineral salts;
- objectionable tastes and odours;
- organic material.

It should also be

- clear and colourless;
- free from dissolved oxygen;
- sterile, that is, free from micro-organisms.

Ideally, a non-variable supply of water should be available at all seasons of the year to allow a standard manufacturing process to be established.

Table 5.1 Soft drink components, general usage and contribution

Component	Typical use level
Water (quality must meet rigid requirements)	Up to 98% v/v when high-intensity sweeteners in use
Bland carrier for other ingredients. Provides essential hydration effects to enable body metabolism.	
Sugars	7–12 % m/v when sole source of sweetener
Contribute sweetness, body to drink. Act as synergist and give balance to flavour	
Fruit juice	Widely variable usage. Usually up to 10% as natural strength, although some specialised lines in this
Provides fruit source identity, flavour, mouthfeel effects. Also contributes to sweetness and acidity.	
High-intensity sweeteners	Use based upon sucrose equivalence (e.g. aspartame might be employed at 0.40–6% m/v as sole sweetener)
Provide sweetness, calorific reduction. Synergist action. Often used in combination e.g. aspartame with acesulfame K	
Carbon dioxide	0.30–6% m/v
Provides mouthfeel and sparkle to drink (carbonates only)	
Acids (e.g. citric)	0.05–0.03% m/v
Contributes sharpness, sourness, background to flavour. Increases thirst-quenching effects	
Flavours	Nature-identical and artificial: 0.10–28% m/m Natural: up to 0.5% m/m
Provide flavour, character and identity to the drink	
Emulsion (flavour, colour, cloud etc.)	0.1% m/v
Carrier for oil-based flavours or colours. Gives cloudy effect in drink to replace or enhance cloud from natural juices	
Colours (natural or synthetic)	0–70 ppm
Standardise and identify colour tone of drink	
Preservatives	Statutory limits apply (e.g. sorbic acid up to 250 ppm in EU)
Restrict microbial attack and prevent destabilisation of the drink	
Antioxidants (e.g. BHA, ascorbic acid)	Less than 100 ppm, subject to user-country legislation
Prevent oxidation, limit flavour and colour deterioration	
Quillaia extract (saponins)	Up to 200 mg/l (EU), up to 95 mg/l (USA)
Acts to provide heading foam, mainly of use in carbonates	
Hydrocolloids (mucilaginous gums)	0.1–0.2% per GMP, minimum amount required to create desired effect
Carrageenans, alginates, polysaccharides, carboxy methyl cellulose etc. Provide mouthfeel, shelf-life stability, viscosity	
Vitamins/Minerals	ADI applies
Used in 'healthy-living' drinks to provide nutritional requirements	

5.5.2 *Quality of fresh water*

The quality of fresh water supplies will be dependent upon the geology of the catchment areas. All fresh water is derived from rainfall; following precipitation, water filters through the upper layers of the soil, extracting minerals and organic material en route. For example, rainfall on chalk areas will result in a supply of water with a high dissolved solids content, high alkalinity and total hardness, whereas the opposite is the case when rain falls on granite. In marshlands and peaty areas water may be pale yellow in colour and will contain appreciable amounts of dissolved organic matter. Such water is sometimes termed 'humic' (because the yellow colour derives from the humic and fulvic acids present) and is likely to possess an unpleasant odour and bitter taste.

5.5.3 *Water hardness*

The term 'hardness' refers to the presence of calcium and magnesium salts. Temporary hardness is due to the presence of the bicarbonates of calcium and magnesium and permanent hardness to calcium and magnesium chlorides, sulphates and nitrates. Total hardness, as might be expected, is the sum of temporary and permanent hardness. Measurement is expressed as the equivalent concentration of calcium carbonate in milligrams per litre or parts per million (m/v) and is also termed 'degree of hardness'. Approximate classifications are

Soft	< 50 mg/l as $CaCO_3$
Medium-soft	50–100 mg/l as $CaCO_3$
Hard	100–200 mg/l as $CaCO_3$
Very hard	200–300 mg/l as $CaCO_3$.

Water for use in soft drinks should ideally be soft or medium-soft.

5.5.4 *Water treatment*

The standard form of water purification involves treatment in a continuous manner with a coagulent (e.g. $Al_2(SO_4)_3$, $Fe_2(SO_4)_3$) and chlorine, together with lime to reduce the alkalinity as necessary (Figure 5.2). A gelatinous precipitate or 'floc' forms (hydroxides of Al or Fe) which absorbs foreign organic matter. The chlorine sterilises the water by virtue of its microbiocidal and oxidising properties.

After treatment, water is passed through a sand filter followed by an activated carbon filter to remove traces of chlorine, and then through a 'polishing' filter (usually a cartridge filter of pore size <10 μm).

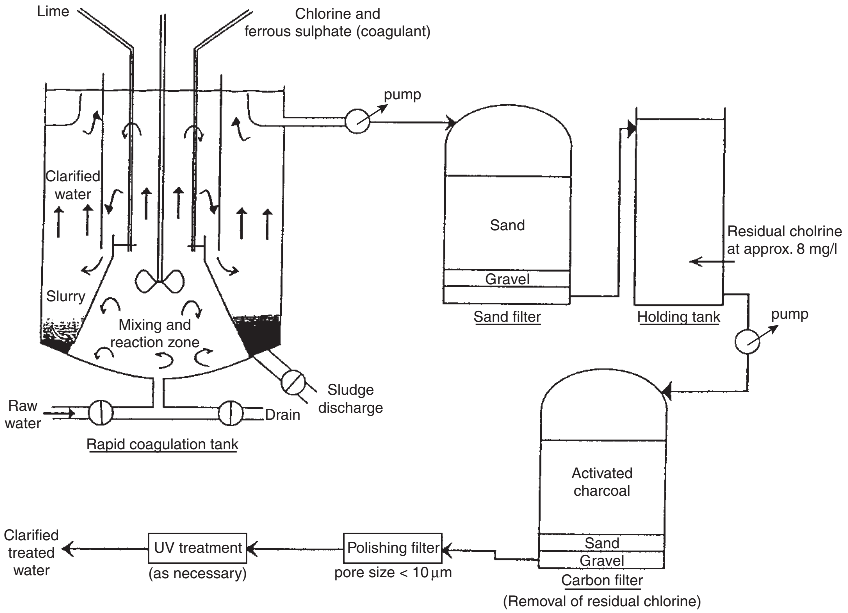

Figure 5.2 Diagram of a water treatment process using rapid coagulation.

5.5.5 Water impurities and their effect

5.5.5.1 Suspended particles

Suspended particles may consist of complex inorganic hydroxides and silicates or, sometimes, organic debris. Particles too small to be easily distinguished can cause difficulties when a drink is carbonated, acting as minute centres of instability resulting in a loss of carbonation, foaming (gushing) at the filler-head and variable fill volumes.

In non-carbonated drinks there may be visible deposits, and sometimes a neck ring in the finished product, caused by agglomeration of smaller particles. Filtration of the incoming water stream is therefore essential.

5.5.5.2 Organic matter

Organic matter is most likely to be present when the water is from soft regions and from surface water reservoir fed supplies. The organic material may include humic acid, algal polysaccharides and polypeptides, protozoa and microbial contaminants. The result is often unsightly porous crystalline precipitation during storage as the organic species, notably algal polysaccharides, respond to the lower pH conditions of the soft drink or react with other components in it.

5.5.5.3 High alkalinity

High alkalinity is due to the presence of bicarbonates, carbonates and hydroxides of the alkaline earth and alkali metals, principally calcium, magnesium, sodium and potassium. The effect of high alkalinity is to buffer acidity in a soft drink, resulting in the creation of a bland taste. It is essential therefore to maintain a consistent alkalinity level. The majority of manufacturers aim for below 50 mg/l as $CaCO_3$. Alkalinity may be reduced by coagulation treatment or by ion exchange.

5.5.5.4 Nitrates

With modern methods of intensive farming in which nitrate-based fertilisers are employed, there has been a noticeable increase in nitrate levels from aquifers lying beneath agricultural land. The recommended limit for nitrate has been given as 50 mg/l by the World Health Organization (WHO). The principal health risk of nitrates involves a condition seen in infants known as methaemoglobinaemia.

5.6 Acidulants

The use of acidulants is an essential part of beverage formulation, with the acid component usually third in order of concentration. Acidulants performs a variety of functions in addition to their primary thirst-quenching properties, which are the result of stimulation of the flow of saliva in the mouth. Because it reduces pH, an acidulant can act as a mild preservative and in some respects as a flavour enhancer, depending on the other components present. In addition, by functioning as a synergist to antioxidants such as butylated hydroxy anisole (BHA), butylated hydroxy toluene (BHT) and ascorbic acid, acidulants can indirectly prevent discolouration and ranciditye.

In carbonated beverages there is the additional effect of dissolved carbon dioxide gas. Although it is not officially recognised as an acidulant, the inclusion of carbon dioxide, under pressure, will certainly provide extra sparkle, mouthfeel, flavour and sharpness in a drink.

Table 5.2 lists the most commonly encountered acidulants.

5.6.1 Citric acid

Citric acid is the most widely used acid in fruit-flavoured beverages. It has a light fruity character that blends well with most fruit flavours, which is to be expected as it occurs naturally in many fruit types. For example, unripe lemons contain 5–8% citric acid. It is also the principal acidic constituent of such fruits as blackcurrants

Table 5.2 Acidulants used in beverages formulations

Acidulant	Molecular weight	Melting point (°C)
Citric acid, 2-hydroxy-1,2,3-propane tricarboxylic acid, $HOOCCH_2C(OH)(COOH)CH_2COOH$	192.1	152–154
Tartaric acid (D-tartaric) 2,3-dihydroxy butanedioic acid $HOOCCH(OH)CH(OH)COOH$	150.1	171–174
Phosphoric acid orthophosphoric acid H_3PO_4	98.0	42.35
Lactic acid (DL-lactic) 2-hydroxy propanoic acid $CH_3CH(OH)COOH$	90.1	18
Malic acid (D-malic) 2-hydroxy butandioic acid $HOOCCH(OH)CH_2COOH$	134.1	98–102
Fumaric acid *trans*-butenedioic acid $HOOCCH{=}CHCOOH$	116.1	299–300
Acetic acid ethanoic acid CH_3COOH	60.0	16–18

and cranberries and is associated with malic acid in apples, apricots, blueberries, cherries, gooseberries, loganberries, peaches, plums, pears, strawberries and raspberries. It is associated with isocitric acid in blackberries and tartaric acid in grapes.

Citric acid was originally produced commercially from lemons, limes or bergamots by pressing the fruit, concentrating the pressed juice and precipitating citric acid as its calcium salt, from which it was subsequently purified. It is now produced by the action of enzymes on glucose and other sugars.

Citric acid is a white crystalline solid and it can be purchased as a granular powder in its anhydrous state or as the monohydrate. Present-day soft drink formulations usually employ the anhydrous form, which is seen to have cost-in-use advantages over what was the more traditionally used monohydrate.

5.6.2 Tartaric acid

Tartaric acid occurs naturally in grapes, where it is present as the acid potassium salt. During the fermentation of grapes, tartaric acid precipitates from solution as crystals, as its solubility decreases with the increasing alcoholic concentration of the wine. Tartaric acid is also a natural component of numerous other fruits such as the currants, blackberries and cranberries.

Tartaric acid can be obtained in four forms: *dextro*-, *laevo*-, *meso*- and the mixed-isomer equilibrium, or racemic, form. Commercially, it is usually available as *dextro*-tartaric acid. This acid has a sharper flavour than citric and it may therefore be used at a slightly lower level to give equivalent palate acidity. (Palate acidity is a purely subjective evaluation and it is generally agreed that a number of acids can be used at concentrations different from those indicated by their chemical acid equivalent, see Table 5.3.)

Tartaric acid can be isolated from the crude deposit of tartrates obtained during the fermentation of wine, in a similar manner to that used for citric acid, that is, by leaching the deposit with boiling HCl solution, filtering it and re-precipitating the tartrates as the calcium salt. The free acid is obtained by treatment of calcium tartrate with sulphuric acid and further purification by crystallisation. Tartaric acid (*dextro*-form) is a white crystalline solid with melting point (m.p.) 171–174°C. Tartaric acid has a strong, tart taste and it complements natural and synthetic fruit flavours, especially grape and cranberry.

If used in beverages, tartaric acid must be perfectly pure and guaranteed for food use. One problem that may need to be addressed is that tartaric acid salts, particularly the calcium and magnesium tartrates, have lower solubility than citric acid. Consequently, there is a tendency for unsightly precipitates of insoluble tartrates to form in hard water, and in such conditions it is preferable to use citric acid.

5.6.3 Phosphoric acid

Phosphoric acid is the only inorganic acid to be widely used in food preparations as an acidulant. It does, however, occur naturally in the form of phosphates in some fruits, for example, limes and grapes. In the soft drinks industry its use is confined almost entirely to cola-flavoured carbonated beverages, where its

Table 5.3 Palate acidity equivalents

Acid	Concentration (g/l)
Acetic	1.00
Ascorbic	3.00
Citric	1.22
Fumaric	1.08
Lactic	1.36
Malic	1.12
Phosphoric	0.85
Tartaric	1.00

Note: These concentrations, in water, were considered to be equivalent (tartness, sourness) from taste trials carried out in the laboratories of Borthwicks Flavours (now Danisco (UK) Ltd.), Wellingborough in 1990. Although subjective they give a proximate comparison of the pure acid effect in solution.

special type of astringent acidity complements the dry, sometimes balsamic, character of cola drinks.

Phosphoric acid has a drier, and perhaps sharper, flavour than either citric or tartaric acid, tasting rather of flat ‘sourness’, in contrast with the sharp fruitiness of citric acid. It therefore appears to blend better with most non-fruit drinks.

Pure phosphoric acid is a colourless crystalline solid (m.p. 42.35°C) and it is usually employed in solution as a strong syrupy liquid, miscible in all proportions with water. It is commercially available in solution concentrations of 75, 80 and 90%. The syrupy nature of its solution occurs at concentrations greater than 50% and is the result of hydrogen bonding between the phosphoric acid molecules.

Phosphoric acid is corrosive to most construction materials and rubber-lined steel or food-grade stainless steel holding vessels are generally recommended.

5.6.4 Lactic acid

Lactic acid is one of the most widely distributed acids in nature and it is used to a great extent by the food industry. Its use in beverages, however, is limited. It has a mild taste relative to the other acids and is used in soft drinks as a flavour modifier or enhancer rather than as an acidulant.

Lactic acid is supplied commercially as an odourless and colourless viscous liquid. It is produced via the fermentation, using lactic acid bacteria, of carbohydrates such as corn, potato or rice starch, cane or beet sugar, or beet molasses.

5.6.5 Acetic acid

Acetic acid has a very limited use in beverages, only finding use where its vinegary character can contribute to a suitable flavour balance in the intended product. It is seldom used in anything except non-fruit beverages.

Pure glacial acetic acid is a colourless crystalline solid (m.p. 16°C) with a suffocating, pungent aroma. It is one of the strongest of the organic acids in terms of its dissociation constant and can displace carbonic acid from carbonates.

5.6.6 Malic acid

Occurring widely in nature, malic acid is closely associated with apples. It is the second major acid, after citric, found in citrus fruits and it is present in most berry fruits. Malic acid is slightly stronger than citric in perceived acidity, imparting a fuller, smoother fruity flavour.

Malic acid is a crystalline white solid (m.p. 100°C) and is highly soluble in water. Being less hygroscopic than citric acid it provides good storage and shelf-life properties. Unlike tartaric acid its calcium and magnesium salts are highly soluble and it therefore presents no problem in hard-water areas.

Malic acid finds use in a variety of products, mostly in fruit-flavoured carbonates. It is the preferred acidulant in low-calorie drinks and in cider and apple drinks, enhancing flavour and stabilising colour in carbonated and non-carbonated fruit-flavoured drinks. Malic acid may also be used to mask the off-taste of some sugar substitutes. Blends of malic and citric acids are said to exhibit better taste characteristics than either acidulant individually.

5.6.7 Fumaric acid

Fumaric acid is not permitted under UK or EU legislation for direct use in soft drinks, although it is permitted under Annex IV of Directive 95/2/EC (modified by directive 98/72/EC), with strict limits, in instant powders for fruit-, tea- or herbal-based drinks. Fumaric acid finds wide use in other countries as an acidulant, notably in the US market, where it has GRAS ('Generally Recommended As Safe') status. Fumaric acid is currently manufactured in the United States via the acid-catalysed isomerisation of maleic acid. In terms of equivalent palate acidity it can be used at lower levels than citric acid and typical replacement is suggested at two parts fumaric acid per three parts citric acid in water, sugar water and carbonated sugar water.

The main drawback in the use of fumaric acid is its slow solubility rate in comparison with citric acid, and special methods need to be employed in its dissolution. It has been claimed that fumaric acid and its salts have a tendency to stabilise the suspended matter in both flash-pasteurised and frozen fruit concentrates (McColloch & Gentile, 1958).

5.6.8 Ascorbic acid

Ascorbic acid, known more familiarly as vitamin C, is used not only as on acidulant but also as a stabiliser within the soft drinks system, and its antioxidant properties serve to improve the shelf-life stability of flavour components. Many of the ingredients used in flavourings are susceptible to oxidation, particularly aldehydes, ketones and keto-esters. Ascorbic acid shields these from attack by being preferentially oxidised and lost, leaving the flavour component unaffected.

It should be noted that although ascorbic acid acts well as a browning inhibitor in unprocessed fruit juices, its effect can be destroyed should the juice be subsequently pasteurised or heat treated. In such cases ascorbic acid can initiate its own chemical browning reaction.

Another disadvantage of ascorbic acid is its effect on some colours in the presence of light. In the case of azo-colours, such as carmoisine, a light-catalysed reaction occurs, resulting in cleavage of the –N=N– linkage and consequent destruction of the chromophore. This accounts for the disappearance of colour and bleaching of the characteristic hue associated with some soft drinks.

5.7 Flavourings

It is the flavour of a drink that provides not only a generic identity but also its unique character. This part of the sensory profile is responsible for pleasing and attracting the consumer. For example, having decided on a cola drink, the consumer will be able to differentiate between colas by virtue of the background flavouring components, which collectively provide a reference point to which the consumer can return, consciously or not, on future occasions whenever a particular brand of drink is selected.

A flavouring consists of a mixture of aromatic substances carefully balanced to convey the right message to the sensory receptors of the consumer. The preparation of such a mixture is a serious matter; the soft drinks flavourist, like the perfumer, must be well versed in the technique, be creative and be able to translate ideas into a practical solution.

While it is often difficult for the consumer to communicate descriptions of what is being tasted, the professional flavourist has no such inhibitions and is seldom at a loss when describing organoleptic attributes, as a personal library of stored knowledge relating to flavouring substances and types can be called on. For example, some descriptors that might be applied to a peach-flavoured drink are

sweet	juicy	fruity	lactonic	astringent
acidic	skinny	floral	estery	aldehydic
ripe	fresh	stewed	jammy	perfumed

Depending on the desired profile, the flavourist may add to, or subtract from, a central theme until an acceptable blend is reached.

Although the art of the flavourist depends largely on individual sensory abilities, it is frequently necessary in present-day flavour work to enlist analytical aid at an early stage of the project. Modern instrumental analytical techniques are capable of detection to extremely low limits, but it is still usually necessary to prepare an extract, or concentrated version, of the target flavour before carrying out the analysis. This may be achieved by solvent extraction, distillation, adsorption chromatography, dialysis, headspace concentration and cryogenic or adsorbent trapping, among other methods.

A good gas chromatographic/mass spectrophotometric (GC/MS) system can be used to identify the profiles of compounds and individual flavouring substances up to, say, 98% of the target flavour, thus bypassing much of the time-consuming preliminary work associated with organoleptic flavour matching.

GC/MS can provide an extremely rapid and reliable assessment leading to a tolerable flavour match requiring only slight 'tuning' adjustments for completion of the work.

In the creation of a flavouring there is inevitably a level of comparison with what is already accepted as the generic base. Thus, a strawberry flavour is at a certain level typical, to a marked degree, of the fruit itself. However, on a commercial level the characterisation of this base flavour into something new will set it apart from the competition and lead to success in the market. Descriptors such as 'fresh', 'cooked', 'jammy', 'green', 'wild', 'ripe', 'full-bodied', 'creamy', 'estery', 'sweet', 'artificial', 'natural' and many others may be applied in the assessment as the taster searches for an adjective to best describe what is being conveyed via neurological pathways from taste sensors to the brain. At best, the subjective nature of such an assessment will move into a common acceptable pathway across a wide number of tasters. It is at this point that the flavour may be identified as a winner, although even at this stage success will depend upon the type of application, marketing strategies and so on.

From the onset of the GC era there has been remarkable progress in the identification of volatile flavouring constituents occurring naturally in fruits, botanicals and so on, and in food and drink products. From a list of around 500 in 1955, the total now stands at around 7,000. The flavourist has many ingredients available to choose from, although in practical terms there is little need to select outside a group of 2,000 when creating that extra modification that will distinguish a new flavouring. The majority of flavourings on the market are derived from fewer than 800 or so of these ingredients. A commercial flavouring will typically contain between 15 and 60 components, although among these there may be natural extracts carrying a composite blend of constituents in their own right.

5.7.1 Flavourings and legislation

Food ingredients have, in most instances, been well investigated in terms of use and effect. They have been categorised and registered under permitted lists as appropriate to local legislation. Flavourings, however, by virtue of their complexity have always existed as a separate group. A distinction is made under various systems as to whether they are of natural origin or not. This follows adoption by the FAO/WHO Food Standard Programme and the Codex Alimentarius Commission of a proposal put forward around 1975 by the International Organisation of the Flavour Industry (IOFI) to divide flavouring substances into three categories.

Natural flavours and natural flavouring substances. These are preparations and single substances, acceptable for human consumption, obtained exclusively by physical processes from vegetable or sometimes animal raw materials either in their natural state or processed for human consumption.

Nature-identical flavouring substances. These are substances chemically isolated from aromatic raw materials or obtained synthetically. They are chemically identical to substances present in natural products intended for human consumption, whether processed or not.

Artificial flavouring substances. For the purpose of the Codex Alimentarius, these are the substances which have not yet been identified in natural products intended for human consumption, whether processed or not. They are hence made available only through synthesis.

Perhaps the most frequently cited register of flavouring substances appears in the FEMA GRAS listings. These listings were compiled by the Flavor and Extract Manufacturers' Association of the United States. They detail those substances 'Generally Recommended As Safe' when used in the minimum quantities required to produce the intended physical (i.e. sensory) effect and in accordance with the principles of good manufacturing practice. Each substance is allocated a FEMA number to enable cross-referencing with other listings, for example, those of the Council of Europe (CoE), the US Food and Drug Administration (FDA) and the Chemical Abstracts Service (CAS).

In the European Union, flavourings have generally been regarded as compound ingredients and concern has been voiced about the safety of their 'undeclared' components. Following extended interaction with representatives of the European flavour industry, trade associations, and other bodies, a new list has been drawn up of all chemical substances currently in use for flavourings in the EU member countries. This was published in the *Official Journal of the EC* on 27 March 1999. The register lists some 3,000 flavouring substances used in or on foodstuffs. Further work is now to be carried out in assessing the level of health risk associated with these ingredients with a view to limitation of their use, as necessary.

5.7.2 Flavourings in application

Flavourings for soft drinks are of two main types: water-miscible and water-dispersible. Water-miscible flavourings are formulated to dissolve easily in water, forming a clear bright solution at dosages usually in the region of 0.1%. They typically contain mainly oxygenated, highly polar compounds. Water-dispersible flavourings are strictly speaking 'insoluble', having in their makeup a relatively non-polar oil phase, usually citrus, which conveys the characteristic zest-like contribution from the peel. This type of flavour is introduced in the form of an emulsion, enabling oil-based flavouring substances to be incorporated in a soluble form.

5.7.3 Water-miscible flavourings

5.7.3.1 Flavouring mixtures

Relatively simple mixtures of flavouring substances, carefully dispensed, are dispersed/dissolved in a suitable carrier solvent system, for example, ethyl alcohol or propylene glycol. Such mixtures will tend to exhibit variation in sensory profile following the immediate blending operation, and a short period of maturation is necessary to allow the flavouring to 'settle' as the components may interact before stabilising. These reactions reflect the reactive nature of the oxygenates responsible for the perception of flavour. Aldehydes, alcohols, esters, ketones, lactones, phenols, terpene derivatives, and so on may interact to form a sort of equilibrium mixture containing acetals, ketals and additional ester types from interactions between some alcohols and acids present in the original mix and from transesterifications and so on (Table 5.4). Once settled, the new flavouring will remain in a relatively stable condition throughout its shelf life under recommended storage conditions.

Table 5.4 Example of a water-miscible flavouring

Peach flavouring	
Vanillin	50.000
SVR (ethyl alcohol 95%)	95.000
Cinnamic aldehyde	0.225
Terpinyl acetate	0.720
Methyl anthranilate	0.720
Linalyl acetate	0.940
Benzaldehyde	1.200
Oil of neroli bigarade	1.440
Geraniol	2.820
Oil of petitgrain, Terpeneless	3.925
Oil of petitgrain	4.000
Amyl butyrate	4.800
Amyl acetate	4.800
Amyl valerate	9.000
Amyl formate	10.800
Ethyl hexanoate	10.800
Ethyl valerate	26.400
Aldehyde C14	75.000
Propylene glycol (solvent)	600.000
Water (solvent)	100.000

Prepare mixture (in order shown), mixing well at each stage
Leave to stand for 24 h. Separate from an upper oil phase
Filter to produce a clear, bright product

5.7.3.2 Flavouring essence

A flavouring essence is a traditional flavouring product prepared by washing a selected oil blend (predominately citrus oils) with an aqueous alcoholic solvent mixture (e.g. 60% ethanol/40% water). It is an extraction process in which the aqueous extract phase becomes the flavouring. The process is carried out under cool temperatures, for example, 5–10°C, either batch-wise or by counter-current extraction. The soluble oxygenated flavouring constituents present in the essential oil blend (e.g. citral in lemon oil) are effectively partitioned between the two phases of the mixture. The low temperatures employed ensure that the transfer of any oil into the hydro-alcoholic phase is minimised as a poorly processed essence will tend to cloud when used in the drink formulation.

Initial mixing can be vigorous and thorough to establish an homogenous state that on settling and separating out will again form two layers, the lower of which comprises the essence. The separation stage is critical in the traditional 'mix and settle' batch method and may take several days to achieve.

Counter-current processes involving, for example, the use of spinning disc or pulse column techniques do not require the same degree of premixing and achieve separation in a shorter period of time. However, as a continuous process it becomes essential to correctly size the plant required in order to maintain an adequate stock. Unlike the batch process, counter-current techniques are not always easily able to accommodate a sudden escalation in product order requirements.

5.7.4 Water-dispersible flavourings

The important water-dispersible type of flavouring is more commonly referred to as an emulsion and is designed to introduce oil-soluble flavouring substances into a beverage system. Emulsions can have the dual role of providing flavour and cloud effects and are produced by the mechanical dispersion of an oil phase, carrying the flavouring components, into an aqueous phase containing selected hydrocolloid materials.

A typical sequence used in emulsion manufacture is shown in Figure 5.3. In order to achieve optimum performance of the product, great care is required at the mechanical stage of emulsion manufacture, and the uniformity and size of droplets in the dispersed oil phase are critical. If the emulsion is to be used in producing a stable cloud in the beverage, and to maximise optical density, then the ideal target is a droplet of about 1–2 μm in diameter. Above these limits, and if the particle distribution curve is not strictly Gaussian, there will be instability due to coalescence of the oil droplets and effects such as clearing-up, clearing-down, ringing and creaming will be observed, sometimes within a few days and certainly on storage. Providing particle, or droplet, size remains uniform, stable emulsions can be produced with a particle size average well below 1 μM (say,

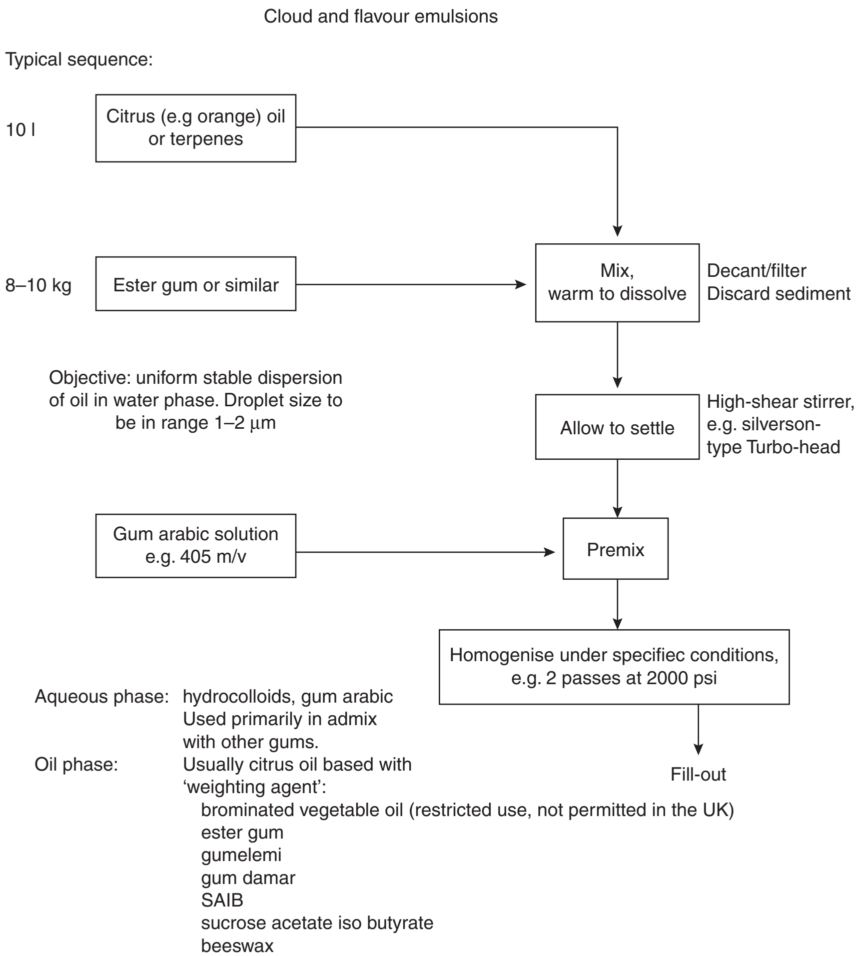

Figure 5.3 Cloud and flavour emulsions.

between 0.3 and 0.5 µM), but there will be a reduced cloud effect (see Figure 5.4). This is not a problem where the dispersion is intended primarily for flavouring purposes.

Although mechanical parameters are important, this is not the only area of control. The selection of the correct mix of stabiliser components is also critical. Beverage emulsions are essentially different from food emulsions. Their application in a mobile, liquid phase at concentrations in the region of 0.1% results in the formation of a uniform dispersion of the component droplets, and in order to remain stable and to avoid the effects already mentioned, these droplets must remain discrete from each other and also not interact with other

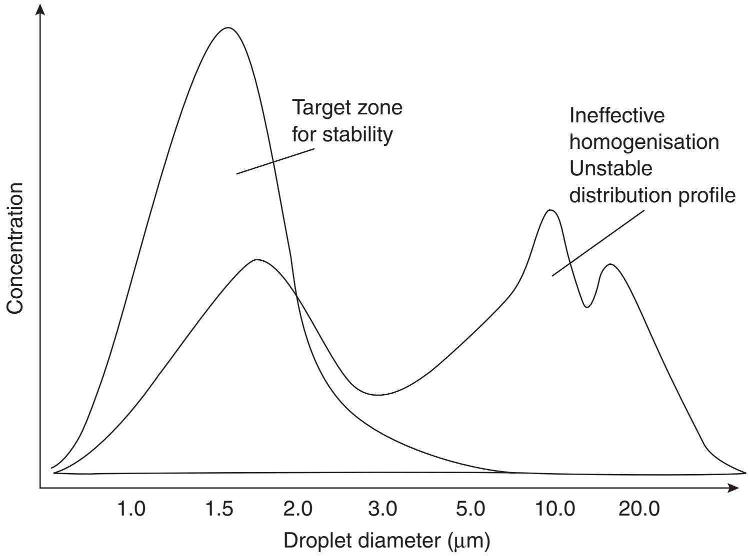

Figure 5.4 Particle distribution in emulsions.

components of the beverage formulation. For example, should another emulsion be present (say, a β-carotene colour emulsion) then because of the likely differences in particle size there will be a tendency for attraction between the two types, with resulting instability.

5.7.4.1 Oil phase

Oil-phase components comprise the base oil (usually citrus as orange/lemon oils or terpenes from the same source), in which is dissolved a suitable cloudifying/stabilising agent such as ester gum or the glyceryl ester of wood rosin (E445), gum damar, gum elemi, sucrose acetate isobutyrate (SAIB) (E444) or beeswax (propolis) (E901). Legislation governing the use of cloudifying agents varies thoughout the world, with the most regulated areas being the United States and the European Union. Currently, ester gum and SAIB are permitted for use in non-alcoholic cloudy drinks under European law, whereas beeswax is limited to use as a glazing agent for confectionery or in certain colouring materials. Ester gum is permitted in the United States whereas SAIB is not. The gum exudates, damar and elemi, are effective cloudifiers but their use in various countries is subject to local interpretation and practice. Of these, gum damar is of more interest since it is the only truly naturally sourced cloudifying/stabilising agent of any consequence. Propolis is also used successfully in certain applications, although like damar it is not officially recognised in the European

Union or United States. Ester gum is produced by the action of glycerol upon pinewood rosin to produce its esterified form, a mixture of di- and tri-glycerides, then purified by a process of steam-stripping in order to de-aromatise it, providing an odourless, tasteless gum type of m.p. 80–90°C. SAIB is produced by controlled esterification of sucrose using acetic and isobutyric acid anhydrides and its composition is consequently dependent upon reaction conditions.

5.7.4.2 Water phase

The water phase is, of course, complementary to the oil phase in that its hydrocolloidal components tend to provide a protective buffer zone around each oil droplet.

One theory is that the polymeric molecules of the hydrocolloid are drawn to the oil–water interface by charge effects doubtless promoted by the oil-soluble cloudifier components referred to above. These micelles will exhibit a composite charge, repelling neighbouring micelles of similar charge, preventing coalescence and maintaining a stable system. Of the many water-soluble polymers that can act in this manner as emulsifying agents, the most regularly used are gum acacia (also referred to as gum arabic) and the modified starches.

Gum acacia has been employed for many years as an emulsifier in beverage emulsions. Unlike other vegetable gums (e.g. tragacanth and guar) it has the advantage that it can remain relatively mobile in aqueous solution at concentrations of 30% m/m and above. The gum is obtained as the exudation from selected species of acacia. Upwards of 500 species of acacia are to be found across Africa, Asia, Australia and Central America, but only the varieties grown in Africa are of commercial interest. Of these, *Acacia senegal* and *Acacia seyal* are the most important. It is *A.senegal* that is the accepted standard for beverages. The two species are grown widely in a broad band of countries across west Africa through to the Sudan and Ethiopia. The gum tears are hand-picked and sorted by local people and transferred to central agencies for grading and distribution on the world markets. Apart from appearance in its raw form and physical quality the main feature of difference is the optical rotation. *A. senegal* is *laevo*-rotatory, with a specific optical rotation of −25 to −35° whilst *A. seyal* is *dextro*-rotatory with a specific optical rotation of +30 to +45°. Gum acacia is a complex polysaccharide with protein groupings, and it is thought that the level of protein present is a major factor in its favourable performance as a beverage emulsifier. It has not been unusual for blends of the two types to be offered to the beverage industry as gum arabic, arranged so that the optical rotation would be always *laevo*-rotatory but falling short of the optimum required purity. The purity specification issued by JECFA (Joint FAO/WHO Expert Committee on Food Additives, 1995) refers to gum arabic as the ‘dried exudation from stems and branches of *Acacia Senegal* (L.)Willdenow or closely related species of *Acacia* (fam. Leguminosae)’. An earlier specification (JECFA, 1990) to the use of specific rotation and nitrogen content as purity criteria has been dropped, which facilitates the use of mixtures of different acacia gums under the description 'gum arabic'.

As with all natural products, the quality and harvest yields of gum arabic are influenced by climatic conditions. In addition, in the region of Sudan and Ethiopia the availability has been affected by political factors, and from about 1984, prices for the gum have shown major fluctuation and, at times, have been far higher than those of other polysaccharides. Because of this the starch industry has made great effort to develop replacements. Perhaps the most successful, to date, are the starch sodium octenylsuccinates, where native starches have been modified by substitution with 1-octasuccinic anhydride (OSA). The introduction of a hydrophobic grouping on to the starch polymer results in highly effective emulsion stabilising properties. The conditions during manufacture of the modified starches are controlled to give some variety in performance, but a key general attribute is the low viscosity of their aqueous solutions, which can enhance formation of uniform droplets during the homogenisation stage of emulsion manufacture. Typical concentrations of the OSA starches when used for beverage emulsions are around 10–12%. Starch octenylsuccinates have the E-number E1450 under European legislation and are generally permitted additives under Annex 1 of Directive 95/2/EC.

5.7.4.3 Brominated vegetable oil emulsions

This section has been included more for its academic interest than from a practical point of view, as the use of brominated oils has been severely restricted by its absence from European legislation over the past 20 years, although until comparatively recently it was in use in India and parts of Asia. The term 'weighted' is frequently used when describing the oil phase. This alludes to the early use of brominated vegetable oil (BVO) to adjust the density of the oil phase in order to stabilise the dispersion when in ready-to-drink (RTD) form. BVO emulsions are no longer permitted in Europe, but they are still in use in some parts of the world, particularly in tropical areas, where the improved stability can greatly enhance the ability to store bottled drinks at high ambient temperatures.

The reaction of bromine with vegetable oils, such as peanut or maize oil, involves the addition of bromine atoms at the unsaturated double-bond sites of the oil to produce a progressive increase in the molecular weight and hence the density, which can in this way be easily adjusted to a predetermined value. Densities in the region of 1.24–1.4, according to the amount of bromine used, are usually achieved, and these are controlled to meet requirements. For example, the density of an oil phase comprising 'folded' orange oil can be adjusted from 0.875 up to, say, 1.03 by the addition of brominated oil, placing it in the density region of the target beverage and hence ensuring a stable system. The

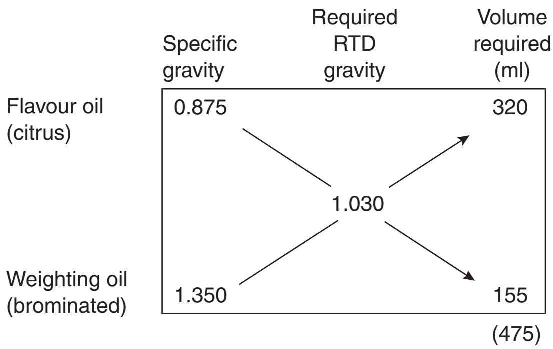

Figure 5.5 Example of the use of weighting oil.

amount of weighting oil required can be calculated by the following method devised by Pearson:

(1) Write the target gravity of the finished beverage in the centre of a square.
(2) Enter the specific gravities of the essential oil and weighting oil at the upper and lower left-hand corners, respectively.
(3) Diagonally subtract the larger from the smaller numbers in each case and place the resulting difference (omitting the decimal place) in the appropriate right-hand corner, giving the volume ratio required (see Figure 5.5).

Thus, 475 parts by volume of the oil mix will comprise 320 parts citrus oil plus 155 parts weighting oil, giving a gravity of 1.030, equal to that of the finished beverage.

5.8 Colours

Those of us fortunate enough to possess optical powers capable of distinguishing a variety of colours will appreciate the influence that this particular sensory dimension exercises on our judgement of matters important to our well-being, such as food and drink. The perception of colour influences a taster's reception of a drink, and as a consequence there is inevitably some controversy over the use of colourings in food and drink.

One point of view states that colours, which possess no measurable nutritional value, can have no role in food or drink other than that of deceiving the consumer. To a certain extent this is true, but to appreciate the full value of colour as a food additive, or more specifically as a soft drink additive, it is necessary to appreciate the synergy between the sensory responses of sight and taste.

Colour provides a means of presenting a beverage to the consumer so that the perceived organoleptic attributes are correctly placed in an ordered sequence of appreciation. Both quality and quantity of colour are of importance, and certain colours will provoke, or perhaps complement, a particular taste. Reds will favour the fruitiness of soft drinks, for example, blackcurrant, raspberry and strawberry. Orange and yellow tend towards the citrus flavours. Greens and blues reflect the character of peppermints, spearmint and cool flavours, sometimes herb-like and balsamic flavours. Browns align with the heavier flavours, for example, colas, shandies, dandelion and burdock. Therefore, the deceit, if ever intended, is aimed at ensuring that consumers are able to maximise their enjoyment of the beverage.

When a soft drink is based in part on fruit juices it may be necessary to restore the appearance of the juice concerned if its natural colours have been destroyed by heat processing, or to intensify these colours when the contribution from the juice is weaker than that normally associated with the effect the compounded drink is intended to convey. Colour adjustment may also be necessary to ensure uniformity of product and to offset natural variations in colour tone and intensity associated with the juice type employed in the beverage formulation.

Above all, colour is a major parameter in the assessment of quality, serving at the time of production to standardise the product. Colour deterioration can provide useful information on any quality changes during storage due to temperature fluctuations or microbial spoilage effects, for example.

The use of food colours is carefully controlled under various legislations (Table 5.5). There is at present no universal listing of colours for soft drinks and it is necessary to investigate the permitted list to ensure compliance for goods to be manufactured in, or exported to, a particular country.

Both the European Union and the FDA have published lists that are subject to regular review. The greatest concern has been expressed over the use of azo-dye colours as certain individuals can demonstrate an allergic reaction to some of these. Allergic reactions have been reported most frequently for sunset yellow (E110, FD&C yellow no. 6) and tartrazine (E102, FD&C yellow no. 5).

Although there are a number of food colours suitable for use in soft drinks, it should be appreciated that the contribution of any one of these cannot be entirely predictable. In any soft drink formulation, the colour component, as with all other ingredients, has to be carefully selected for its performance in the presence of certain acids, flavourings, antioxidants and even preservatives. It is essential, therefore, at all stages of development that meaningful storage trials are completed to ascertain the real contribution from colour in the newly finished beverage.

Table 5.5 Permitted food colourings derived from natural sources (EU Directive 94/36/EC)

Colour	Sources	Shade	E-no.	Stability	
				Light	Heat
Anthocyanins	Grape skins, elderberry, red cabbage, hibiscus	Red-purple-blue, pH dependent	E163	Good	Good
Water-soluble colours; natural indicators; red in acidic solutions (most stable) and bluer as pH increases					
Beetroot Red	Red beetroots (*Beta vulgari*)	Pink to red	E162	poor	poor
Water-soluble, limited stability when exposed to heat, light and oxidation; most stable between pH 3.5 and 5					
Carmine	Cochineal insect (*Dachtilopius coccus*)	Strawberry red, orange/red hues	E120 E160(a)	Excellent Fair	Excellent Good
Soluble in alkaline waters, solubility decreasing with lowering of pH, will precipitate below pH 3					
Annatto	Seeds of annatto shrub (*Bixa orellana*)	Orange	E160(b)	Fair	Good
Bixin is oil-soluble and frequently used for beverages in the form of its emulsion; norbixin is water-soluble in alkaline conditions and will precipitate in acid pHs; versions stabilised to about ph 3 are available					
Beta-carotene	Carrots, algae, palm, synthesised	Yellow to orange	E160(a)	Fair	Good
Oil-soluble; colour sensitive to oxidation; shade varies with concentration; water-dispersible versions available					
Paprika	Red pepper (*Capsicum annum*)	Orange to red	E160(c)	Fair	Good
Oil-soluble colour; sensitive to oxidation; water-dispersible versions available					
Lutein	Aztec marigold (*Tagetes erecta*)	Yellow	E161(b)	Good	Good
Oil-soluble egg-yellow colour; good stability; the xanthophyll lutein is a carotenoid colour which occurs in green leaves, vegetables, eggs and some flowers					
Curcumin	Turmeric (rhizomes of *Curcuma longa*)	Yellow	E100	Poor	Good
The purified colour, curcumin, is not water-soluble, but water-dispersible systems are available					
Chlorophylls	Green-leafed plants	Green	E140, E141	Poor, good	Poor, good
Natural chlorophyll (E140) is oil-soluble but water-dispersible forms are available; copper chlorophylls (E141), effectively chemically modified natural extracts, are water-soluble					

Table 5.6 Artificial (synthetic) colours permitted in soft drinks to a maximum level of 100 mg/l[a]

Colour	E-no.	Colour stability			Colour contribution
		Light	Heat	Acids	
Quinoline yellow	E104	Good	Good	V. good	Greenish yellow
Tartrazine (FD&C yellow no. 5)	E102	Good	Good	V. good	Lemon yellow
Sunset Yellow (FD&C yellow no. 6)	E110	Good	Good	V. good	Orange shade (similar to orange peel)
Carmoisine (azorubine)	E122	Good	Good	Good	Bluish red
Ponceau 4R	E124	Good	Good	Good	Bright red
Patent blue FCF	E131	Good	Good	Poor	Bright blue
Indigotine (FD&C blue no. 2)	E132	Fair	Poor	Fair	Dark bluish red
Brilliant blue FCF (FD&C blue no. 1)	E133	Good	Good	Good	Greenish blue
Green S	E142	Fair	Good	Good	Greenish blue

[a] With the proviso that individual levels of E110, E122 and E124 may not exceed 50 mg/l (EU Colour Directive).

Food colours are broadly divided into two classes: natural and artificial. In the United States, these are listed as either 'exempt from certification' or 'certified'. The natural colours are botanical extracts, with the exception of carmine (a red colour), which should perhaps be termed an entomological extract as it is obtained from the insect *Dactilopius coccus*, sometimes termed the cochineal beetle, which breeds and feeds on particular cacti indigenous to Central and South America. Table 5.6 lists artificial colours permitted in soft drinks under EU legislation.

5.9 Preservatives

A preservative can be defined as any substance that is capable of inhibiting, retarding or arresting the growth of micro-organisms or any deterioration of food due to micro-organisms or as masking the evidence of any such deterioration. In the European Union, defined maximum levels of permitted preservatives are given according to the food substrate concerned. For soft drinks that can be consumed without dilution, European Directive No. 95/2/EC applies (see Table 5.7). The *p*-hydroxybenzoates previously cited in the legislation are no longer permitted for use in soft drinks although they are still included under certain food uses.

As mentioned earlier, carbon dioxide, though not added specifically as a preservative, contributes to the inhibition of the growth of micro-organisms and, coupled with other factors (e.g. pH), contributes to the stability of a drink.

Table 5.7 Preservative limits under European Directive 95/2/EC

Preservative	Concentration (mg/l)	E-no.
Sulphur dioxide (carry-over from fruit concentrates only)	20	E220
Benzoic acid	150	E210
Sorbic acid	300	E200
Benzoic/sorbic acids in combination	150/250	E210/E200

Carbon dioxide is deemed to be effective at volumes over 2.5 or 3.0, and for this reason the incidence of spoilage in carbonated beverages is less than that in non-carbonated versions. ('Volumes' of CO_2 in general terms refers to the number of times the total volume of the gas, adjusted to 760 mmHg and 0°C, can be divided by the volume of liquid in which it is dissolved.)

Although preservatives can be used to good effect in beverage formulations they should never be considered infallible, and there is no substitute for stringent quality and hygiene controls at every stage of manufacture. Within their own product specification, raw materials should be assigned workable limits for microbial activity so that there is little chance of excessive contamination in the finished beverage product. Equally, all processing plant, machinery and containers likely to come into contact with the product during manufacture should undergo a thorough cleaning (sanitisation) before use.

Certain strains of yeast, moulds and bacteria can survive in relatively low pH conditions and some of these can exist and grow in the presence of certain preservatives; so it is important that everything is done to prevent their multiplying. Under favourable conditions, a typical rapidly growing yeast strain can double its numbers every 30 min, and at this rate in 12 h one yeast could become 16.7×10^6 cells, provided no inhibitory factor is present.

5.9.1 Micro-organisms and beverages

Although there is little evidence of the formation of toxic fermentation products in beverages, the problem of spoilage frequently arises. Because of their utilisation of sugars, yeasts are of most immediate concern.

Yeasts are classified with the fungi and are unicellular for most of their life-cycle. Together with moulds and bacteria they can bring about a deterioration in flavour, producing taints, off-notes, differences in mouthfeel and so on. Most yeasts can grow with or without oxygen, whereas most bacteria cannot survive in it. The majority of yeasts thrive in temperatures between 25 and 27°C; some can survive at temperatures over 70°C and others can exist, apparently quite comfortably, at 0–10°C. Bacteria exhibit some similar diversity in their characteristics, with an optimum growth temperature at around 37°C.

Soft drinks provide an ideal growth substrate for many micro-organisms, providing them with adequate supplies of the nutrients they require. Apart from water, the environmental necessity, typical requirements are sources of carbon (carbohydrates), nitrogen (amino acids), phosphorus (phosphates), potassium, calcium (mineral salts) and traces of other minerals, for example, sulphur, iron, cobalt and even vitamins. Because of its obvious link with protein formation during cell growth, the presence of combined nitrogen is of particular importance. Also, when it is introduced to beverages via fruit pulp or caramel (colouring), there will be a greater susceptibility to spoilage by certain micro-organisms.

Perhaps the most difficult aspect of dealing with microbial contamination in soft drinks relates to the delay factor: an apparently good-quality product may leave the bottling line for storage and distribution only to be returned at a later date, maybe after several weeks, when severe deterioration has taken place. Fortunately, such occurrences are seldom encountered in today's soft drinks industry, but to any manufacturer it is a nightmare scenario that must be avoided at all costs. A bottled drink constitutes a unique system, which can inhibit or enhance the growth of micro-organisms. Micro-flora, if present, will enter a dormant stage during which their chances of survival are related to their immediate surroundings. Following this lag stage, during which specific micro-flora may adapt to their new environment and start to grow, there is a burst of species-dependent activity, during which the population doubles repeatedly at a steady rate. Since a bottled drink is a closed system, waste products and diminishing nutrients will serve to slow down the growth and eventually bring it to a standstill, when the death rate increases and all activity stops. At this point the product, although perhaps not a health hazard, has been spoiled and can no longer serve its intended function.

5.9.2 Sulphur dioxide

Because of the ease with which it can be produced, gaseous SO_2 was one of the first chemical compounds manufactured and used by humans. By Roman times it was used as a preservative by burning sulphur before sealing wine into barrels or storage jars. It is one of the most versatile agents used in food preservation and is well known for its microbiocidal effect on bacteria, moulds and yeasts. Nowadays it is generally employed in the form of a sulphur-dioxide-generating salt. For example, sodium metabisulphite is converted thus in acid medium:

$$\underset{\text{(MW 190)}}{Na_2S_2O_5} + H_2O \rightarrow 2NaHSO_3$$

$$2NaHSO_3 + 2H^+ \rightarrow 2Na^+ + 2H_2O + \underset{\text{(MW } 2 \times 64)}{2SO_2}$$

Table 5.8 Preservatives and their salts

Preservative	E-no.	Alternative form used at equivalent level	E-no.
Benzoic acid (m.p. 122°C)	E210	Sodium benzoate	E211
C_6H_5COOH		Potassium benzoate	E212
Benzene carboxylic acid		Calcium benzoate	E213
Sorbic acid (m.p. 133°C)	E200	Sodium sorbate	E201
$CH_3CH{=}CH_2{-}CH_2{=}CHCOOH$		Potassium sorbate	E202
2,4-Hexadienoic acid		Calcium sorbate	E203
Sulphur dioxide (gas)	E220	Sodium sulphite	E221
SO_2		Sodium hydrogen sulphite, sodium bisulphite	E222
Sulphurous anhydride			
		Sodium metabisulphite	E223
		Potassium metabisulphite	E224
		Calcium sulphite	E226
		Calcium hydrogen sulphite, calcium bisulphite	E227
		Potassium bisulphite	E228

that is, 190 parts the metabisulphite produces 128 parts SO_2. Table 5.8 lists the various salts of the main preservatives.

The microbiocidal effect increases as the pH falls below 4.0, and because of this SO_2 is ideally suited for most soft drink formulations. However, its preserving action is impaired by a tendency to react with many of the fruit components of soft drinks to form organic sulphites, in which state the SO_2 is said to be ‘bound’. Although the preservative properties are due mainly to free SO_2, it is necessary to analyse for total SO_2 (i.e. free plus bound) as legislation for safe levels refers only to maximum total concentrations.

Although SO_2 is used to good effect in the preservation of concentrated citrus juices, with typical concentrations of 1,000–2,000 ppm m/v, it is now limited under EU legislation to no more than 20 ppm in non-alcoholic flavoured drinks containing fruit juice, and only as carry-over from concentrates (ref. 95/2/EC; see Table 5.7). There are a number of specific drink products which are permitted to contain higher levels of SO_2 under the same legislation – for example, concentrates/dilutables based on fruit juice and containing not less than 2.5% barley (barley water). However, since this limit has been much reduced from a previous level of 70 ppm, the onus has been squarely placed on manufacturers to attain improved manufacturing practices in terms of plant hygiene.

JECFA has recommended an ADI of not more than 0.7 mg/kg body weight for SO_2.

Disadvantages associated with sulphur dioxide are that some tasters can detect it as an unpleasant backnote or taint and it has a tendency to provoke allergic reactions in some individuals. Asthma sufferers tend to be affected by gaseous sulphur dioxide, small traces of which can promote an asthmatic attack. There is a risk with foods containing sulphites of gas liberation upon swallowing.

5.9.3 Benzoic acid and benzoates

Benzoic acid occurs naturally in some fruits and vegetables, notably in cranberries, where it occurs in amounts of the order of 0.08% m/m (Fellows & Esselen, 1955). It is also found in some resins, chiefly in gum benzoin (from *Styrax benzoia*), and in coal tar. Commercially available benzoic acid is produced by chemical synthesis.

Pure benzoic acid is a white powdery crystalline solid (m.p. 122°C) only sparingly soluble in water at normal temperatures. Because of this, it is added to the drink in the soluble form of its sodium or potassium salts. It is normal practice to disperse the benzoate completely during batch makeup before addition of the acid component, with its resulting pH reduction, to avoid localised precipitation of the 'free' benzoic acid due to its solubility having been exceeded (the solubility of benzoic acid = 0.35% m/v at 20°C). It is the free or undissociated form of benzoic acid that exhibits preservative action and hence its use is only effective when low pH values are encountered, ideally below pH 3, at which point the degree of dissociation reduces to below 10%.

Benzoic acid is generally considered to exhibit an inhibitory effect on microbial growth, although it is of little use for bacterial control, where the greatest problem will occur at pH values above 4, outside the effective limit mentioned above. Improved results are obtained when it is used in conjunction with other preservatives, for example, SO_2 or sorbic acid, due to synergistic effects. It is interesting to note that the current European Directive, which sets individual limits of 300 mg/l for sorbic acid and 150 mg/l for benzoic acid in non-alcoholic flavoured drinks, nevertheless permits a joint preservative use of up to 250 mg/l sorbic acid with 150 mg/l benzoic acid.

Allergic responses to benzoic acid have been reported, particularly among children known to be made hyperactive by other agents, for example, tartrazine. The maximum ADI for benzoic acid, recommended by JECFA, is 5 mg/kg body weight.

5.9.4 Sorbic acid and sorbates

Sorbic acid is found naturally in a number of fruits and vegetables, notably in the juice of unripe mountain ash berries (from *Sorbus aucuparia*), where it occurs together with malic acid. Sorbic acid and its salts are among the most widely used preservatives in the world. In soft drinks the most commonly used form is potassium sorbate because, like benzoic acid, there are problems in preparing its solution (the solubility of sorbic acid = 0.16% m/v at 20°C). In common with benzoic acid, as a microbial inhibitor, sorbic acid and its sorbates show reduced effectiveness with increased pH. Although activity is greatest at low pH values, sorbates have the advantage of being effective at pH values as high as 6.0–6.5, in contrast with benzoic acid, for which the comparative range is pH 4.0–4.5. The undissociated form, as with benzoic acid, is primarily responsible for its preservative action.

In the United States, sorbates are classified as GRAS and have maximum permissible levels in various foods of 0.05–0.3%. The European Union permits levels within the range 0.015–0.2%, although in soft drinks the limit is 0.03% (300 ppm m/v).

In addition to being less toxic than benzoates, sorbates seem to be less obtrusive in terms of taste detection by certain individuals and allergenic reactions. Overall, sorbates are considered to be one of the safest food preservatives in use, and WHO has set the ADI for sorbates at 25 mg/kg body weight.

5.10 Other functional ingredients

The functionality of the ingredients already discussed tends to be self-evident as contributing to the main identity of a drink. Except for the preservatives, these ingredients are used primarily for taste and colour. There are others whose contribution is to improve performance and further characterise the drink.

5.10.1 Stabilisers

We have already referred to the use of stabilisers in the production of water-dispersible flavourings and/or emulsion based cloudifying systems. Such additives, as well as contributing to stability, are also used, where appropriate, in soft drink formulations to impart stability to natural clouds, for example, dispersions of fruit solids, and to improve mouthfeel characteristics by increasing the viscosity of the drink.

In EU legislation there are over 50 E-numbered materials with stabilising properties for food use, although perhaps no more than 10 are used on a regular basis in soft drink formulations. These include the alginates, carrageens, vegetable gums, pectin, acacia, guar, tragacanth, xanthan and carboxy methyl cellulose. Also included under number E999 is extract of quillaia, which is permitted specifically for use in soft drinks and, apart from its use as an emulsifier, is valued for its heading properties.

5.10.2 Saponins

The saponins occur in the roots of many plants, notably the genus Saponaria, whose name derives from the Latin *sapo*, meaning soap, because of the lather-like reaction that occurs when parts of these plants are soaked in warm water.

This ability to generate foams finds use in beverages such as ginger beer, shandy, cream soda and cola formulations to improve and standardise heading foam characteristics.

Saponins for beverage use are sourced from quillaia bark (*Quillaia saponaria* Molina) and the yuccas. Of the latter species, two main varieties are used in the United States for production of the water extract: the Mohave yucca (*Yucca mohavensis*) and the Joshua tree (*Yucca brevifolia*). At the levels used these additives are colourless and tasteless; the dried extract, however, possesses an acrid, astringent taste. Permitted limits are quoted in terms of the dry weight of the extract. In the European Union, subject to Directive 95/2/EC, quillaia is permitted only in non-alcoholic drinks, to a maximum level of 200 mg/l.

5.10.3 Antioxidants

Perhaps the most common problem encountered during storage of a beverage relates to the oxidation effects involving certain ingredients. Both flavour and colour components can be subject to deterioration in the presence of dissolved oxygen, to the detriment of the product. Antioxidants are therefore included in those formulations containing ingredients most vulnerable to oxidation. Oxidation can frequently be attributed to the oxygen permeability of the plastic materials used in container manufacture, but it is essential that the oxidation process should not start at the production stage of the drink or any of its ingredients.

Citrus-flavoured drinks, notably lemon drinks, are frequently susceptible to oxidation and so antioxidants may feature in their formulation.

Oil-based, water-dispersible flavours (emulsions) are protected by the addition of oil-soluble antioxidants such as butylated hydroxy anisole (BHA) and butylated hydroxy toluene (BHT) to the oil phase before the emulsification process; 1,000 mg/l is the typical usage level in essential oils. Since the flavour emulsion will be used at the rate of about 0.1%, the level of antioxidant in the finished beverage will be of the order of 1 mg/l, which will safely comply with an ADI of 5 mg/kg body weight for either additive.

Increasing use is being made of natural and nature-identical antioxidants because in many countries use of BHA and BHT continues to be restricted on health grounds. Ascorbyl palmitate (6-*O*-palmitoyl-L-ascorbic acid) and its sodium and calcium salts, natural extracts rich in tocopherols (vacuum-distilled from soya-bean oil, wheat germ, rice germ and cottonseed oil, for example) and synthetic α-, γ- and δ-tocopherols are used to good effect in preventing oxidative deterioration in oil-based systems. In combination, ascorbyl palmitate and α-tocopherol (vitamin E) synergise to exhibit enhanced antioxidant properties.

5.10.4 Calcium disodium EDTA

This mixed salt of ethylene diamine tetraacetic acid (EDTA) is prepared by reacting the acid with a mixture of calcium and sodium hydroxides. It acts as a sequestrant, its binding action removing traces of metal ions present in raw materials or process water. These metals, for example, iron, can destabilise a beverage by a tendency to catalyse degradation of flavouring components, causing oxidation and off-notes. Their removal serves to maintain stability of the products during storage and to increase shelf life.

Under European Directive 95/2/EC, calcium disodium EDTA is permitted only in a limited number of foods, including some canned and bottled products, with maximum levels specified in each case. In the United States (Code of Federal Regulations) it is permitted to a level of 33 ppm in canned carbonated soft drinks, to promote flavour retention.

5.11 Investigation of a finished soft drink

After this review of many of the ingredients used in the preparation of a soft drink, it is now appropriate to look 'behind the label' of a product sold in the United Kingdom that contains at least some of these materials. This will demonstrate how the various parts complement each other.

The product to be considered is a notional low-calorie pineapple and grapefruit crush. Although the term no longer falls under any legal definition (the 1964 UK Soft Drinks Regulations required a minimum of 5% v/v of single-strength juice), a 'crush' is generally accepted to be a carbonated drink containing natural fruit juice. The label on this product might carry the description

Sparkling low-calorie pineapple and grapefruit crush

and its ingredients panel would be presented in one of the following ways, or possibly as a combination of both:

Ingredients: Carbonated water. Concentrated fruit juices (pineapple, grapefruit) (5% when reconstituted). Citric acid. Acidity regulator (sodium citrate). Artificial sweeteners (aspartame, saccharin). Flavourings. Preservative (sodium benzoate). Antioxidants (ascorbic acid, ascorbyl palmitate). Colour (lutein)

Ingredients: Carbonated water. Concentrated fruit juices (pineapple, grapefruit) (5% when reconstituted). Citric acid (E330). Acidity regulator (E331). Artificial sweeteners (aspartame, saccharin) (E951, E954). Flavourings. Preservative (E211). Antioxidants (E300, E304). Colour (E161b).

The ingredients are presented in descending order of concentration and listed under their respective categories, where they may be named or referred to by their E-number, if appropriate. Flavourings are, at present, listed as a category and there is no need to name their individual components, but this could change under future EU legislation.

In order to manufacture the above beverage, the bottler will require a recipe, such as:

Low-calorie pineapple and grapefruit crush made from eight-fold pineapple and grapefruit compound

Aspartame	137.0 g
Sodium saccharin	90.7 g
20% sodium benzoate solution	206.0 ml
8-fold pineapple and grapefruit compound	12.5 l
Lutein colour liquid	50.0 g
Water to make	100.0 l

This recipe produces 100 l of low-calorie pineapple and grapefruit bottling syrup intended for dilution in the ratio of 1 part syrup to 5.66 parts carbonated water (by volume). It would be recommended that this product be pasteurised before carbonation to three volumes CO_2.

If made from the above recipe, the syrup and beverage will conform to the following specification:

	Syrup	Beverage
Aspartame (mg/l)	1,372	206
Acid (% m/v as citric acid anhydrous)	3.53	0.53
Saccharin (mg/l)	693	104
Benzoic acid (mg/l)	600	90

The description of the compound/concentrate used in the recipe as eight-fold relates to the bottling syrup, not the finished drink:

Eight-fold pineapple and grapefruit compound

20% sodium benzoate solution	1.18 l
6–1 concentrated pineapple juice	40.00 l
6–1 concentrated grapefruit juice	4.44 l
Citric acid anhydrous	27.90 kg
Tri-sodium citrate	1.34 kg
Ascorbic acid	0.54 kg
Ascorbyl palmitate	0.16 kg
Pineapple flavouring	2.13 l
Grapefruit flavouring	4.26 l
Pineapple flavouring	0.27 l
Water to make	100 l

This compound exists as a vehicle to introduce the characterising components into the beverage: thirst-quenching properties from the acid, flavour and body from the juice (also contributing to mouthfeel). A major part of the identity of the final drink is provided by the flavouring ingredients. In order to stabilise and protect these, oil- and water-soluble antioxidants are included.

Table 5.9 Ingredient formulae for flavourings used in the notional pineapple and grapefruit crush (litres)

Flavourings		Active flavour components
Pineapple flavouring (2.13 l)		
Ingredients:		
pineapple esters 200-fold comprising approximately 90% water		0.21
Pineapple flavouring (0.27 l)		
Ingredients:		
Lemon oil T'less	1	
Sweet orange oil 12–1	2	
Ethyl caproate	10	
Isoamyl butyrate	14	
Allyl caproate	18	
Ethyl butyrate	59	
Ethyl propionate	53	
Glycerol	48	
Water	232	
Ethanol (SVR)	563	
	1,000	0.04
Grapefruit flavouring (4.26 l)		
Ingredients:		
Cedarwood oil (10% in IPA)	2	
12–1 concentrated grapefruit oil	4	
4–1 concentrated grape oil	5	
Lemon essence oil	8	
5–1 concentrated grapefruit	15	
Grape oil Israeli	20	
Grapefruit essence oil	48	
Isopropanol (food grade)	898	
	1,000	0.43
Total active flavouring components		0.68

The exact quantities of flavouring materials can be determined from the ingredient formulae in Table 5.9.

Thus, 100 l of the eight-fold pineapple and grapefruit compound contains no more than 0.68 l of flavouring materials. This is used in the bottling syrup at 12.5% v/v, which is diluted 1 part syrup to 5.66 parts carbonated water to provide the finished drink. The effective flavour dosage rate is given by the product of the two dilution factors, that is, $8 \times 6.66 = 53.28$ times, and the real concentration of flavouring materials at RTD beverage strength is therefore $0.68 \div 53.28 = 0.0128\%$ v/v or, as it is more frequently expressed in the flavouring industry, 128 ppm. This is quite a modest input for the level of flavour impact it is able to generate.

5.12 Food safety

Although the primary function of food for humans is survival, it now has the additional associations with health, enjoyment and acceptability. Today's consumer looks to suppliers and manufacturers for a product with which there is no associated risk in consumption and which is marketed in accordance with strict observance of the laws governing food safety.

Although microbial contamination must always be a point of concern for food, it becomes much less of a potential hazard for beverages where the lower pH conditions make the survival of pathogenic species virtually impossible and the likelihood of food poisoning equally remote. In addition, any attack from, say, yeasts and moulds will manifest itself as spoilage and be easily detectable either visually or organoleptically well before any lasting danger can be done. However, to be assured of complete safety, it is necessary to look further, into the actual ingredient makeup of the drink itself.

The safety of food additives and other ingredients is monitored according to guidelines issued by the joint committee of JECFA, WHO and the FAO. Knowledge of health safety is gained primarily as a result of animal feeding trials coupled with relevant short- or long-term toxicological investigations. In later stages of testing, humans may also be included in the studies to ascertain that their physiological reactions are similar to those found in animals.

Because of potentially longer-term ingestion, the standards applied to food additives in feed trials are significantly higher than those applied to pharmaceuticals, for example, where, in order to treat successfully some illness, certain side-effects caused by the treatment can be tolerated. The ADI is an estimate of the amount of a food additive, expressed in mg/kg body weight per day, that can be consumed safely over an entire lifetime. ADI levels are set by JECFA after considering the results of various feeding trials. As a general rule, the ADI value is set at one hundredth of the intake that produces virtually no toxicological effects in long-term animal feeding trials.

The registering of a 'new' ingredient, or perhaps the retention of an existing one, under the European Miscellaneous Additives Directive is a complicated and long-drawn-out procedure, not just because of the strict protocols employed in the assessment itself, but also because of the accompanying exchanges taking place between all interested parties representing the member states in setting up the application: agricultural and trade associations, consultancies, research establishments, and the like whose opinions may be called upon in the preparation of a suitable dossier laying out the justification for inclusion of an additive. The associated costs are formidable. Currently, starting from scratch, the costs involved in preparing a monograph on the constituent and physical properties of a proposed additive, together with undertaking a full toxicological study, will be of the order of £230,000 ($425,000). Thus it is little wonder that new additions to the list are few in number.

An ongoing issue periodically provoking media coverage is that of the intolerance of members of the population to certain food ingredients. This is of most concern when serious allergenic reactions occur. The apparent growing prevalence of severe allergic reactions to peanuts during the 1990s is a case in point; through the manufacturing industries, it prompted a series of controls on the raw material supply line to ensure products are, where appropriate, free from nut components and labelled accordingly. In 1996, the European Scientific Committee for Food (SCF) issued a report on adverse reactions to food and food ingredients. A level of intolerance to food additives of 0.026% was given in the report, which is equivalent to about 3 people in 10,000 of the population being affected.

5.13 Future trends

With a current world population of over six billion, the challenge facing the soft drinks industry remains largely unchanged in essence, but it is perhaps increasingly more complex. To provide products that are wholesome, durable and acceptable in character and of interest to an ever-growing market has been the objective for many years, but with the increasing realisation that almost anything we consume can have detrimental as well as beneficial effects, the focus today is very much on additives, processing aids and functional ingredients.

The term ‘functional drinks’ is now part of the vocabulary, and this is an area where there will be ample scope for future development. The general aim of a functional drink is not just to alleviate thirst, but also to contribute to a sense of well-being in the consumer by assisting with diet, metabolism, improved lifestyle and so forth. Healthy lifestyle drinks, drinks with added vitamins, drinks with probiotic properties and isotonic drinks have already entered the market and provide a base for more innovation. Such claims for drinks as being rich in fibre, fat-free, sugar-reduced, beneficial in calcium adsorption by the body, improving intestinal flora, probiotic, with ascorbic acid and energy reduced will no doubt continue to be seen and will have to be supported with sound evidence in order to comply with labelling regulations.

During the past decade, legislative controls have become more stringent, with the gradual phasing out or reduction in use of many of the additives that have hitherto been considered an essential part of the production formula, and this process is bound to continue. In conjunction with this, improved packaging, aseptic processing techniques and the selection of top-quality raw material ingredients will be in vogue, creating a highly specialised system of presentation designed at all times to satisfy the consumer – for this is what the successful marketing of soft drinks depends upon.

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6 Non-carbonated beverages

P.R. Ashurst

6.1 Introduction

Non-carbonated beverages represent an important segment of the market for soft drinks but they present some special technological issues for product developer and manufacturer alike. The principal groups of non-carbonated beverages are as follows:

- dilutable drinks
- ready-to-drink pre-packaged beverages
- fruit juices and nectars.

As indicated elsewhere in this volume, the technical issues relating to tea, coffee and milk-based drinks are not considered except insofar as they relate to the marketplace or where they are used in soft drinks as ingredients.

Soft drinks are low-pH beverages that are based mostly around fruit-derived ingredients or incorporate fruit flavours. They are an important source of hydration but are usually selected on the basis of pleasant taste and convenience of use. There are some soft drinks, of which cola-flavoured beverages are the most prominent, which do not rely primarily on fruit flavours. Some of these non-fruit-flavoured products are almost invariably produced only in a carbonated form, whereas others, such as peppermint, almost always appear only in dilutable and non-carbonated form.

The particular issues that must be addressed when non-carbonated beverages are to be produced relate mainly to the prevention of microbial spoilage and the deterioration of product taste and appearance as a result of oxidation, and to enhancement of flavour. Carbonated beverages use the presence of carbonation to boost flavour characteristics and provide palate stimulation.

Thus there are different considerations to be applied to formulation and packaging when non-carbonated beverages are produced.

6.2 Dilutable beverages

6.2.1 Overview

Dilutable beverages have been widely used for many years as a low-cost, convenient means of producing soft drinks on the consumers' premises.

The product formulation is often broadly similar to that for a syrup produced by a carbonated beverage manufacturer who subsequently dilutes this intermediate with carbonated water before packing into the containers sold to consumers.

Dilutable products, on the other hand, offer the consumer a number of advantages, including the ability to use differing syrup : water ratios, the ability to produce variable volumes of end-product and the possibility of using different diluents such as water, alcoholic drinks or milk.

Although syrups in various forms have been around for many years, a particular milestone was the production and use of concentrated orange juice as a dilutable 'syrup' in the United Kingdom during the Second World War.

Some readers may recall that the product was a 60°Brix concentrated orange juice packed in 8 fluid ounce (200 ml) 'medical flats' – glass bottles with flat sides and rolled-on metal caps with cardboard inserts. The product was produced for and distributed by the UK government as a means of enhancing the nutritional intake of babies and young children, particularly in respect of their vitamin C needs.

Although this product was available following the war years, it spawned the development of other products such as whole fruit drinks, 'squashes' and cordials, which became and remain the mainstay of the United Kingdom and Commonwealth dilutables market.

6.2.2 Nomenclature

Dilutable products were given the particular product designations used above, and to ensure consistency between manufacturers, the United Kingdom introduced legislation in 1964 which defined, for the first time, specific compositional requirements for these products. These regulations, known as the 1964 Soft Drinks Regulations (Anon, 1964), were revoked some 31 years later when it was recognised that many factors rendered these compositional constraints unnecessary. In particular, this move coincided with a change of emphasis in legislation towards improved labelling and consumer choice. For example, the imposition of regulations in the United Kingdom requiring percentages of fruit ingredients to be declared (Anon, 1998) was one of a number of factors that facilitated such a change of emphasis.

An outline of the principal fruit component compositional requirements of the UK 1964 Soft Drinks Regulations compared with those of today is shown in Table 6.1.

In addition to reserved descriptions for these products, the 1964 regulations also defined minimum levels for carbohydrates and imposed various compositional constraints. It is perhaps noteworthy that at the time of writing, the only compositional constraint that is legally imposed in the United Kingdom is that for a quinine content (milligrams/litre) to enable a product to be described as 'Indian Tonic Water'.

Table 6.1 1964 UK reserved descriptions for dilutable fruit drinks

Product description	Juice/fruit minimum content 1964	Juice/fruit minimum content post-1995
Squash	25% v/v cloudy juice	No minimum
Cordial	25% v/v clear juice	No minimum
Whole fruit drink	10% w/v comminuted citrus fruit	No minimum
X flavour drink or cordial	No minimum	No minimum
Citrus barley water	15% citrus juice	No minimum

Despite the removal of compositional legislation, the descriptions of dilutable soft drinks in the United Kingdom are still widely used today. Consumers and enforcement authorities alike still have an expectation that a product described as a 'squash' will be a cloudy product containing a significant proportion of fruit juice. Similar expectations still apply to the other products mentioned above.

6.2.3 Ingredients

Other chapters of this volume deal in more detail with the ingredients of all soft drinks, and readers requiring more information should refer to them. However, it is appropriate here to make reference to special issues concerning ingredients insofar as they relate to dilutable soft drinks. The main ingredients of dilutable soft drinks are set out in Table 6.2.

6.2.3.1 Fruit components

6.2.3.1.1 Concentrated juices. It will be evident from the section on nomenclature that the principal fruit components that are used in dilutable soft drinks are fruit juices (both clear and cloudy) and whole fruit preparations – the so-called comminutes.

Fruit juices and comminutes that are added to dilutables (and other non-carbonated drinks) may be either freshly pressed or in the form of a concentrated juice. It is self-evident that if a significant proportion of juice (25%, for example) is required in a dilutable drink the addition may be difficult unless a concentrated juice is used. In practice, most non-carbonated beverages use concentrated juices and comminutes to obtain the required level of fruit components.

The concentration of most fruit juices is conveniently measured in degrees Brix, although the strict interpretation of this measure refers to pure solutions of sucrose in water (e.g. 10°Brix is 10% w/w sucrose in water). For juices with a high proportion of sugars to acids, such as orange, pineapple and apple, this is

Table 6.2 Principal ingredients of dilutable soft drinks

Nutritional	Non-nutritional
Fruit components	Preservatives
Carbohydrate syrups	Colours
Acidulants	Emulsifiers and stabilisers
Other nutritional components, e.g. vitamins and minerals	Antioxidants
	Acidity regulators
	Intense sweeteners
	Flavourings
	Clouding agents

a useful and convenient means of measuring concentration. In some instances a correction factor may be introduced to take account of the acidity (see Chapter 10). Brix measurement is simply related to refractive index and there is a slightly different relationship between the refractive index and concentration of citric or malic acids and that of simple sugars.

The observed Brix (and acidity) of a given freshly pressed juice will vary over a limited range depending on a number of plant variables such as seasonality, variety and location. However, concentrated juices are produced to an industry standard and so there will be slight variations in the degree of concentration required to achieve the standard of concentrated juice.

For example, frozen concentrated orange juice (FCOJ) – the industry standard material for orange – is traded as 65–66°Brix concentration. Oranges that are used may, on pressing, yield a juice of variable Brix – say, from around 10° to as much as 14 or 15°. Thus, the degree of concentration required to produce 65–66°Brix concentrate will be slightly different for a 10°Brix juice compared with a 13°Brix juice.

Most countries therefore adopt a 'standard' Brix for juices to facilitate the production of a comparable product when juices are reconstituted. In most European countries this standard is 11.2°Brix for orange juice. A similar approach will be adopted when calculating the amount of a concentrated juice required to deliver, say, 25% juice in a dilutable drink.

The typical concentrated juices and comminutes used by the industry for manufacturing dilutable drinks are shown in Table 6.3.

6.2.3.1.2 Comminutes. The process of comminution refers only to citrus products, where the oils that reside in the flavedo (coloured peel) have intense flavour characteristics. At its simplest, comminution involves taking a complete orange (or other citrus fruit) and making a pulp from it. This pulp will have a much more intense flavour than juice alone but because of the presence of much peel and albedo (pith) it would be unacceptable in taste to most consumers. Thus, the process of comminute production, developed in the immediate post-war years, is typically as set out in Figure 6.1.

Table 6.3 Typical concentrated juices and comminutes for manufacturing dilutable drinks

Fruit	Typical concentrate	Comminute
Orange	Frozen concentrated juices 65–66°Brix	30–60°Brix (3 : 1–6 : 1)
Lemon	Frozen juice or sulphite-preserved 4 : 1 or 5 : 1 clear or cloudy product	25–40°Brix (4 : 1–6 : 1)
Lime	450 g per litre citric acid = 6:1–3:1	Not normally produced
Apple	69–70°Brix concentrate	Not available

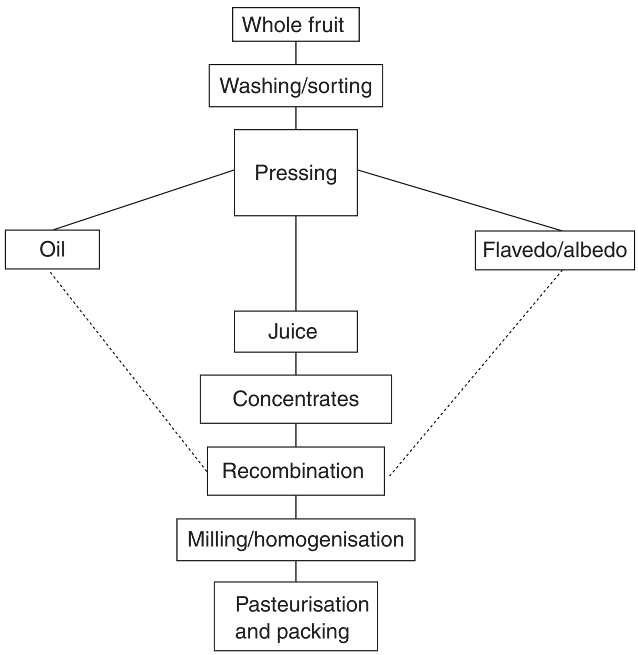

Figure 6.1 Simplified outline process for citrus comminute production.

The components of the citrus fruit are separated into the principal products: citrus oils, juice and residual peel and pith. After concentration of the juice, the oils and some peel and pith will be recombined with the concentrated juice. The whole mixture will then typically be finely milled and homogenised before being pasteurised.

Finished comminutes are often available at between about 35 and 60°Brix. When used for making whole fruit drinks they deliver, apart from a more intense fresh flavour, cloud and colour.

6.2.3.1.3 Other fruit components. Other fruit components that may be used in the manufacture of non-carbonated beverages, particularly dilutables, include pectins and aroma substances obtained during the concentration of fruit juices. These components do not normally count towards the fruit content of products as they are usually classified as types of permitted additives.

6.2.3.2 Carbohydrates

Carbohydrates still feature as important components of many non-carbonated beverages, and they are particularly important in the manufacture of dilutable drinks. Historically, the UK Soft Drinks Regulations of 1964 required dilutable drinks to have a minimum level of 22.5% w/v carbohydrates unless they were declared to be 'low calorie'. The regulations assumed a five times dilution factor (1 part dilutable plus 4 parts water) and thus a minimum carbohydrates level of 4.5% w/v in finished drinks.

Today, manufacturers in most countries can choose how much carbohydrate (if any) to use, the information being passed to the consumer by the product label.

Sweeteners generally are dealt with in more detail in Chapter 4.

6.2.3.2.1 Sucrose. The preferred carbohydrate for most manufacturers is still sucrose, although its 2004 price within Europe is so artificially high that other alternatives are often sought and are increasingly used. Sucrose is readily available as a bulk dry solid or as a 67°Brix syrup and it is in this latter form that most manufacturers will use it.

6.2.3.2.2 Invert sugar. Invert sugar, sometimes referred to as partially inverted refiner's syrup, is produced by acid or enzymic hydrolysis of the disaccharide sucrose into its 2004 component parts of fructose and dextrose (glucose). Invert syrups usually contain a mixture of sucrose, fructose and dextrose. The main advantage of such a syrup is the reduced likelihood of crystallisation and an increase in osmolality, which may be useful in reducing spoilage risk.

Because of the development of fructose-containing glucose syrups, invert sugars are little used now. Some product formulators maintain that the sweetness of invert syrup is marginally greater than that of sucrose at the same strength.

6.2.3.2.3 Glucose syrups. Glucose syrups are a group of industrial syrups manufactured from starch – usually corn starch (maize). The starch may be hydrolysed by either acid or enzymic hydrolysis or, more usually, a combination of the two. Glucose syrups are normally referred to as having a dextrose equivalent (DE), which broadly relates to the percentage of dextrose in the mixture of carbohydrate produced on hydrolysis. In general, the DE also gives an indication of the sweetness of the syrup. Typical glucose syrups that are commercially available include 42DE and 63DE syrups. Products available from the hydrolysis of starch include pure dextrose, glucose syrups with a range of carbohydrate components and maltodextrins.

Glucose syrups are often used in energy drinks, where a high level of carbohydrate is required (e.g. 20% at drinking strength) but without the sickly sweetness that this strength of sucrose would bring. There can also be commercial advantages in using glucose syrups as the solids levels are usually around 80% w/w compared with the maximum of 67% w/w for sucrose syrup. One particular technical disadvantage is that glucose syrups are often extremely viscous and if allowed to cool to below 30°C can become very difficult to handle. The solids levels in glucose syrups are measured in degrees Baumé rather than degrees Brix and some examples of the relationship between degrees Baumé, degrees Brix and physical characteristics are shown in Table 6.4.

6.2.3.2.4 Modified glucose syrups. An important development in the production of alternative carbohydrate sources for beverage and other food uses has been the production of fructose-containing glucose syrups. One such product is known as high-fructose glucose syrup (HFGS). It is widely used in

Table 6.4 Comparison of degrees Brix, degrees Baumé and physical characteristics of carbohydrate syrups

Degrees Brix	Degrees Baumé	Refractive index at 20°C	Specific gravity 20°/20°C
0	0	1.3330	1.0000
5	2.79	1.3403	1.0197
10	5.57	1.3478	1.0400
15	8.34	1.3557	1.0610
20	11.10	1.3638	1.0829
25	13.84	1.3723	1.1055
30	16.57	1.3811	1.1290
35	19.28	1.3902	1.1533
40	21.97	1.3997	1.1785
45	24.63	1.4096	1.2047
50	27.28	1.4200	1.2317
55	28.54	1.4307	1.2451
60	32.49	1.4418	1.2887
65	35.04	1.4532	1.3187
70	37.56	1.4651	1.3496
75	40.03	1.4774	1.3814
80	42.47	1.4901	1.4142

the United States and to a lesser extent in Europe, where the commercial advantages of use are less. In these products, starches (usually corn starch) are hydrolysed to dextrose syrup. A further enzymic modification then takes place whereby a proportion of the dextrose present is converted to fructose. Depending on the proportion converted, the resulting level of fructose can reach up to 100% or more of the dextrose level to give a product that is chemically similar to invert sugar syrup and has similar technical and organoleptic properties. Syrups with a lower proportion of dextrose converted to fructose have also been found to be a useful carbohydrate source for beverage manufacture. The approximate comparative sweetness values of various carbohydrate sweeteners are shown in Table 6.5.

6.2.3.2.5 Fructose syrup. In addition to the glucose/fructose syrups mentioned above, a fructose syrup has been produced using inulin as a source. Inulin is the fructose analogue of starch, and the chicory root is the standard source for commercial hydrolysis. Fructose syrups are usually too expensive for routine use in beverage production but they have been employed where a particular claim is to be made for fructose. They have also been used for the adulteration of fruit juices as they are chemically difficult to detect. Detection is possible at the sub-molecular level by techniques such as stable isotope ratio measurement. Fructose is also manufactured using sucrose as a starting material.

6.2.3.3 Intense sweeteners

More details about intense (or artificial) sweeteners appear in Chapter 4, but no chapter dealing with beverage manufacture would be complete without mentioning these important ingredients. A comparative picture is shown in Table 6.6.

6.2.3.3.1 Saccharin. Intense sweeteners have been widely used for many years. The oldest, saccharin, was used as a sugar substitute during the Second

Table 6.5 Comparison of carbohydrate sweeteners

Carbohydrate	Approx sweetness compared with sucrose	Typical form
Sucrose	1.00	67°Brix syrup or solid
Invert sugar	1.00–1.1	Syrup
Glucose syrup	0.4–0.8	Syrup
High-fructose glucose syrup	1.0–1.1	Syrup
Glucose/fructose syrups	0.8–0.9	Syrup
Fructose syrup	1.05–1.1	Syrup

Table 6.6 Comparison of intense sweeteners

Sweetener	E-Number	Typical sweetness factor compared with sucrose	Maximum UK permitted use (mg/l)
Sucralose	(955) provisional	450–500	300
Saccharin	954	450–550	80 (as imide)
Aspartame	951	160–200	600
Acesulfame K	950	160–200	350
Cyclamic acid	952	30–40	400
NeoHDC	959	200–300	30

World War and for many years was used together with sucrose as a mainstay of beverage sweeteners. Saccharin, by experiment, has a sweetness factor compared with sucrose of 450 for the soluble form (sodium saccharin dihydrate) and around 550 for the much less water-soluble imide form. Despite commercial advantages saccharin is little used now because of its bitter aftertaste.

6.2.3.3.2 Aspartame. Aspartame is a widely used intense sweetener that has excellent taste characteristics. It is a peptide made from two amino acids, phenylalanine and aspartic acid, but will in an acidic beverage medium slowly hydrolyse to its components. The fact that aspartame is a source of phenylalanine is of concern to consumers with certain complaints, and suitable label declarations are now required by law. Technically, this slow hydrolysis brings about loss of sweetness.

6.2.3.3.3 Acesulfame. Acesulfame K has similar taste characteristics to aspartame but without the disadvantages of hydrolysis causing loss of sweetness. The product has found wide use in beverages.

6.2.3.3.4 Cyclamic acid. Cyclamic acids in the form of cyclamate salts were in wide use in the 1965–75 period but because of a sudden scare that they could be a cause of certain cancers were removed from the marketplace. Cyclamates were re-permitted on a limited basis in Europe in around 1995 but have found little commercial use since then.

6.2.3.3.5 Neohesperidin dihydrochalcone. Neohesperidin dihydrochalcone (NeoHDC) is a sweetener of natural origin that has been chemically modified. It has found little use in the beverage or food industries.

6.2.3.3.6 Sucralose. Sucralose is the most recently permitted artificial sweetener. It is a chemically modified sugar but has a very high sweetness factor, comparable with that of saccharin, but without the unpleasant aftertaste.

The sweetness profile of sucralose is claimed to be excellent and it has already found some use in the beverage and food industries.

6.2.3.4 Other ingredients

6.2.3.4.1 Acidulants. The preferred acidulant for dilutable (and other) soft drinks is citric acid, which is readily available both as a crystalline solid (citric acid anhydrous) and as a 50% w/w solution in bulk. Other acidulants that are used in specific products include malic acid, lactic acid and tartaric acid. Phosphoric acid, until recently permitted only in cola drinks, is now available for use in the United Kingdom but has so far found little, if any, use in dilutable products. Acids other than citric are usually employed only where a slightly different taste profile is needed. Ascorbic acid is usually employed as an antioxidant rather than as a direct acidulant.

6.2.3.4.2 Preservatives. Despite the requirement that most dilutable drinks should be pasteurised (see Section 6.4), the use of chemical preservatives in these products is, in most situations, almost essential. The main reason for this is that dilutable products are used over a period of time during which the container will remain part full or ullaged. The storage period will vary from user to user and may be as short as a few hours from first opening to several weeks or even months. During this time the consumer expects a product to remain free from fermentation, mould growth or other microbial development and to retain an acceptable taste. Preservatives permitted in the United Kingdom include benzoic acid, sorbic acid and sulphur dioxide (in limited situations). Dimethyl dicarbonate (velcorin) is permitted but little used in dilutables.

Sulphur dioxide remains a key preservative in dilutables containing fruit components, where it is permitted (at least in the United Kingdom) at a rate of 250 mg/l. This preservative, which is a gas in solution in the product, will diffuse into the product headspace and help to minimize microbial development.

It is normal to use additionally a mixture of both benzoic and sorbic acids, added as their sodium and potassium salts respectively. Current UK preservative regulations permit a maximum level of 300 mg/l of sorbic acid and 150 mg/l of benzoic acid, both at drinking strength. For this reason it is normal to suggest on the product label a dilution ratio, which can then be used as a factor in calculating the amount of these preservatives to be used. An example is set out in Table 6.7.

In most dilutables, these levels would be more than adequate to deliver enough preservation after pasteurisation, and a typical preservative mix for a dilutable containing up to 25% fruit juice might be as follows:

Sulphur dioxide	150 ppm (parts per million)
Benzoic acid	500 ppm
Sorbic acid	800 ppm

Table 6.7 Preservative levels

Preservative	Max. ready-to-drink level mg/l	Dilution recommended for product	Max. level in dilutable
Benzoic acid (sodium benzoate addition)	150	1 + 4 (i.e. 5× dilution)	750
Sorbic acid	300	1 + 4 (i.e. 5× dilution)	1500

Table 6.8 Convenient conversions for preservative materials

Preservative	Molecular formula and weight	Added form	Molecular formula and weight	Conversion factors	
				Salt to preservative	Preservative to salt
Sulphur dioxide	SO_2 MW 64	Sodium metabisulphite	$Na_2S_2O_5$ MW 190 ($+2 \times SO_2$)	0.674 (128/190)	1.484 (190/128)
Benzoic acid	$C_7H_6O_2$ MW 122	Sodium benzoate	$C_7H_5O_2Na$ MW 144	0.847 (122/144)	1.1803 (144/127)
Sorbic acid	$C_6H_8O_2$ MW 112	Potassium sorbate	$C_6H_7O_2K$ MW 150	0.747 (112/190)	1.696 (190/112)

The actual additions would be sodium metabisulphite (solution or solid) and aqueous solutions of sodium benzoate or potassium sorbate. Convenient conversions for these materials are shown in Table 6.8.

Because of the limited solubility of benzoic and sorbic acids in water, great care must be exercised during the manufacturing process of dilutables to ensure that acidification does not result in precipitation and loss of the preservatives.

There is a small but growing market for high-value dilutables that are declared to be free from preservatives. These products must be adequately processed using in-bottle pasteurisation and the label clearly marked with the need for time-limited storage in refrigerated conditions.

6.2.3.4.3 Flavourings. Flavourings are widely used in dilutable soft drinks to boost or substitute those occurring naturally. There are other publications that deal with this topic in more detail, but, in brief, it is necessary to ensure that appropriate beverage flavours are selected to produce adequate solubility. Most manufacturers of dilutables will use either natural or nature-identical flavours.

6.2.3.4.4 Colourings. Most dilutable beverages are formulated with added colourings, although, depending on the fruit preparation used, many products will have a significant level of colour delivered by the fruit components. In the United Kingdom, artificial colours are now little used in dilutable beverages except for lime juice cordial, which is usually marketed as a clear product with a synthetic lime-green colour that is difficult to achieve by means of natural ingredients.

The available natural colourings offer a limited range of yellow through orange to red/purple colours for products. The most common natural colours used in dilutables include β-carotene, apocarotenal, curcumin and anthocyanins.

To obtain maximum colour stability a careful balance must be achieved between sulphur dioxide and ascorbic acid contents to avoid bleaching the colours.

6.2.3.4.5 Remaining additives. Various other additives are employed in dilutable soft drinks manufacture including antioxidants, acidity regulators, emulsifiers and stabilisers. Stabilisers are particularly important for ensuring physicochemical stability of the product to avoid unsightly oil ring formation or undue sedimentation of fruit components. Cloudy agents are often used to boost the turbidity of natural fruit components. These ingredients can also be made to incorporate citrus oils and colourings by creating an oil-in-water emulsion using a mixture of permitted emulsifiers and final emulsification of cloud to a low particle size ($<10\ \mu m$).

6.2.3.4.6 Compound ingredients. Compound ingredients were widely used at one time for the manufacture of dilutables. They are still available today but are much less widely used. This probably reflects a number of factors such as the disappearance of many small soft drinks manufacturing companies and the need for precise fruit component content. A compound would typically contain all the components to make a dilutable except for the water and carbohydrate. Thus, a manufacturer would purchase, for example, a 10-fold orange squash compound. By adding the required amounts of sugar and water the manufacturer would make 10 times the volume of compound (i.e. 25 l of 10-fold compound would make 250 l of orange squash) into a product that would contain the legal minimum fruit content.

6.2.4 Manufacturing operations

The manufacture of dilutables is essentially a very simple process, with the required ingredients being mixed in order in a large vessel. After checking the final volume for process variables the mixture is then flash pasteurised and filled into the required containers.

The process is diagrammatically summarised in Figure 6.2.

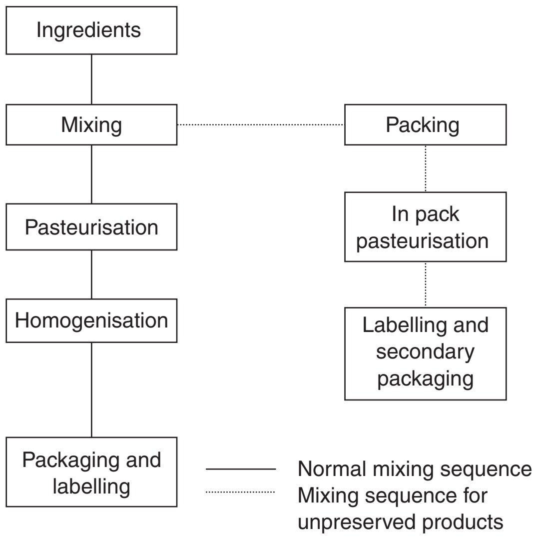

Figure 6.2 Process for manufacture of dilutables.

6.2.4.1 Ingredients

Addition of the ingredients in the correct order is essential to avoid production problems. The normal order starts with the presence of around 30–50% of final product volume of process water to which preservatives other than sulphur dioxide are first added. This volume should be as large as possible to allow the addition of carbohydrates and fruit components, which follow in that order. At this point, the volume should be approaching 90% of final volume to allow the dilution of preservatives. Acidulant is then added, followed by colourings, flavourings and all other components.

The last ingredients to be added are cloud emulsions. Sulphur dioxide should ideally be added after the final make-up water to avoid loss to atmosphere of the gaseous preservative.

6.2.4.2 Mixing

A stainless steel mixing vessel, fabricated from 316 grade stainless steel with some form of cover that allows access for ingredient addition, is an ideal unit for mixing dilutables. The vessel is normally fitted with a stirrer, the power and design of which take account of whether sugar is to be added as a crystalline solid (and thus dissolved) or added as a syrup. Either a top-mounted propeller stirrer or a side-entry unit will mix components adequately, especially if the inside surface of the vessel is fitted with fixed baffles. The use of a stirrer that creates a sufficient vortex to draw in air should be avoided.

High-shear stirrers can be a useful way of mixing components, but they often draw in air and can destroy added emulsions. Mixing can also be done through an external circulating loop with an in-line pump or emulsifying mixer. All systems should ideally be connected to a clean-in-place (c.i.p.) system.

6.2.4.3 Pasteurisation

6.2.4.3.1 Flash pasteurisation. For a normally preserved dilutable soft drink the typical conditions for flash pasteurisation are 85–90°C for 30–60 s. The actual conditions should be determined by reference to the quality of ingredients used, although pasteurisation must never be used as a means of employing sub-standard components. Products containing particulate such as fruit cells should be pasteurised in a plate pasteuriser with 3–4 mm spacing or in a tubular pasteuriser. Great care must be taken to avoid microbial contamination downstream of the pasteuriser. Pasteurised product should be stored in a very clean (or even aseptic) bulk buffer tank prior to filling.

6.2.4.3.2 In-pack pasteurisation. In-pack pasteurisation is normally reserved for dilutable products that are made without preservatives. Unless there are particular circumstances that demand a preservative-free product (e.g. manufacture of a certified organic product), preservatives should always be used in dilutables because of the way the drinks are used and stored. A dilutable without preservatives is very vulnerable to microbial contamination, which can lead to fermentation and possibly bottle bursting. Dilutables without preservative must be labelled to encourage refrigerated storage and short shelf life.

In-pack pasteurisation normally demands very large and expensive tunnel pasteurisers which have several stages. Bottles are introduced into a pre-heating stage, typically around 40°C, to reduce thermal shock, and then into the pasteurisation zone, which will normally be at 70°C, for some 20 min. Following this are two hydro-cooling zones. The first of these reduces product temperature to around 40°C; the second, to ambient temperature. Recovery of heat is essential to an economically viable operation.

Final product temperature should ideally be below 20°C to avoid the phenomenon of 'stack burn' where packed and palletised product that is not adequately cooled will effectively be 'slow cooked'. This can result is excessive browning and the development of a cooked taste.

6.2.4.4 Homogenisation

Some manufacturers homogenise all cloudy dilutable products to obtain maximum physical stability for the product, but others achieve the same result by careful ingredient selection. If homogenisation is to be used, a piston-type unit is preferred with an operating pressure range of around 50–100 bar. As with all beverage manufacturing plant, effective cleaning is essential.

6.2.5 Filling and packaging

Gravity fillers are normally employed for dilutable products, and filling speeds tend to be fairly slow as container sizes are relatively large. For most dilutables the smallest container is usually 0.7 l with sizes up to 3 or 5 l being common.

Most manufacturers now use polyethylene terephthalate (PET) bottles, which provide a good degree of protection from oxygen ingress but without the weight disadvantages of glass packs. For PET bottles, closures are normally moulded polyethylene (LDPE or HDPE), whereas manufacturers packing in glass will normally use roll-on pilfer proof (ROPP) caps made of aluminium.

6.2.6 Product range

The dilutables of the 1960s were fairly limited in range – orange squash, whole orange drink, lemon squash, lemon barley water, lime juice cordial and black-currant cordial being a typical product spectrum. Seasonal products that were added to the range included ginger and peppermint cordials.

Over the past 20 years, the UK market for dilutables has developed and grown to include high-value products that have become a niche market. The range of dilutable products available today is much wider and includes products such as elderflower cordial, summer berry fruits, lime and lemongrass, to name but a few.

6.3 Ready-to-drink non-carbonated products

6.3.1 Overview

There has always been a market for ready-to-drink (RTD) non-carbonated products, but it has been a difficult market to develop because consumers need persuading away from making their own RTD products using dilutables.

The market has often been seen as one of low-quality products with an over-riding convenience factor, but over the last 25 years packaging developments and increasingly affluent consumers have encouraged the development of this market. Early products in the market were packed in pre-formed plastic cups, pouches or early TetraPaks, but today there is a wide variety of packing options available.

6.3.2 Formulations

RTD non-carbonated drinks are usually made by formulations and processes that are identical to the manufacture of dilutables except that the dilution takes place at the manufacturer's rather than the consumer's premises. However, because many non-carbonated drinks often sell in low-price markets, many of the formulations used contain little if any fruit components, although, as mentioned above, a market has now developed for higher-unit-value products in more expensive packaging forms.

6.3.3 Special problems

There are particular problems in the manufacture of non-carbonated RTD beverages that are not aseptically packed. These relate to microbial contamination. Products that have no carbon dioxide in their head space are particularly vulnerable to contamination by moulds and certain types of bacterial infection. For many years it was possible to control such potential contamination by the use of low levels of sulphur dioxide (50 ppm). Changes in European Preservative Regulations now make the use of this preservative in RTD formulations (but not dilutables) illegal unless it is ‘carried over’ from a fruit component, when up to 20 ppm SO_2 may be present. Even at this level, the gaseous preservative is rapidly lost and is quickly ineffective.

To avoid such microbial problems manufacturers must either employ aseptic packing lines, which are very capital-intensive, or use flash pasteurisation and scrupulous downstream hygiene and close control over formulations.

One significant difference in these RTD products is that levels of preservatives will normally be raised to close to the permitted maximum to gain maximum benefit.

A further potential problem with non-carbonated RTD products is that they invariably contain atmospheric air in their headspace as there is no carbon dioxide to displace it. This often leads to undesirable oxygen levels in the product with resulting flavour and colour deterioration in a short time period.

Accordingly, it will often be necessary to adjust the product formulation to incorporate appropriate levels of antioxidants, such as ascorbic acid, and to use flavour and colour preparations that are stable to oxidation.

6.3.4 Manufacturing and packing

The normal manufacturing and packing sequences for both aseptic and non-aseptic products are shown in Figure 6.3.

Various alternative configurations can be used, and in particular some manufacturers employ non-aseptic form–fill–seal operations which usually produce either in-line cup packs or cartons such as TetraPak or Combibloc packs.

6.3.5 Packaging types

Many non-carbonated RTD products that are not pure fruit juices or nectars are packed in either pre-formed or form–fill–seal plastic packages, although an

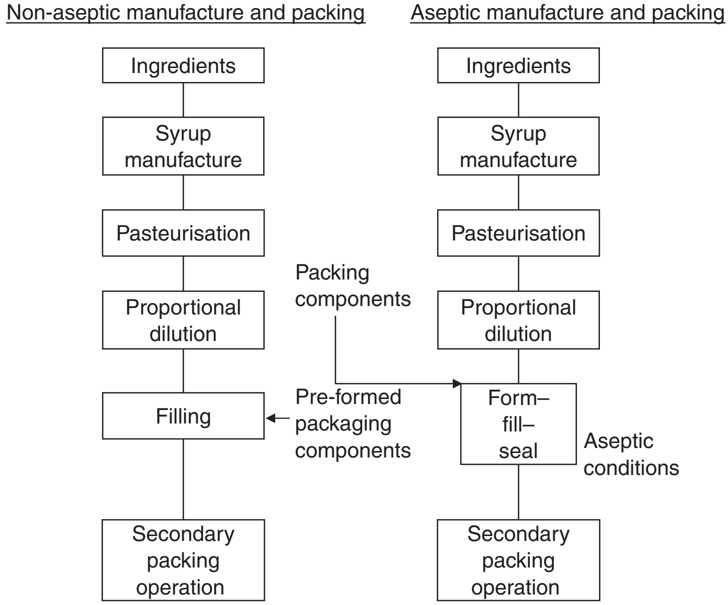

Figure 6.3 Normal manufacturing and packing processes for aseptic and non-aseptic products.

increasing number are now packed in PET bottles. Flexible pouches have also been used by a number of manufacturers.

Depending on the shelf life required, the use of some form of barrier in the packaging is highly desirable or even essential. Much of the packaging used today will be based on a rigid or semi-rigid container employing polystyrene as a major component. The incorporation, often in the form of laminated structure, of a barrier plastic can significantly enhance the product shelf life by reducing the rate of oxygen transfer. Some flexibility can be introduced into containers by incorporating polyethylene into the laminate.

The increased use of PET bottles for packing non-carbonated RTD drinks probably reflects the availability and convenience of this form of packing coupled with the low oxygen transfer rate.

6.4 Fruit juices and nectars

This chapter will not provide the background to the production of fruit juices and nectars, as that is dealt with elsewhere in this volume. However, fruit juices and nectars represent the largest volume of non-carbonated beverages that are sold in almost every marketplace. It is therefore appropriate that some aspects of these products, particularly those relating to processing and packaging, are mentioned here.

6.4.1 Processing

Fruit juices and nectars are highly susceptible to fermentation and other forms of microbial spoilage and with few exceptions it is essential that some form of pasteurisation is employed when these products are packaged. The exceptions that are seen usually relate to freshly squeezed orange (or other) juices that are processed directly from fresh fruit and packaged immediately. These products have a very short shelf life, usually a few days, and are maintained by storage at temperatures between 0 and 5°C.

There is also a market for reconstituted fruit juice made from concentrate and not further processed but maintained, during its short shelf life, by refrigeration.

For all other fruit juice and nectar products either frozen storage or in-pack pasteurisation will be used, although some manufacturers who employ a hot fill process.

6.4.1.1 Flash pasteurisation

Typical flash pasteurisation operations for fruit juices and nectars will employ a plate pasteuriser with heat recovery and final product cooling. Typical flash pasteurisation conditions will use temperatures between 85 and 95°C with holding times varying between 15 and 60 s. Selection of the appropriate conditions will depend on the product, including the level of microbial load pre-pasteurising. If enzyme deactivation is required as well as microbial removal then a temperature between 90 and 95°C will normally be used. At these temperatures, holding times are normally reduced to around 15 s.

Juices containing cells, particulate material or products that are particularly viscous, such as some of the tropical juices, may be pasteurised in tubular units or plate pasteurisers with wide (3–5 mm) spacing.

For aseptic packaging operations, flash pasteurisers are often linked integrally with the aseptic packaging unit, either directly or via an aseptic buffer tank. When flash pasteurisation is used, care should be taken to minimise product recirculation when the pasteuriser is in divert mode. Excessive recirculation can lead to thermal damage to the product, resulting in unpleasant cooked flavours and product browning.

6.4.1.2 In-pack pasteurisation

In-pack pasteurisation is often regarded as a foolproof operation, although product integrity will ultimately rely on the seal provided by the pack closure.

In-pack pasteurisation can be achieved at very low cost by simply immersing bottled product, with closures tightly applied, in tanks of heated water. A pre-heat tank at around 40°C should be employed to minimise thermal shock to the containers, and the main pasteurising tank will be held at around 70°C. A single container into which is inserted a remote temperature probe should be used to ensure that the whole contents of the bottles reach pasteurising temperature.

The normal means of achieving in-pack pasteurisation is to use a tunnel pasteuriser. These are large, capital-intensive pieces of plant and require significant floor space and provision of services. Most units work by using water sprays in a pre-heating zone, pasteurising zone and cooling zone(s). Some form of heat recovery is almost essential if a tunnel pasteuriser is to work economically.

After containers leave the pasteuriser they should be air-dried and then labelled. Typical pasteurising conditions will be 70–75 °C for up to 20 min.

6.4.1.3 Hot filling

Hot filling provides a further means of ensuring the microbial integrity of fruit juices and nectars. The bulk product is heated to the required temperature then filled into containers and the closure applied. If glass bottles are used, they should be pre-heated, for example, by a warm water spray, before filling to minimize thermal shock. Following filling, containers are usually rotated through 360° to ensure contact between hot liquid and the whole inside of the container and cap.

Depending on the fill temperature, which is usually around 70–80°C, the filled containers will be held for the required time before being placed in a hydro-cooler. Containers should be cooled to below 25°C before being stacked. This will avoid further low-temperature ‘cooking’ of product inside a stack of containers. Labelling is carried out after air-drying the containers.

6.4.1.4 High-pressure pasteurisation

Claims have been made for the successful high-pressure pasteurisation of fruit juice in containers. The equipment required is expensive and the process is carried out as a batch operation, which tends to be both slow and ineffective. It may, in future, provide a very interesting means of low-temperature pasteurising of fresh juices, thus retaining all the flavour characteristics of the product.

6.4.2 Packaging

6.4.2.1 Boxes

Most fruit juices for retail sale are now in cartons, a high proportion of which will be aseptic packs. Cartons are formed, filled and sealed in a single operation, which will either be clean or aseptic depending on the product and shelf life sought. Typical packs include TetraPaks, Combibloc and Elopak.

The long-shelf-life packs for aseptic products are often made of a board, foil, plastic (polyethylene) laminate which gives protection from oxygen ingress and light as well as mechanical strength and an excellent surface for printed material. Cardboard packs for short-shelf-life products are often a simpler laminate excluding foil.

6.4.2.2 Bottles

The selection of a container for fruit juices will often be based on a combination of the technical, cost and marketing needs. Many outlets for fruit juices require relatively small unit packs and these will invariably be glass bottles. In the United Kingdom, most fruit juice in bottles is limited to these small units (e.g. 200 ml), whereas many European markets prefer larger (up to 1 l) glass bottles with wide necks.

Glass bottles will normally be pasteurised using either the hot fill method or in-pack pasteurisation.

There is some limited use of plastic bottles for juice packing – mostly related to short-shelf-life products sold from the chill cabinet. Plastics are now available to support aseptic filling or even hot fill operations, but they have found limited support for the sale of fruit juices.

6.4.2.3 Cans

The sale of fruit juice in cans to either the retail or industrial markets has largely died out with the availability of other forms of packaging and storage. Some juices from developing country suppliers (e.g. mango juice) are still supplied in cans containing around 5 kg. This reflects the technology and packaging available in the supply country.

6.4.2.4 Bulk packs

Juice for industrial use has, over many years, been packed in a wide variety of drums. Typical drums are open-head steel containers with the juice packed inside several plastic bags. This package is the usual container for frozen juices and typically contains around 200 l.

Plastic drums have also been widely used over many years without the need for plastic liners, but they are less suitable for freezing as the plastic has a tendency to become brittle and may rupture. Plastic drums usually contain 200–250 l, although larger containers have been very successfully used – especially for chemically preserved juices. The Israeli manufactured 'Rotoplas' container was probably the best-known example. This container typically held around 1300 l.

Aseptic bulk packing has now become a well-established means of packing concentrated or RTD juice. Containers are available from as little as a 5 l bag-in-a-box to be dispensed from a bar up to a 1 000 l bin in a 1 m^3 pallet box.

Finally, the transportation of fruit juice in temperature-controlled bulk road tankers of up to 25 000 l is well established as is intercontinental transfer by shipping tankers.

Further reading

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7 Carbonated beverages

D. Steen

7.1 Introduction

Naturally occurring carbonated mineral waters have been known for a long time. These effervescent waters exist as a consequence of excess carbon dioxide in an aquifer dissolving under pressure. Although claims for the medicinal properties of these mineral waters have been grossly exaggerated, the presence of carbon dioxide does make aerated waters and soft drinks both more palatable and visually attractive: the final product sparkles and foams. The first non-carbonated soft drinks appeared during the seventeenth century. In 1767, Joseph Priestley produced the first man-made, palatable carbonated water. Three years later a Swedish chemist, Torbern Bergman, invented a process that produced carbonated water from the reaction between chalk and sulphuric acid, allowing the commercial production of aerated mineral water. In 1783, Jacob Schweppes, a young watchmaker and amateur scientist, perfected an efficient system for manufacturing carbonated mineral water and founded the Schweppes Company in Geneva. He relocated to Drury Lane, London, England in 1790. Since then, the addition of flavourings to aerated waters has seen the development of major soft drinks brands throughout the world. To meet the need for carbonated soft drinks, the soda fountain was developed by Samuel Fahnestock in the United States in 1819. The patenting of the Crown cork by William Painter in 1892 and the automatic production of glass bottles using a glass-blowing machine by Michael J. Owens in 1899 were notable achievements that at last allowed carbonated soft drinks to be successfully bottled without significant loss of carbonation. Since then, developments in closure technology, polyethylene terephthalate (PET) bottle production, can design and manufacture, syrup making methods, carbonation technology and filling machine manufacture have led to the worldwide beverage industry as we know it today.

7.2 Carbon dioxide

Carbonation is the impregnation of a liquid with carbon dioxide gas. Carbon dioxide is a non-toxic, inert gas that is virtually tasteless and is readily available at a reasonable cost. It is soluble in liquids (the degree of solubility increasing as the liquid temperature decreases) and can exist as a gas, liquid or solid. When dissolved in water it forms carbonic acid. It is carbonic acid in combination with the product that produces the acidic and biting taste found in carbonated waters and soft drinks. Above a certain level of carbonation, carbon dioxide also has a preserving property, which is a bonus from its use.

Carbon dioxide gas is heavier than air; it has a specific gravity of 1.53 under normal conditions of temperature and pressure. It has a molecular weight of 44.01 and does not burn, although it will support the combustion of magnesium. It is a fairly stable compound that decomposes into carbon and oxygen only at very high temperatures. It can cause death by suffocation if inhaled in large amounts. The gas is easily liquefied by compression and cooling. When liquid carbon dioxide is quickly decompressed it expands rapidly and some of it evaporates; this evoporation removes sufficient heat that the remainder cools into a solid. Carbon dioxide is part of the atmosphere, making up about 1% by volume of dry air. In various parts of the world it is formed underground and issues from fissures within the earth. This happens notably in Italy, in Java and in Yellowstone National Park in the United States. Carbon dioxide is a known contributor to the greenhouse effect; the proportion in the atmosphere increases each year, thus disrupting the natural carbon dioxide cycle.

The phase diagram in Figure 7.1 shows the effect of temperature and pressure on the state of carbon dioxide. At the triple point, carbon dioxide can exist in the three states as a solid, a liquid or a gas by just a small perturbation. All phases are in a state of equilibrium at the triple point, which is at 5.11 bar and −56.6°C. Above 31°C, it is impossible to liquefy the gas by increased pressure; this is termed the critical point. At normal temperatures and pressures carbon dioxide is a colourless gas; at high concentrations it has a slightly

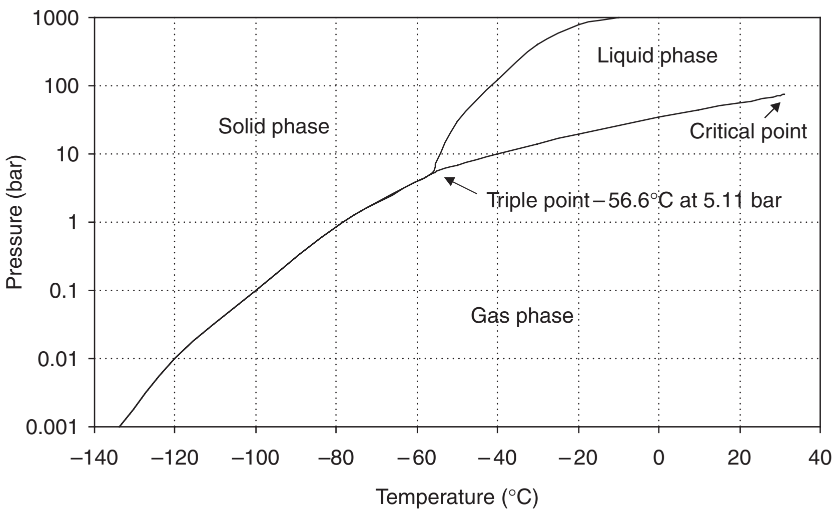

Figure 7.1 Carbon dioxide phase diagram.

pungent odour. Carbon dioxide cannot exist as a liquid at atmospheric pressure. Liquefaction can be achieved by compression and cooling between the pressure and temperature limits at the triple point and the critical point. Above the critical point of 31°C it is impossible to liquefy the gas by increasing the pressure above the corresponding critical pressure of 73 bar. When liquid carbon dioxide under pressure is released into the atmosphere it will be as a gas and a solid only, in the form of a dense white cloud due to the solid content and the condensation of atmospheric moisture at the low temperatures obtained. The solid will fall to the ground as snow, which, when compressed, forms a translucent white solid known as dry ice.

7.3 Carbon dioxide production

Several methods of carbon dioxide production are in commercial use. These include the reaction between sulphuric acid and sodium bicarbonate, the combustion of fuel oil, the extraction of carbon dioxide from the flue gas of a boiler or similar heating facility, the distillation of alcohol and the fermentation of beer; carbon dioxide is also a byproduct of fertiliser manufacture. Following manufacture the gas must be cleaned to ensure it is free from impurities and is fit for purpose. Two typical processes are described below.

7.3.1 Fermentation

When a sucrose- or other simple carbohydrate-based solution is mixed with yeast and oxygen in a fermenter, carbon dioxide vapour and alcohol are produced. The carbon dioxide can then be passed through a separator to remove any trace carry-over of foam. Once the foam has been removed the carbon dioxide is compressed. It is then scrubbed with water in a packed tower, removing water-soluble impurities such as alcohol, ketones and other aroma chemicals produced during fermentation.

7.3.2 Direct combustion

A hydrocarbon fuel such as light oil or natural gas can be burned specifically in order to produce carbon dioxide. The flue gas from this process, which contains less than 0.5% oxygen by volume, is cooled and scrubbed to remove any impurities that may be present. The resultant gas is then passed through an absorbent tower, where it comes into contact with a carbon dioxide absorbing solution. The absorbing solution, now rich in carbon dioxide, is pumped to a stripper tower, where the heat from the combustion of the fuel is used to release the carbon dioxide in vapour form. The absorbing solution is then recycled and reused. The resultant carbon dioxide vapour is then cooled and further treated to meet the requirements for use within a beverage.

Large manufacturers of aerated waters and carbonated soft drinks produce their own carbon dioxide on site using packaged systems, thereby reducing their operating costs and minimising logistical problems.

7.3.3 Quality standards

The European Industrial Gases Association working with the Compressed Gases Association of America and the International Association of Beverage Technologists has prepared a specification for liquid carbon dioxide for use in foods and beverages. This is shown as Table 7.1. It is to this minimum standard that all carbon dioxide to be delivered to soft drinks and aerated mineral water bottles is manufactured.

Table 7.1 Commodity specification for carbon dioxide (CGA/EIGA limiting characteristics)

Component	Specification
Assay	99.9% v/v min.
Moisture	50 ppm v/v max. (20 ppm w/w max.)
Acidity	To pass JECFA test
Ammonia	2.5 ppm v/v max.
Oxygen	30 ppm v/v max.
Oxides of nitrogen (NO/NO_2)	2/5 ppm v/v max. each
Non-volatile residue (particulates)	10 ppm w/w max.
Non-volatile organic residue (oil and grease)	5 ppm w/w max.
Phosphene[a]	$\leq$0.3 ppm v/v
Total volatile hydrocarbons (calculated as methane)	50 ppm v/v max. of which 20 ppm v/v max. non-methane hyderocarbons
Acetaldehyde	0.2 ppm v/v max.
Benzene	0.02 ppm v/v max.
Carbon monoxide	10 ppm v/v max.
Methanol	10 ppm v/v max.
Hydrogen cyanide[b]	<0.5 ppm v/v
Total sulphur (as sulphur)[c]	0.1 ppm v/v max.
Taste and odour in water	No foreign taste or odour

[a] Analysis necessary only for carbon dioxide from phosphate rock sources.
[b] Analysis necessary only for carbon dioxide from coal gassification sources.
[c] If the total sulphur content exceeds 0.1 ppm v/v as sulphur then the species must be determined separately and the following limits apply: carbonyl sulphide, 0.1 ppm v/v max.; hydrogen sulphide, 0.1 ppm v/v max.; sulphur dioxide, 1.0 ppm v/v max.
Source: With thanks to Messer Gases.

7.3.4 Delivery to the customer

Carbon dioxide is delivered as a liquid, most frequently by road tanker. It is then transferred to pressurised vessels of 5–50 tonne capacity and held at a pressure in the region of 20.5 bar at −17°C, the temperature being maintained by the use of a small refrigeration unit. This is shown schematically in Figure 7.2. To change the carbon dioxide from the liquid to the gas phase all that is required is to vaporise the liquid by heating it using either steam, water or electricity. This is normally done in tubular heat exchangers. The most common system is to use steam, which has traditionally been available already in soft drinks plants for bottle washing, pasteurisation and factory heating. The use of water is becoming more common as it is cheaper and the process actually reduces the temperature of the water, which saves subsequent product cooling costs as well as the cost of steam generation. A typical 2 tonne/h carbon dioxide vaporisation system requires a constant flow rate of 50 m^3/h of water at ambient temperature to be effective. Electric vaporisers are expensive and rarely used. Yet another method is the use of ambient air blown across evaporator coils. It is claimed that this achieves significant energy savings over conventional steam vaporisation. Considering Figure 7.2, it is important to ensure that both the gas phase and the liquid phase in the tanker and the receiving tank are connected. This allows the gas displaced by the liquid delivered to the storage tank to flow into the tanker and thus balance the pressure between the two vessels. Non-return check valves must be incorporated to ensure no backflow occurs. The system needs to

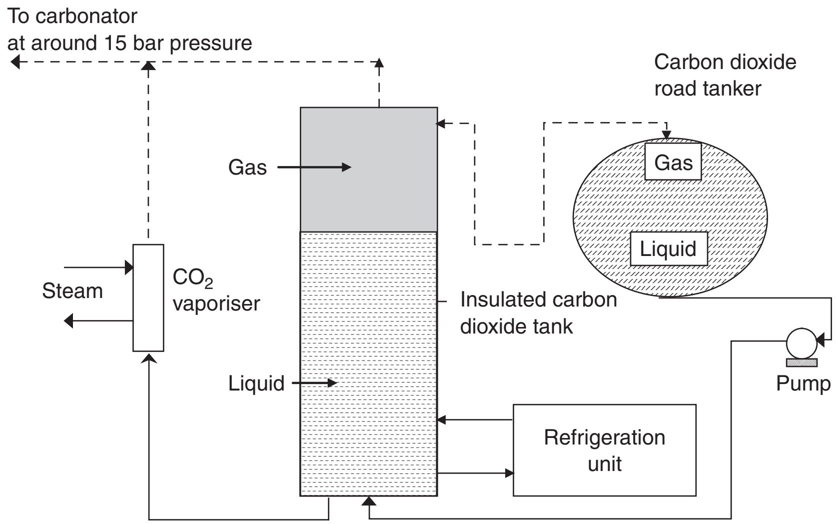

Figure 7.2 Carbon dioxide delivery process.
Note: No valves are shown.

be installed in accordance with the recommendations of the gas supplier, and regular planned maintenance on the system is necessary to ensure any risks are minimised.

7.3.5 Precautions

The user must take great care when using carbon dioxide to ensure that it is fit for purpose. Scares such as the detection of residual benzene in CO_2 and the risk of contamination from nuclear plants have to be considered. All batches supplied must have a certificate of conformance. The supply chain must be regularly audited, including the actual carbon dioxide manufacturing plant, storage and distribution. A full hazard analysis and critical control points (HACCP) survey of the site-installed system is required to ensure any inherent risks are minimised. A list of possible impurities is given as Table 7.2. Depending on the method of carbon dioxide production, the certificate of conformation for each batch must include analytical checks on the relevant listed compounds. These analytical checks need to be carried out to the standards agreed by the International Society of Beverage Technologists (ISBT). The risk of contamination is very real. If too much gas is withdrawn from the top of the tank then contaminants can build up due to distillation of the liquid carbon dioxide within the tank. Other possible risks are oil contamination from the transfer pump between the delivery tanker and the user's tank, degradation of the delivery hose, contamination of the hose connections by particulates, water, oil or mud, and contamination by the backflow of cleaning fluids from the user's process. Dedicated tankers are required that are not used for non-food applications including nuclear plants or where a risk of cross-contamination exists.

7.4 Carbonation

7.4.1 Basic considerations

For a liquid–gas mixture in a sealed container, equilibrium is said to exist when the rates of gas leaving and entering the liquid solution are equal. Take any PET bottle of carbonated soft drink and shake it: the liquid–gas interface will initially fob, but after a short while the equilibrium condition will have been reached. Fobbing is a term used within the carbonated beverage industry to denote product foaming. If the cap is then opened and some of the contents poured out, the cap replaced and the shaking repeated, the bottle will go from being limp before the shaking to being rigid. Gas has come out of solution to attain the equilibrium condition. This state is just stable. Any decrease in pressure or increase in temperature will render the mixture metastable, that is, supersaturated, so that the temperature/-pressure combination is insufficient to keep the carbon dioxide in solution. If this

Table 7.2 Possible trace impurities by source type (excluding air, gases and water)

Component	Combustion	Wells/ geothermal	Fermentation	Hydrogen or ammonia	Phosphate Rock	Coal gasification	Ethylene oxide	Acid neutralisation
Aldehydes	✓	✓	✓	✓		✓	✓	
Amines	✓			✓				
Benzene	✓	✓	✓	✓		✓	✓	✓
Carbon monoxide	✓	✓	✓	✓	✓	✓	✓	✓
Carbonyl sulphide	✓	✓	✓	✓	✓	✓	✓	✓
Cyclic aliphatic hydrocarbons	✓	✓		✓		✓	✓	
Dimethyl sulphide		✓	✓		✓	✓		✓
Ethanol	✓	✓	✓	✓		✓	✓	
Ethers		✓	✓	✓		✓	✓	
Ethyl acetate		✓	✓	✓		✓	✓	
Ethyl benzene		✓		✓		✓	✓	
Ethylene oxide						✓	✓	
Halocarbons	✓					✓	✓	
Hydrogen cyanide	✓					✓		
Hydrogen sulphide	✓	✓	✓	✓	✓	✓	✓	✓
Ketones	✓	✓	✓	✓		✓	✓	
Mercaptans	✓	✓	✓	✓	✓	✓	✓	
Mercury	✓					✓		
Methanol	✓	✓	✓	✓		✓	✓	
Nitrogen oxides	✓		✓	✓		✓	✓	✓
Phosphine					✓			

Radon					✓			✓
Sulphur dioxide	✓	✓	✓	✓	✓	✓		✓
Toluene		✓	✓	✓		✓	✓	
Vinyl chloride	✓					✓	✓	
Volatile hydrocarbons	✓	✓	✓	✓		✓	✓	
Xylene		✓	✓	✓		✓	✓	

Note: Examples of specific analytical methods are given in the ISBT document. *Analytical Methods for CO_2 Analysis.*

Source: As prepared by an ad hoc working group of the European Industrial Gases Association working in conjunction with the Compressed Gases Association of America and the International Society of Beverage Technologists (ISBT), EIGA Document 76/01.

occurs, gas is spontaneously released, giving rise to fobbing. If the mixtures are agitated or some irritant, such as small particulates, is added to the mix, the rate of gas release will be even more pronounced. This is due to nucleation sites being generated by the presence of these particulates or other gases, such as air.

Any carbonated product that is kept in a container that is open to the atmosphere will gradually lose carbonation. This is due to the gas being liberated into the atmosphere as the liquid–gas interface continually strives to achieve the equilibrium condition. In a closed container the gas fills the container headspace, thus increasing the headspace pressure. This happens quickly at first and then slowly as equilibrium is approached. The rate of transfer of gas from the product to the headspace depends on the proximity of the headspace pressure to the equilibrium pressure, the temperature of the liquid, the nature of the beverage, the extent of any agitation and the presence of any irritants. A quiescent, stable product will take many hours to reach equilibrium when not subjected to any external forces such as agitation, movement, temperature or pressure change. However, the same product roughly shaken will take only seconds to achieve the equilibrium condition. The faster the rate of change towards the equilibrium condition, the sooner this condition will be reached. For a given volume, the amount of carbon dioxide that can be retained in solution depends on the temperature and pressure. The lower the temperature, the greater the amount of carbon dioxide that is retained. Conversely, the higher the temperature, the greater the pressure required to maintain the carbon dioxide in solution. Henry's law was postulated by William Henry (1774–1836) and states that 'The amount of gas dissolved in a given volume of solvent is proportional to the pressure of the gas with which the solvent is in equilibrium.' Charles's law (Jacques Charles, 1746–1823) states that 'The volume of an ideal gas at constant pressure is directly proportional to the absolute temperature'. These two laws can be combined to form the universal ideal gas law:

$$pV = mRT$$

where p is the absolute pressure, V is the volume, m is the number of moles of gas, R is the gas constant (for that particular ideal gas) and T is absolute temperature. A mole is that quantity of a substance which has a mass numerically equal to the molecular weight of the substance. For carbon dioxide, the molecular weight is 44.01 and R is 0.18892 J mol^{-1} K^{-1}. From this relationship the carbonation chart shown as Figure 7.3 can be deduced. Here the concept of carbonation volumes is introduced. Volumes 'Bunsen' measures the gas volume at atmospheric pressure (760 mm of mercury) and the freezing point of water (0°C). It is defined as the number of times the total volume of dissolved gas can be divided by the volume of liquid in the container. As an example, a product with 4 volumes carbonation will contain a volume of carbon dioxide four times the volume of the beverage. A 1 l container carbonated to 3.5 volumes would contain 3.5 l of carbon dioxide, and likewise a 3 l container carbonated

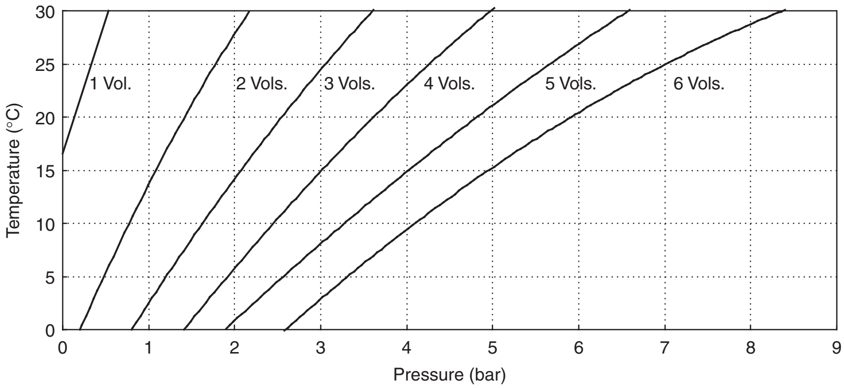

Figure 7.3 Carbonation chart.

to 4 volumes carbonation would contain 12 l of carbon dioxide. One volume 'Bunsen' is equivalent to 1.96 g carbon dioxide per litre. This is often simplified to 2 g/l. For PET bottles, the smaller the container normally the higher the carbonation volumes, as the rate of loss of carbon dioxide by permeation due to a high surface-to-volume ratio is large. A 2 l bottle can easily meet a shelf-life requirement of no more than 15% carbonation loss in 12 weeks. This will reduce to around 9 weeks for a 500 ml bottle and some 7 weeks for a 250 ml bottle. The light weighting of PET bottles gives rise to thinner wall thicknesses and hence greater permeation and a shorter product shelf life. Cans have carbonation levels up to a maximum of 3.5 volumes; above this, the internal pressures that can be generated during expected use would cause can rupture or deformation. Glass bottles can be designed to accommodate higher pressures for products such as tonic water, which is traditionally a high volume carbonation product. The level of carbonation will depend on design and wall thickness.

7.4.2 Carbonation measurement

The measurement of carbonation is carried out using a device similar to that shown as Figure 7.4. It consists of a jig in which the container can be restrained, and a piercer attached to a pressure gauge. The container is placed in the jig, and is first of all pierced and, then shaken before the pressure is measured. The release valve is opened until the pressure gauge reads zero and all the gas has been exhausted from the container headspace. The release valve is then closed and the container shaken again. The pressure is re-taken. The container is released from the jig and the temperature of the contents measured. A carbonation chart is then used to determine the volumes of carbonation.

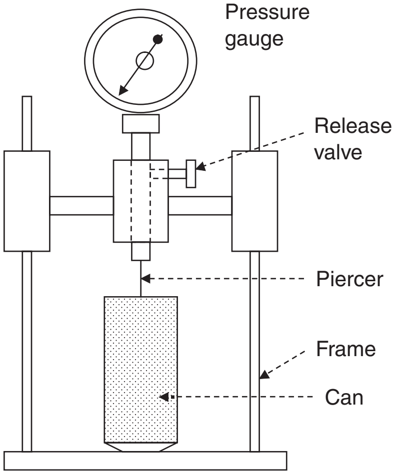

Figure 7.4 Measurement of carbonation.

The pressure must be released from the container before the pressure reading is taken because of the problem of air inclusion in the beverage. This gives a two-gas system of air and carbon dioxide, and it is necessary to release the air to determine how much carbon dioxide is present. Air has approximately one-fiftieth the solubility of carbon dioxide in a liquid. Hence any air contained within the beverage will exclude some 50 times its own volume of carbon dioxide. Under Dalton's law of partial pressures, the pressure of a mixture of gases is equal to the sum of the pressures of the individual constituents when each occupies a volume equal to that of the mixture at the temperature of the mixture.' The main constituents of air are around 79% nitrogen and around 21% oxygen, ignoring for simplicity the presence of the inert gases. So in any carbonated mixture carbon dioxide, nitrogen and oxygen will be present. Owing to the differing solubility and proportions of oxygen and nitrogen, the dissolved air actually contains 35% oxygen and 65% nitrogen as the solubility of nitrogen is low. (This oxygen enrichment can give rise to spoilage problems if care is not taken to minimise the amount present.) The presence of air will give rise to a higher pressure and hence a false reading of the volumes carbonation from the carbonation chart; thus the amount of air present clearly has to be minimised when taking carbonation measurements. If we consider a bottle with a gas head-space of 5% of the bottle volume, on the first snift the gas loss would be 5% of the bottle volume. On the second snift we would lose a further 5%. If carbon dioxide only were present in the headspace, we would expect to lose 5% pressure on the first shake and some 7% by the second shake. If other gases were present, we would lose more pressure. Thus, the amount of air present in the product can also be estimated during carbonation measurement. If excess air is found (often caused by air entrainment or poor sealing with the filler bowl) then action needs to be taken to minimise its presence. This can be seen from an example where, by volumetric analysis, it has been found that the headspace of a carbonated drink container contains 90% carbon dioxide, 3.5% oxygen and 6.5% nitrogen. Considering the properties of perfect mixtures, which is only an approximation of the actual situation, it follows from Dalton's law that if

$$x_i = n_i/n$$

where *i* corresponds to on individual component, *n* is the mole fraction defined as $n = m/M$, *m* is the number of moles and *M* is the molecular weight, then

$$p_i = x_i\, p$$

For our example, if the mixture pressure is taken as 4 bar, we can develop the following table:

	M_i	V_i / V	$(V_i / V)M_i$	m_i/m	p_i
Oxygen	32	0.035	1.12	0.026	0.14
Nitrogen	28	0.065	1.82	0.043	0.17
Carbon dioxide	44	0.90	39.60	0.931	3.60
Total		1.00	42.54	1.000	4.00

The carbon dioxide pressure is only 3.6 bar. From the carbonation chart, assuming a temperature of 20 °C, the volumes carbonation at 4 bar would be 4.3 but only 4.1 at 3.6 bar. Hence, with the presence of air we have lost 0.2 volumes carbonation. This is the maximum tolerance normally given for volumes carbonation during the manufacture of soft drinks.

7.5 Syrup preparation

Most products are traditionally prepared as a syrup-plus-water mix, in a ratio of some 1 part (volume) syrup to between 3 and 6 parts (volume) water. This allows a concentrated batch of syrup to be made and then proportioned with water to form the final product. For a sugar-based product the syrup would typically consist of 67°Brix sugar, citric acid, flavourings, colourings, preservatives and water. The ingredients are carefully weighed out and added to the mixing vessel. The syrup is pre-prepared and fully tested before being sent to the proportioner for mixing with water and subsequent carbonation. This is carried out in the syrup room as a batch process, allowing the multitude of soft drink flavours to be catered for.

Various methods exist to accurately proportion syrup and water, though the most popular current system uses flow meters. The syrup is usually dosed though a mass flow meter and the water is dosed volumetrically using a magnetic induction flow meter. This allows for density variations within the syrup to be accounted for to give the required Brix of the final product, since a mass flow meter works on the same Coriolis principle as a densitometer, although the degree of accuracy of measuring density using a mass flow meter is an order of magnitude less than if a densitometer is employed. (The Coriolis principle is an effect whereby a mass moving relative to a rotating frame of reference is accelerated in a direction perpendicular both to its direction of motion and to the axis of rotation of the frame. It explains why water flows down a plughole clockwise in the northern hemisphere and anticlockwise in the southern hemisphere, and is named after G.G. de Coriolis (1792–1843).) The density of water, within the range under consideration, does not vary significantly and hence the simpler volumetric flow meter can be used. The latest adaptations of these proportioners allows for the final product either to be collected in large vessels of some 30 000 l capacity or greater or to be fed direct online to the carbonator, with the syrup being individually proportioned as a premix and online metering of sugar, citric acid and other components. The accuracy of mass flow meters ensures the product is produced at the required Brix, thus ensuring conformance to specification, tight cost control and minimum wastage. A typical system is sketched as Figure 7.5. The older Mojonnier system uses a fixed orifice to meter the syrup component whereas the water is fed through a variable orifice, both operating under a constant head pressure as shown schematically in Figure 7.6. Other systems in use employ volumetric dosing pumps.

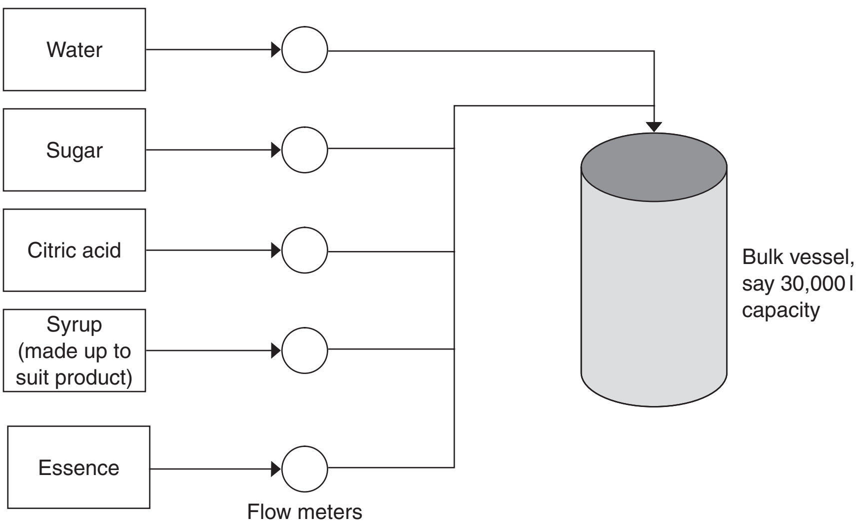

Figure 7.5 Product preparation using flow meters.
Note: It is normal to water-flush between introducing each component to ensure the feed lines are clear of the previous ingredient.

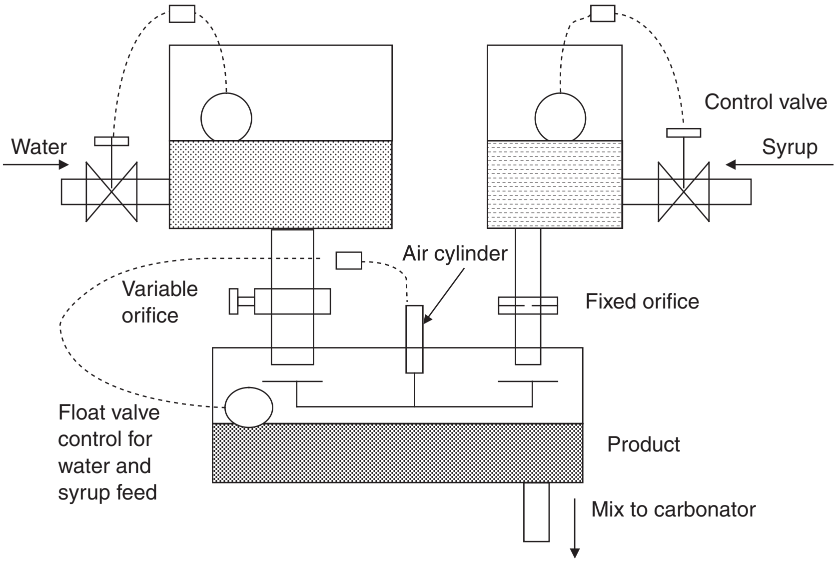

Figure 7.6 Mojonnier proportioning system.

7.6 De-aeration

Why de-aerate? As discussed earlier, the presence of air in a product causes product deterioration, as well as giving a false reading of the level of carbon dioxide present due to the partial pressures involved. Experience has shown that the aim should be to reduce the level of air within a product to below 0.5 ppm wherever possible. In this way the product will be at minimum risk from deterioration due to the presence of oxygen; hence shelf life will be improved and filling problems minimised. The presence of air and carbon dioxide causes nucleation sites within the products, giving rise to the phenomenon known as fobbing. The higher the air content the more difficult it is to hold carbon dioxide in solution. Two main methods of de-aeration exist, vacuum and reflux, both of which are normally applied to water before mixing with syrup rather than to the final product. By de-aerating only the water no product contamination of the equipment occurs and consequently less product risk is involved. Also the frequency of cleaning is decreased. In modern plants, it is normal to de-aerate all the water used in the product, including the water used to make up the syrup content, thus minimising the amount of air that is present in the final product. Simple methods such as introducing all liquids into mixing vessels through the base of the vessel will minimise the entrainment of air, as will careful mixing of the product.

The most effective method of de-aeration is to atomise water into a vessel held under a vacuum. In this way air is stripped out as the atomised water is exposed to the vacuum. Alternatively, if a positive carbon dioxide pressure is applied in a sealed vessel (reflux de-aeration) the air attaches to the carbon dioxide, in a process known as nucleation, and is then driven off through a vent. Often these two processes are combined, such that the effective use of vacuum de-aeration followed by reflux de-aeration will give an air content of less than 0.5 ppm in the water and of order 0.5 ppm in the final product, as the syrup will also have been produced from de-aerated water.

7.7 Carbonators

Carbonators take the form shown schematically in Figure 7.7. The final product is fed to a vessel pressurised with carbon dioxide gas. The rate of flow and the pressure of the carbon dioxide are critical to ensure the correct carbonation level. The greater the liquid surface area exposed to the carbon dioxide, the higher the rate of absorption of the carbon dioxide by the liquid. The carbon dioxide is often sparged into the liquid under pressure; this allows small bubbles of gas to be formed which can be easily absorbed by the liquid. The higher the pressure, the smaller the gas bubbles formed at the sparger and the greater the gas bubbles' surface area made available for the gas to be absorbed by the liquid. Early carbonators used refrigeration to carbonate at around 4°C. A typical system is sketched as Figure 7.8. Water alone is often carbonated to ensure minimum contamination of the system by syrup. The product is spread over chilled plates, such that the product runs down the plates as a thin film. This is carried out in a constant-pressure carbon dioxide atmosphere, the lighter, displaced air being bled off. Chilling the product as a film maximises the surface area

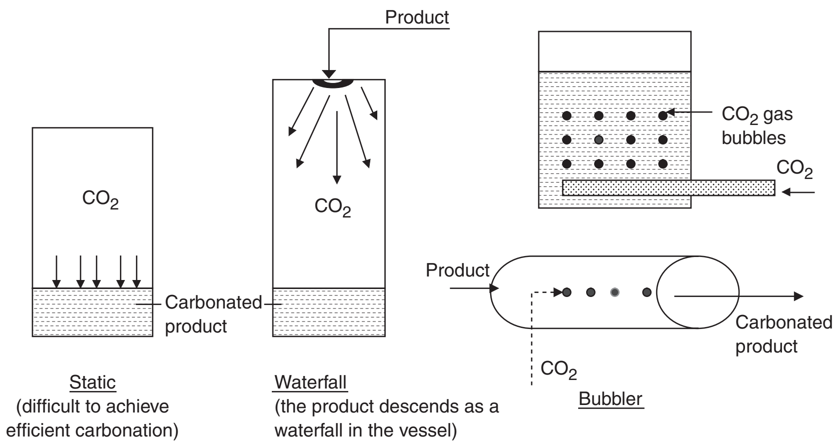

Figure 7.7 Carbonation methods.

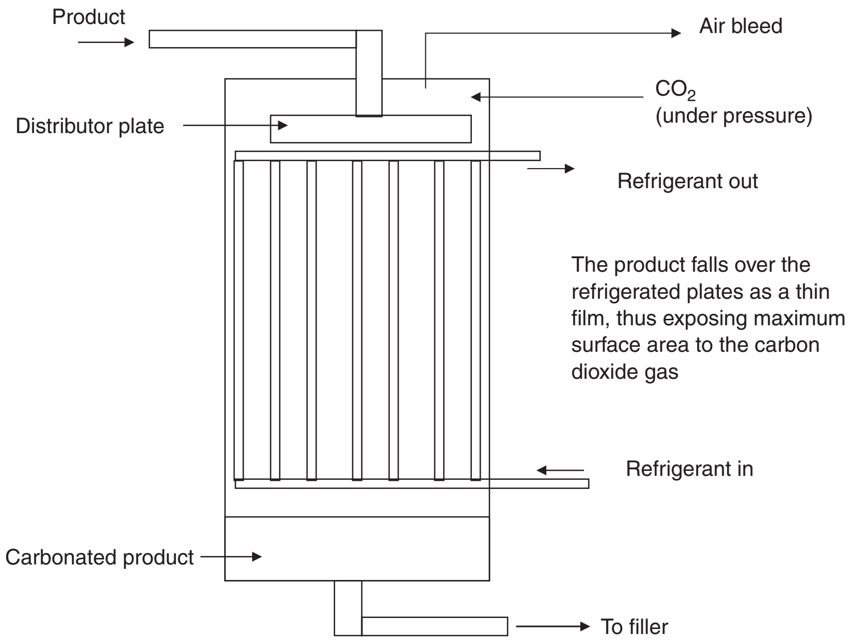

Figure 7.8 Refrigeration carbonation.

available to the carbon dioxide, thus promoting effective carbonation. This has the added benefit that at a lower temperature the carbon dioxide stays in solution more easily, thus minimising future filling problems. However, the energy usage is high and packaging problems are created due to condensation within shrink-wrapped packs as the temperature reaches ambient conditions. This is especially a problem for steel cans, where corrosion can easily occur. To overcome this problem the packaged product must be warmed to ambient conditions, thereby further increasing the energy load.

7.8 Filling principles

A carbonated product made to specification has then to be filled into the required container at a commercially viable filling rate. This is achieved under gravity, the rate of flow being dependent on the head difference between the filler bowl and the container. The rate of flow will increase if an overpressure is introduced. With reference to Figure 7.9, the pressure from the top of the filling bowl to the outlet of the filling valve provides the driving force to fill the container. The example shows a bottle, but the principle is the same for a can or carton. The rate of flow to fill the container is a function of the overpressure

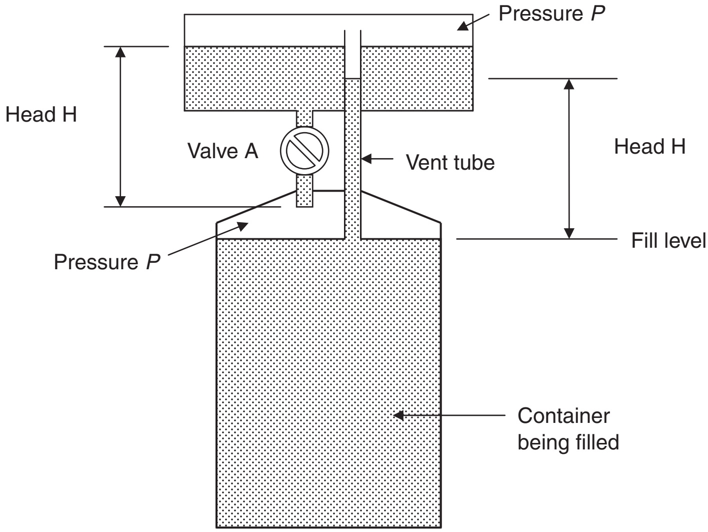

Figure 7.9 Principles of gravity filling.

1 Seal container to the filler
2 Both the container and the header tank are under the same pressure *P* due to the connecting vent tube.
3 Open the liquid valve A.
4 Filling starts.
5 Filling stops when the vent tube is covered by the liquid and the pressure heads H equate.

applied to the top of the filling bowl (p), the viscosity of the liquid to be filled (μ), the diameter of the filling tube (d) and the length of pipe (l). For simple flows this can be expressed by the Poiseuille formula as

$$\text{Volumetric rate of flow}, V = \frac{\Pi p d^4}{128 \mu l}$$

This is a much simplified equation for very-low-speed laminar fluid flow. In actual practice, the rate of flow would probably be turbulent and proportional to $\sqrt{p}$ rather than to the pressure alone. It does demonstrate that for viscous liquids it is necessary either to increase the filling tube diameter or to increase the driving pressure to maximise the flow rate through an orifice. Considering the process in more detail reveals some of the problems facing the filler designer, especially with regard to how the process is controlled. It is simple to envisage how a container is filled under gravity alone: it is the same as filling a bottle from the kitchen tap. To control the process under pressure with carbonated product is more complex. However, if the pressure in the container and the pressure of the gas in the filler bowl headspace are the same, gravity filling conditions will apply. This is exactly what is done.

7.8.1 Gravity filler

It is first necessary to seal the container to the filler bowl such that no leakage can take place around the seal. This applies whether the container is glass, plastic or metal. The filler bowl is filled to a given level, which is maintained within close tolerances by means of float valves. This ensures a near-constant pressure head during the filling process. In terms of Figure 7.9, with the container sealed to the filler bowl, valve 'A' is opened and filling commences. By means of a vent tube the gas within the container is expelled, the rate of flow of liquid into the container being proportional to the rate of flow of gas displaced. When the liquid reaches the vent tube it will fill the tube until the pressure within the vent tube equates to the filling tube pressure. When this equilibrium condition is achieved, the liquid flow stops and the filling valve can be closed. As the container is lowered from the filler bowl the liquid within the vent tube will drain back into the container. Small amounts of liquid are left in the vent tube due to surface tension effects that are dependent on the characteristics of the liquid being filled. The cycle is shown in Figure 7.10.

This process requires certain standards for the container as well as the filler. A bottle must have a standardised neck finish to allow it to seal effectively at all times with the filler bowl. It must also have sufficient top load to withstand the forces involved during the filling process. Most modern bottle fillers now lift

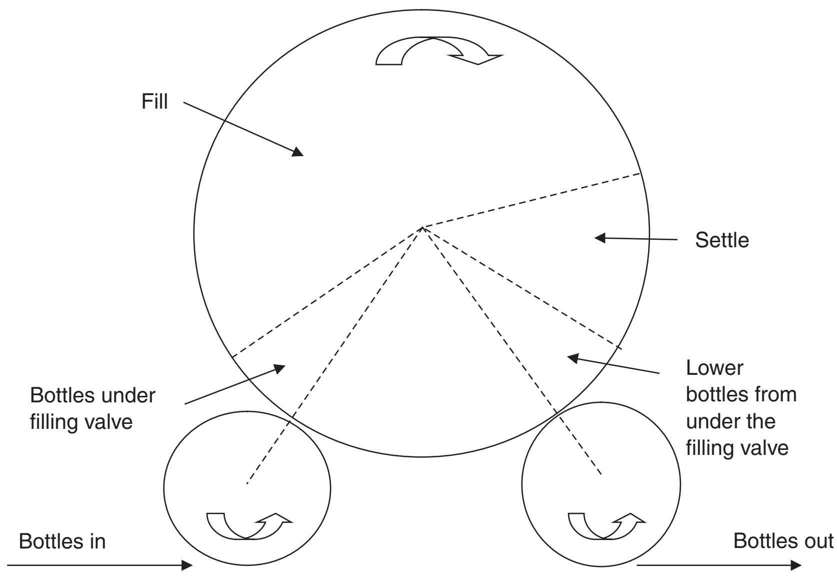

Figure 7.10 Gravity filling cycle.

PET bottles by the neck to overcome possible deformation problems during the process. This also allows light weighting of the bottle for both environmental and commercial reasons. To achieve commercially acceptable filling speeds the majority of fillers are rotary. Bottles are fed into the filler by conveyor to an infeed worm and star-wheel in single file. This mechanism incorporates a clutch-operated bottle stop which, under normal conditions, rotates freely. Should the filler be stopped for some reason, the bottle stop will engage automatically. From the star-wheel, bottles are fed to a bottle-lift stirrup sited below an individual filling valve and lifted by the neck to the seal with the filler bowl. These filling valves are sited at equal intervals around the base of the filling bowl. The filling bowl itself is annular to allow all the feed pipes and central drive to function. The rotation of the filling bowl gives rise to a centrifugal force, and the flow through apertures into individual valves is very complex. The flow paths are short, such that the flow through a valve is not fully developed. The closer the filling valves on a small-pitch filler, the more the flow into individual valves is influenced by the flows into adjacent valves. Consequently, design is based on experimental results and experience with fillers in practice rather than using computational fluid mechanics.

For each filling valve a centring bell acts as the seal for the bottle, this seal being made of food-grade rubber. The hardness of the rubber, any defects on the bottle sealing surface, the state of the sealing rubber and the sealing pressure all affect the performance of the seal, which is critical to good filling. Each filling valve is opened and closed by an actuating lever from cams fitted to the stationary filling frame. The valves are actuated when the filling bowl, and hence the valve-actuating lever, pass the cam. If no bottle is in position, this is sensed because the centring bell has not been lifted into position, and no filling takes place. The actual flow through a valve is complex. For example, there could be some 100 valves sited around the base of the filling bowl. The liquid has to flow through each of these orifices and then into the container via an annulus between the vent tube and the side of the valve. A spreader is usually fixed to the vent tube to deflect the product on to the container side wall to minimise turbulence conditions in the liquid within the bottle. Each bottle has a defined filling level, which should be as close to the sealing surface as possible commensurate with the expansion of the liquid during its potential lifecycle as a result of temperature variations. Once filling is completed, a short settling period is allowed before the bottles are lowered from the filling bowl and passed to the capper to be sealed.

7.8.2 Counter-pressure filler

The process above forms the basis of counter-pressure filling of carbonated liquids into a container. To achieve gravity filling the pressure within the filling bowl and the container must be equated. A typical process cycle is shown in Figure 7.11. Once the container is sealed to the filler bowl, the gas valve is opened and the gas within the filling bowl headspace flows under pressure into the container displacing air at atmospheric pressure. In modern fillers this air is vented to the atmosphere, but on older fillers it is often vented to the filler bowl headspace, causing the problems associated with gas mixtures discussed previously. In practice, however, since air is lighter than carbon dioxide, the majority of the air can be vented off. The example shown in Figure 7.12 illustrates how evacuating the air from the bottle prior to filling can reduce the air content. It can be seen that it is not feasible to completely eliminate all the air within a bottle prior to filling. However, the air within the bottle can be minimised if it is evacuated away from the filler and not into the filler headspace.

Once the pressures are equated, the gas valve is closed and the liquid valve opened. Flow commences, stopping when the pressures are equated. A short settling period is then allowed before the liquid valve is closed and the gas within the container headspace is snifted (vented) off. This settling period, and subsequent snifting are required because otherwise, when the container is lowered from the filler bowl, the pressurised gas within the headspace will be exposed to the atmosphere. This would result in severe fobbing of the product

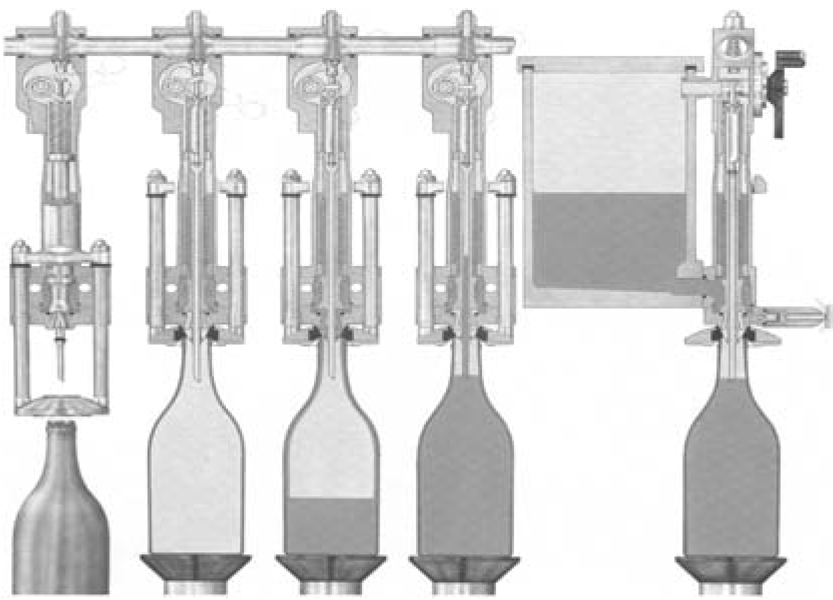

Figure 7.11 Counter-Pressure filling cycle.

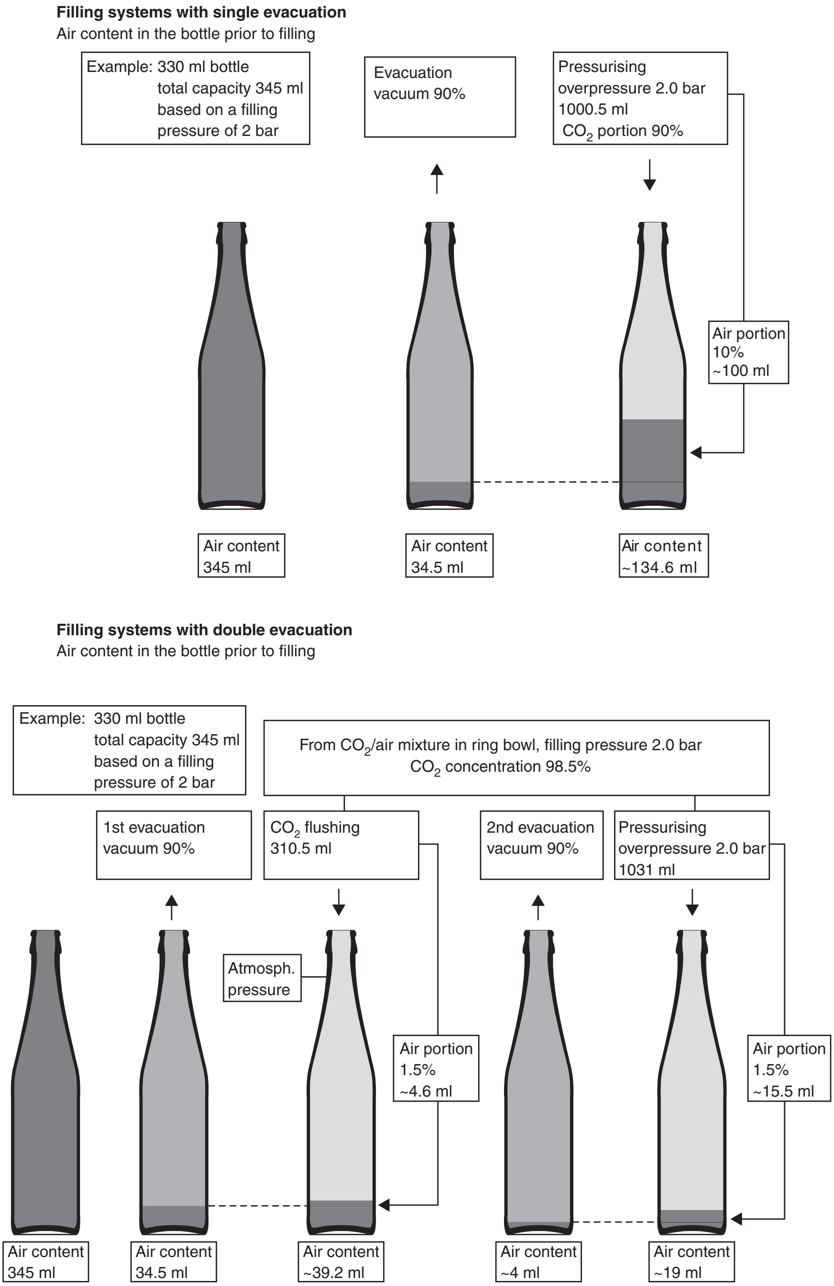

Figure 7.12 Bottle evacuation.
Source: By courtesy of Krones (UK) Ltd.

within the container until equilibrium conditions were reached. To overcome this potential problem, the pressure within the headspace is gradually reduced by snifting the gas off externally to the filler. Often this process is repeated several times in short bursts allowing a short rest period between snifts. The rate of flow of the gas is controlled by the diameter of the snift valve orifice. Snifting is the most difficult part of the process to control, especially if the product itself is lively – cream soda, say. Care when snifting off to atmosphere has to be taken as the snifted gas often contains a product mist, which can cause contamination as it condenses on the surface of the filler and surrounding plant. A conventional filling cycle is illustrated in Figure 7.13. A diagrammatic representation of how bottles are fed through the system using an infeed worm and star-wheels is shown in Figure 7.14.

Considerations during the process include ensuring that filling is as quiescent as possible, otherwise 'fobbing' will occur. The term 'fobbing' is generally applied to a carbonated product that is still 'lively' and has not reached a state of equilibrium. Fobbing can be induced by excessive agitation during filling and too fast a snifting process. For this reason great care must be taken to ensure that the process is under control at all times. There is an increasing tendency for carbon dioxide to come out of solution with higher temperatures as well as with greater volumes of carbonation. Most modern fillers can operate at up to 22°C, though any liquid temperature above 20°C will tend to increase the risk

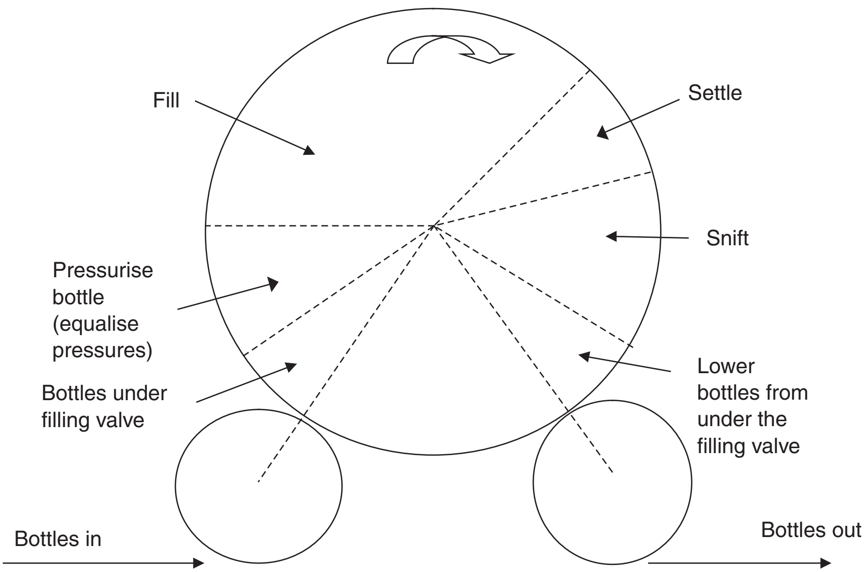

Figure 7.13 Counter-pressure filling cycle.

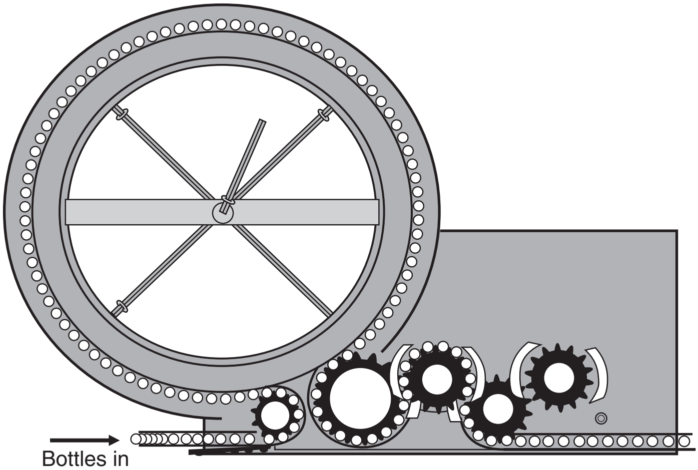

Figure 7.14 Filler layout showing infeed worm and star-wheels.
Note: Each circle represents a container.

of fobbing. Most fillers to operate at around 14°C, which has been found to be a good compromise temperature when considering energy consumed to cool the product and the efficiency of the filling process, as the lower the temperature, the more easily carbon dioxide will stay in solution. Filling down the insides of a bottle will reduce the level of fobbing as will a carefully designed container. The former is achieved using swirl-type valves that impart a tangential force to the liquid, thereby forcing it to the sides of the bottle. Any discontinuity along the side wall of a container will cause the liquid to 'jump' off the inner surface side wall and fall directly on to the product in the base of the bottle, thus increasing the level of agitation and hence causing fobbing. Likewise, too high a shoulder on a bottle will not allow the liquid to flow along the inside walls as early as possible, giving rise to increased agitation of the liquid and loss of carbon dioxide from solution. In addition, the shape of the bottle shoulder can exaggerate any inconsistency of fill level control, especially with tapered bottle shoulders. Bottle top load is a critical factor. On older fillers that use bottle-lifters whereby the bottle is lifted by the base, any bottle with a low top loading ability will tend to buckle during the snifting process, thereby causing some loss of product, especially if fobbing is induced, and hence a reduced fill level in the container. When filling carbonated products with fruit cells it is usual to employ siphon-type valves, which allow a smoother passage through the filling valve, though with a loss of performance and a requirement for a lower carbonation level.

The choice of a filler depends on the number of bottle sizes being considered for use on it. For example, if 3 l PET bottles are to be filled, a 126 mm pitch between filling valves is required. If only small bottles up to 500 ml are to be filled, then a 70 mm pitch will suffice. This has a direct effect on the size of the filler and its footprint on the factory floor. Unless the filler bowl level is kept within tightly controlled limits, pressure head variations will affect the rate of flow into containers. Systems such as that shown in Figure 7.15 need to be employed. Wherever possible there should be minimal contact between any instrumentation and the product. Conventional float valves should be avoided and simple capacitance probes, which are easily cleanable but small and very effective, should be used. It is not uncommon in older fillers to only have one float valve. This often gives rise to filler bowl flooding, which may lead to inconsistent fill level and poor counter-pressurisation of the container pre-filling.

A filler is usually constructed of 316 stainless steel to ensure all surfaces can withstand chemical attack from both the product and cleaning processes, as well as being easily and effectively cleanable. The annular bowl is supported on a central frame, which also includes the drive motor and pipework supports. Each valve is individually piped to the central frame, connected via a rotary valve. Most drives are now invertor controlled through a process logic controller (PLC), giving a wide range of possible operating outputs. Valve design has improved greatly over the last few years, so that swirl valves now have sufficiently large passageways to enable many fruit- or cell-based drinks to be processed, instead of using the slower siphon valves, with their tendency to give rise to fobbing. Even so, owing to the nature of the design of counter-pressure fillers, the vent tube will always contain some product, which will

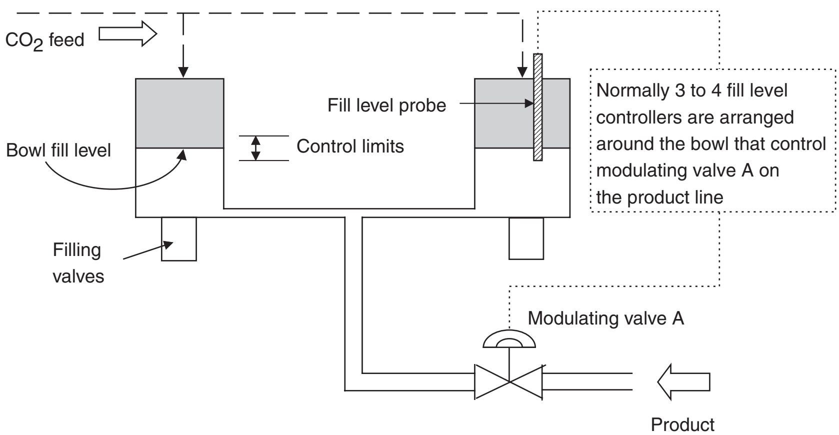

Figure 7.15 Filler bowl level control.

inevitably be carried across to the next fill cycle unless it is blown out prior to the new cycle commencing. The cutoff for the vent tube occurs at the full filling rate, and so is never perfect because of the momentum of the liquid product during filling. The traditional filling valve with springs can thus be easily contaminated since the springs are submerged in the product itself. This can cause cross-contamination from one product to another following a flavour change unless care is taken to ensure an effective cleaning regime.

7.8.3 *Other filler types*

Many modern types of filler are electronically controlled. They fall into two main categories, volumetric and capacitance probe fillers. The capacitance probe filler uses a capacitance probe to detect the point at which filling should cease. The probe is sited in the centre of the filling aperture of the valve, and is calibrated along its length to detect when the fill level is being approached and then progress until the actual shut-off at the end of filling. This sophisticated type of filler allows the initial fill through the swirl valve to be slow, to minimise the effect of fobbing, then fast until the fill level is approached. As the fill level is approached the probe senses this and allows the fill rate to be reduced, allowing a very accurate end-of-fill cutoff to occur. The use of electronic valves within the system ensures that the whole operation can be controlled electronically. The snift can be more finely controlled than in counter-pressure fillers by applying a small back-pressure for the initial snift, followed by a normal snift operation to atmospheric pressure. Owing to the fact that the capacitance probe filler is more complex than traditional fillers, it does require a fuller understanding of the physics of the operation to achieve the best results. A typical electronic probe filler is shown in Figure 7.16.

The volumetric filler is also electronically controlled, ensuring an accurate dose of product to each container. For PET bottles, which ‘creep’ under carbonation pressure, there is the drawback that they will have differing fill levels over a filling cycle due to the fact that no two bottles are ever exactly identically blow-moulded. With light weighting, this problem is exacerbated.

Two main types of volumetric filler exist. In the first type, a predetermined volume of liquid can be dosed using either a magnetic inductive volumetric flow meter or a mass flow meter. Alternatively, measuring cylinders can be used that incorporate a level probe. The product is fed to a set level in the metering cylinder, at which point the flow valve is closed off from the supply tank. A typical can filler operating at 1,500 cans of 330 ml capacity per minute will fill as per the supply contract within ±2.5 ml. However, a standard deviation for a volumetric filler as low as ±0.58 ml has been quoted. As with the probe filler, these fillers are fitted with electro-pneumatic valves, enabling accurate control of the

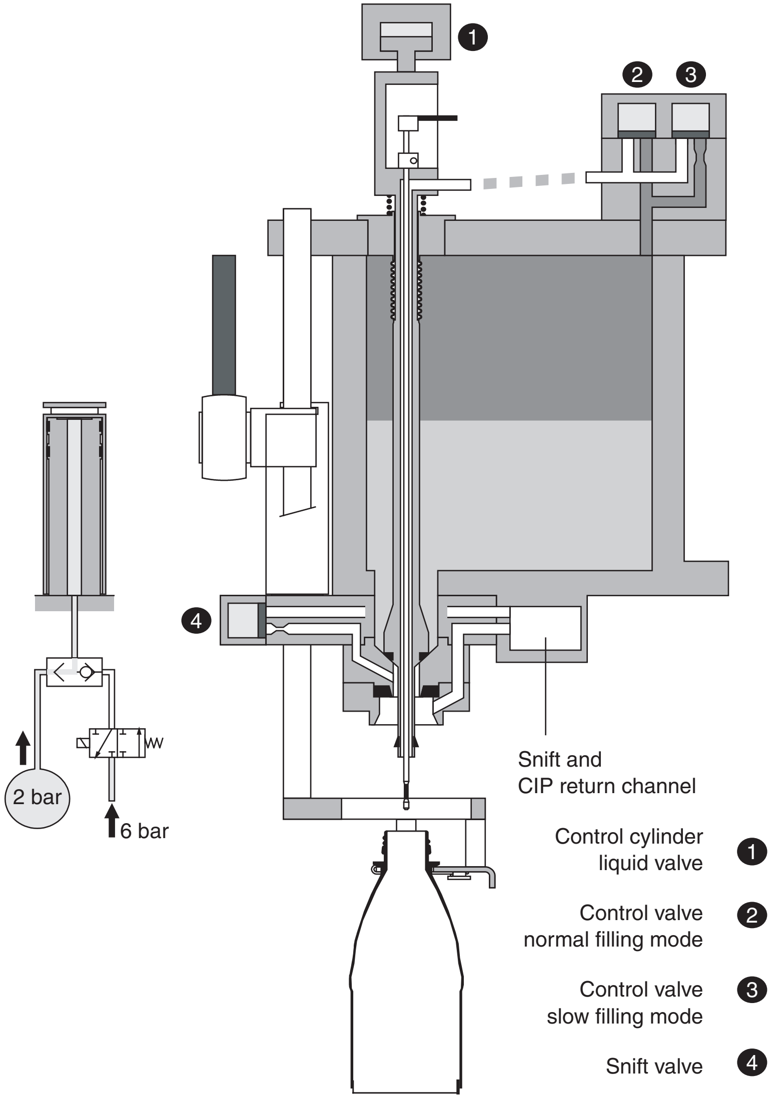

Figure 7.16 Electronic probe filler.
Source: By courtesy of Krones (UK) Ltd.

process. Care has to be taken to ensure these valves are kept clean and in good condition as they are critical to the operation. Any sluggishness in a valve's operation will affect performance. Two typical volumetric filler operations are shown in Figure 7.17.

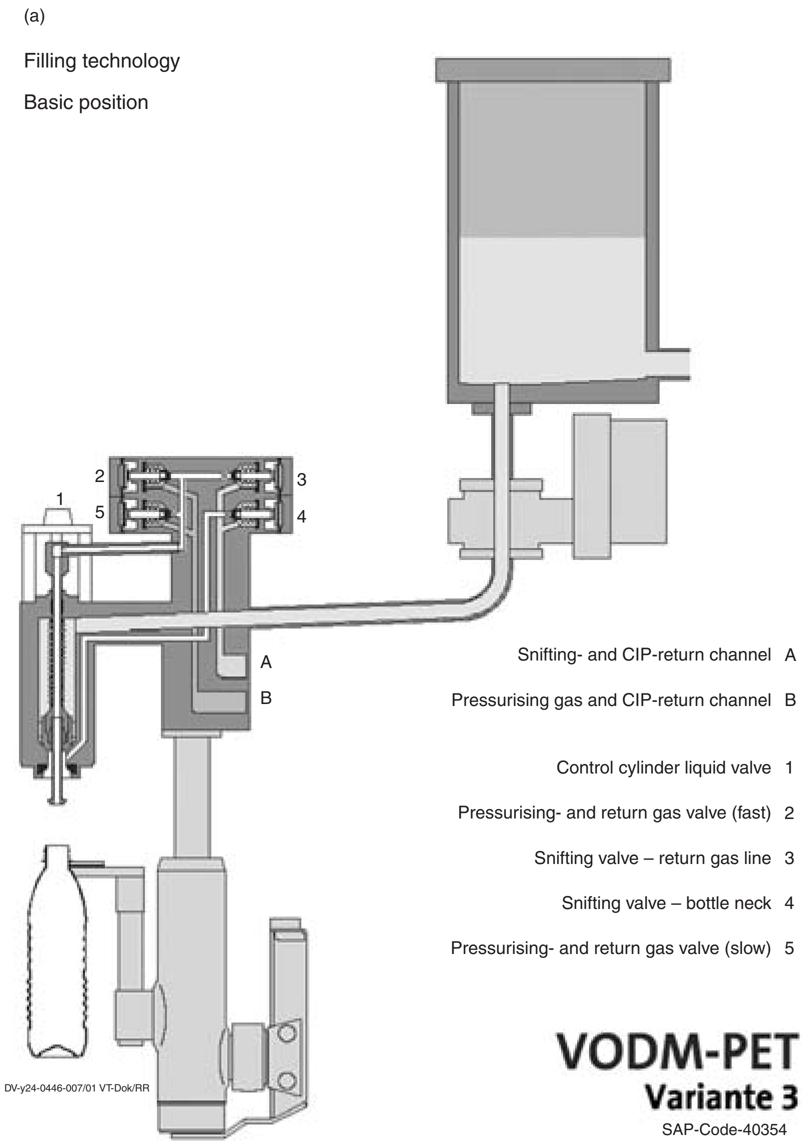

Figure 7.17 (*Continued*)

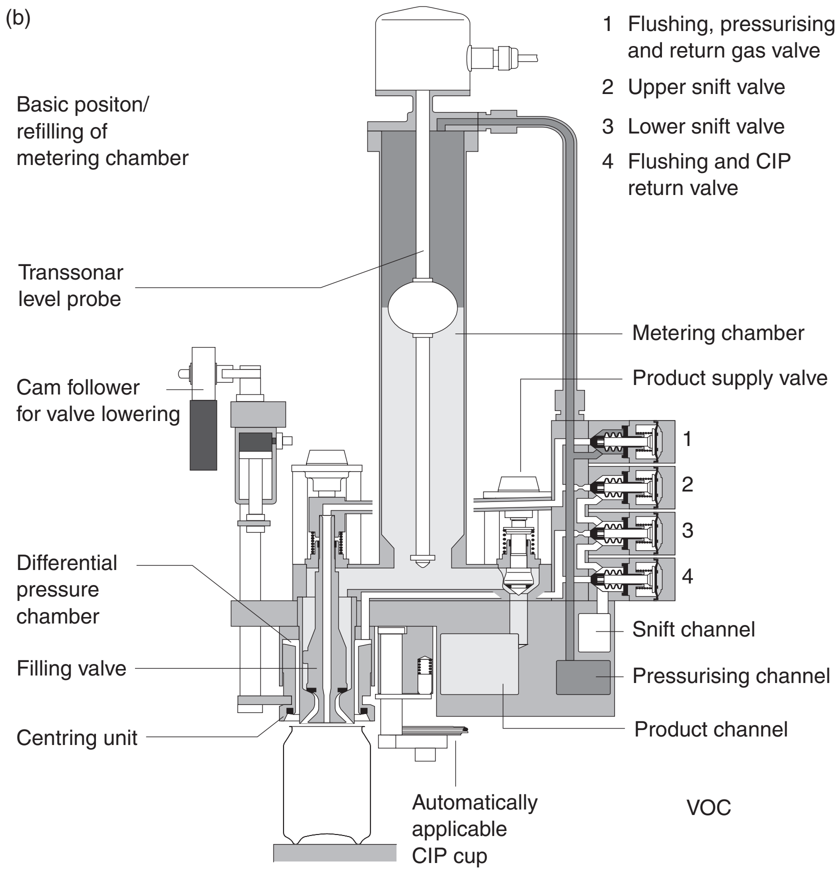

Figure 7.17 (a) Volumetric filler using flow meters. (b) Typical volumetric filler using a measuring cylinder.
Source: By courtesy of Krones (UK) Ltd.

7.8.4 Clean-in-place systems

All modern fillers are designed for clean-in-place (CIP) to ensure the sterility of the system. The CIP process operates from a centrally located system that is piped to the filler. A return cycle to the CIP set from the filler is included, the temperature of the return liquor being sensed at the filler outlet. Specifically designed CIP cups are attached to each filling valve to allow the CIP process to take place entirely through the filler bowl and filling valves and all associated pipework at sufficient velocity and contact time to ensure effective cleaning is achieved. A typical operating cycle would be

(1) Rinse the filler and filling valves for a set time using rinse water to drain. This removes particulates. The rinse water used is often the final rinse water from the preceding CIP operation so as to conserve water.
(2) Circulate hot caustic soda solution (*c*.1.5% w/v) at 80°C (as recorded at the filler outlet) for some 15 min. The caustic soda is returned to the hot caustic tank through filters.
(3) Circulate hot water at around 75°C for 15 min to allow the interface with the caustic to flow to the drain. A conductivity probe is included within the circuit to ensure that all the caustic lye has been removed. The hot water is recirculated to conserve energy.
(4) Use cold water as a final rinse.

Careful records of the CIP process need to be maintained to ensure the process used has been effective. A typical CIP process is shown in Figure 7.18.

7.9 Process control

Process control is the essence of modern factory systems. It is imperative at all times to have the process fully under control and for full records to be kept.

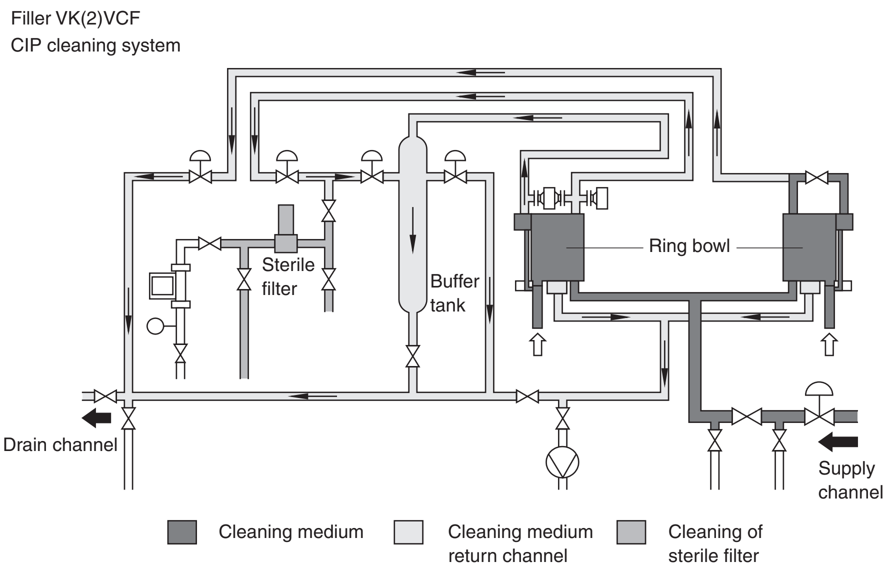

Figure 7.18 Typical CIP system.
Source: By courtesy of Krones (UK) Ltd.

Online instrumentation and feedback control is the key to a successful operation. The syrup premix is produced as a batch offline. All the weighed ingredients are logged on a computer-controlled weighing system that can feed back data to the central factory system. The level of de-aeration of the water can be continuously logged using an online probe, and the water quality itself must be regularly analysed. The treated water is monitored for turbidity problems online, with automatic shut-off should a problem arise. The carbon dioxide and all materials delivered should have a certificate of conformance to an agreed, signed specification. The premix, water, acid and sugar, if required, are fed by flow meter, with online Brix control feedback, either to a bulk vessel or direct to the carbonator and are checked against an agreed recipe within the PLC of the dosing system. At all times, records need to be computerised and backup copies kept. Automatic shutdown occurs should a problem be found by the online instrumentation. The effectiveness of the carbonation process can be detected online by taking regular samples and checking pressure and temperature against the specification. The feedback control system will regulate the process within agreed limits. The filler itself is fully PLC controlled, often using a touch-screen control system. This allows production to be carried out with Brix and carbonation levels controlled within ±0.05 Brix and carbonation values of the set-point. It should be remembered that the filler is just part of the filling line process. This is illustrated in Figure 7.19 for a typical PET bottling line. The line is usually controlled by the blow moulder, but for small bottle lines silos are used

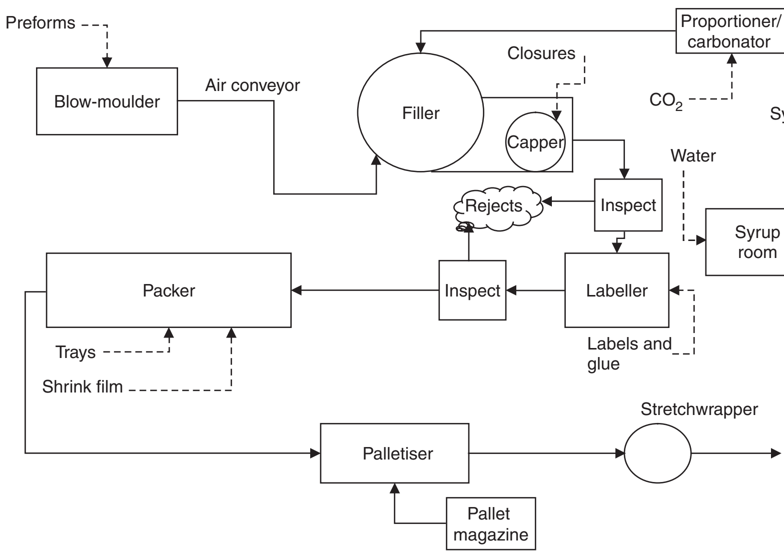

Figure 7.19 Typical PET filling line.
Note: For high-throughput items, display trays can be used.

to buffer the bottles and allow the blow moulder to operate as efficiently as possible. The overall line efficiency is the multiplication of all the individual unit efficiencies comprising the line. Even the conveyors between the machines have a mechanical efficiency. However, if sufficient bottle accumulation between machines is provided by the conveying system this will improve line efficiency by providing a buffer to overcome short duration machine stoppages. Such accumulation systems are of three minutes or more in duration and are designed to overcome typical machine stoppages of the order of 30 seconds.

7.10 Future trends

It is difficult to imagine what the next major step forward in beverage technology will be. We already blow-mould PET bottles direct online to the filler via an air conveyor. These bottles are manufactured and filled in just over 2 min, unless a silo system is used to store the bottles and act as a buffer between the blow-moulder and filler and hence increase operational output. Such silos, which tend to be used for PET bottles up to 500 ml capacity, also offset the usual efficiency imbalance between the blow-moulder and the less efficient filler. One obvious trend is the rationalisation of bottle shapes and sizes, such that common diameters, heights and fill heights are used. This reduces the number of size changes required on the blow-moulder and filling line, thus eliminating setup and startup time losses. Further light weighting of PET containers will continue, especially as the cost of PET resin is directly proportional to oil prices. This means that the filling process needs to handle flimsier containers. This will preclude many of the older fillers from still using full bottle-lifts rather than not neck handling. Rapid size changes will become even more important, as will the dedication of filling lines, wherever possible, to one bottle size and if possible one product. The light weighting of cans will continue, though at a lower rate than the major reductions seen in the past 20 years. This will imply more careful control of the pressure compensation to ensure effective sealing of the can with the filling bowl without any can crush occurring. More processes will become PC rather than PLC controlled, thereby facilitating easier programming. The snifting operation will incorporate more sophisticated instrumentation to minimise the risk of fobbing and will be carried out in as short a time as possible to ensure the filling-cycle operation itself is optimised. More flexible operations will be required as marketing staff try to gain increasing market share for their products. This will see the emergence of ‘cottage industry’ lines as separate cost centres within large dedicated-line factories, thus allowing the true cost of producing minor flavours and specialised containers with short run lengths to be determined. The real flexibility will come from packaging changes rather than improvements in carbonation practices and filling techniques. The increasing sophistication of consumers will require even more aseptic and semi-aseptic filling operations. For non-carbonated products fully aseptic processes are now operational whereby the product being filled does not come into contact with the filler bowl. This situation cannot be achieved for carbonated products; so the aseptic state can only be approached but not actually achieved with current technology. It is in this area that further developments will take place, driven by consumer demand.

Further reading

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8 Processing and packaging

R.A.W. Lea

8.1 Introduction

This chapter covers juice extraction, blending, processing, factory layout, filling and packaging. The purpose of processing and packaging is to produce a product that is wholesome and refreshing in a pack of convenient size for the consumer. The topic is extensive and so only the main points are covered in this chapter. However, equipment suppliers provide a good source of free information and their names can be found from trade magazines, trade associations, etc.

The designer of a blending/filling system must have a full understanding of the process: its limitations, the raw materials, the packaging materials and their levels of contamination and the means of controlling the contamination to enable wholesome product with adequate shelf life to be produced.

8.2 Juice extraction

The details of the juice extraction process will depend on the fruit, but this section outlines a typical method. Some processors size the extraction plant to handle all the juice they will require for the year within the harvesting period; others will freeze or otherwise store the fruit and extract juice as it is required on a much smaller plant.

The fruit is normally delivered in large containers (or lorry loads) to a reception area where it is washed and screened to remove foreign bodies. The fruit is then milled, heated, mixed with enzyme to break it down and held for a period (enzyming is a time/temperature process). The juice is separated using either centrifuges or fruit presses and processed for aseptic storage as single-strength juice (see Section 8.4) or concentrated by evaporation of water and stored. Many soft drinks are made from bought-in concentrates produced by a specialist supplier.

8.3 Blending

There are three main blending routes, which can be used individually or in combination to form a product. Traditionally this is carried out in the 'syrup room' as it used to be normal to produce the drink as a syrup and dilute it prior

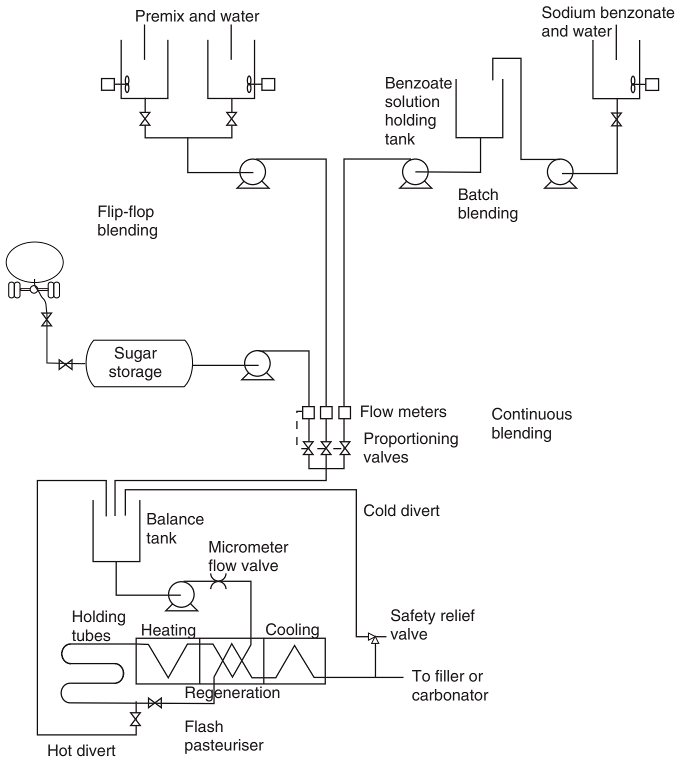

Figure 8.1 Typical process flowsheet.

to filling (before that it was usual to fill it as two separate phases: syrup and carbonated water). The three routes are

(1) batch
(2) flip-flop
(3) continuous.

Figure 8.1 shows a typical combined process flowsheet.

8.3.1 Batch blending

The ingredients, usually powders or liquids, are mixed together in a large batch tank. Bulk ingredients can be metered or weighed from storage. The storage tanks or silos are filled by bulk tanker. The minor ingredients are often combined in a smaller premix tank prior to addition to the main mix tank. Some of the ingredients cause an endothermic or exothermic reaction on solution and may need heating or cooling to maintain temperature. There is a variety of stirrers and mixers available for the mix and premix tanks, ranging from simple propellers to specially designed high-shear mixing heads.

Powders are dissolved either directly in the main mix tank or premix tank or indirectly using a vortex-type mixer (Figure 8.1) where powder is dropped into the vortex of a horizontally mounted pump head recirculating the fluid from and to the batch tank. Some specialised versions of this mixer can handle very viscous blending applications (50,000 cP or more).

8.3.2 Flip-flop blending

This is really a special case of batch blending. In batch blending systems, once the batch has been made and checked for quality it is pumped into a holding tank before being sent to the line. In flip-flop systems a batch is made in one mix tank and then fed directly to the line while the next batch is made in another mix tank. The second batch is sent directly to the line when the first batch has finished: batches flip-flop between the tanks. Flip-flop batching is often used for premixing in batch blending or when mixing time is tight (i.e. the time taken to pump the batch out of the mix tank to storage is too long); however, batch mixing systems are generally cheaper as only one mix tank is required.

8.3.3 Continuous blending

In continuous blending, streams of raw materials and premixes are blended into one another on a continuous basis. The proportions are closely controlled either using a system of positive displacement proportioning pumps (either the stroke or speed is accurately controlled) or using flowmeters to control the throughputs of each stream via speed-controlled pumps or proportioning valves to throttle the flows. The metered liquids then pass through an in-line mixer to complete the blending; this mixer can be static (a series of blades creating turbulence) or powered to create high shear. Continuous systems are used when the number of streams is limited; for example, water, sweetener, premix 1 and premix 2 (assuming stable premixes can be made). Systems with more than eight streams are unusual because of difficulties in accurately controlling the streams and validating the process.

8.4 Processing

In this section, we consider the means by which the product is rendered microbiologically secure. This is usually accomplished by the addition of heat; although chemical means are also used in some circumstances (i.e. dimethyldicarbonate, which breaks down into water and carbon dioxide and is thus safe for the consumer). There are five main processes for juices and soft drinks:

(1) flash pasteurisation
(2) hot filling
(3) in-pack pasteurisation
(4) aseptic filling
(5) chilled distribution.

The choice of process depends on the level of microbiological contamination of the raw materials and packaging, and the ability of the product to withstand growth of micro-organisms (e.g. preservative levels and sugar content). Also of great importance is the ability of the product to withstand heat.

The theory of pasteurisation is very simple: the product is heated and kept at or above the pasteurisation temperature for a certain time. It is the responsibility of the processor to specify the temperature and time. For pasteurisation to be microbiologically safe, it is essential that the correct conditions are maintained during the complete period of operation and that reinfection is prevented.

8.4.1 Flash pasteurisation

The raw juice is normally passed through a balance tank (or feed tank) before being fed to the pasteuriser.

The drink is generally heated by hot water in a plate or tubular (spiral) heat exchanger to the desired pasteurisation temperature and held at that temperature for the specified time in a holding tube before being cooled to the filling temperature (usually ambient) using chilled water. Normally, flash pasteurisers have a regenerative section (see Figure 8.1); this is an energy-saving feature whereby the incoming raw product is initially heated by the hot product returning from the holding coil, which in turn is cooled. The energy used for heating is regenerated. To prevent reinfection the equipment must be sterilisable using culinary steam or hot water and be impervious to microbiological spores (i.e. there should be no risk of contamination from unpasteurised product or product trapped in ‘dead legs’ or equipment that is not bacteria tight, e.g. valves and pumps). In some countries regulations require that pasteurised product is always at a higher pressure than the raw product or heating or cooling medium on the other side of the plates. This is usually achieved using a boost pump between the incoming regenerative section and the heating section.

The equipment should be designed such that the treatment time is correct. This is usually achieved by controlling the flowrate and passing the product through a holding tube of known volume. The flowrate and temperature (at the end of the holding tube) are critical to the process and should therefore be monitored.

In the basic system described above, if the temperature drops below the pasteurisation temperature or the flowrate exceeds that for the correct holding time there is no other choice than to shut down the process and to clean and resterilise the equipment before production can be restarted. The consequences of this may be limited by diverting the flow of insufficiently pasteurised product back to the balance tank: forward flow may be resumed once correct conditions are restored. The divert valve should be placed sufficiently downstream of the temperature monitoring probe that the system response time (probe, controller and valve) is less than the time taken for the unpasteurised product to reach the valve.

Pulsations and vibrations should be kept to a minimum (e.g. pumping and valve switching) because they reduce the life of plates by mechanical damage leading to fatigue failure. To ensure product security, plates should be crack tested on a regular basis (annually).

Pasteurised product is either sent to a proportioner and diluted to the desired strength (if produced as a syrup) and then carbonated and mixed with carbon dioxide or sent direcly to a filling machine; excess product is diverted (cold divert) back to the balance tank of the pasteuriser. The system is typically sized for a flow of 5–10% in excess of that required; the positive forward flow maintains the sterility of the system so that bacteria, etc. cannot pass up the cold divert line against the flow.

8.4.2 *Hot filling*

In hot filling product is heated (in a heat exchanger), sent to the filler hot and filled into containers. The containers are closed and held at or above the required temperature for a specified time prior to being cooled, usually in a tunnel with water sprays. In this system not only has the product been heat treated but so has the container.

A typical regime would be to heat the product to 87°C (monitored at the heat exchanger) and send it forward to the filler bowl. The temperature is monitored in the filler and should it fall below a preset temperature, say 85°C, filling is inhibited, the filler emptied and its contents replaced with fresh hot product. Emptying the filler is often by means of a pump; care is needed in the selection of the pump as it is operating at high temperature, increasing the risk of cavitation (localised boiling in the pump suction due to reduced pressure). The product is then filled into the containers, where the equilibrium temperature is typically 83–84°C. If glass containers are being filled, hot water at, say, 60°C should be used to rinse the containers thus limiting the effect of thermal shock of hot liquid on cold bottles. The containers should then be held on a conveyor for the predetermined time, say 2 min, at above 80°C. This time and temperature are the microbiological control requirements and are thus important parameters that should be specified (the other values are not the controls for the process; however, the filling temperature and conveyor speed are the engineering parameters that are controlled to achieve the specifications). For some applications, it may be necessary to tilt the product so that the hot liquid wets the cap and neck area of the container. However, this is not often required: the head space typically reaches 75°C with the above regime and this temperature is normally adequate. Even with gable-topped cartons, in which there are four triangular areas surrounded by ambient air, if these areas do not reach an elevated temperature, experience has shown that product is not put at risk (this has been a challenge tested by the author). After the holding section, the containers are cooled to below 30°C (to limit thermal degradation).

8.4.3 In-pack pasteurisation

Generally, in-pack pasteurisation is the severest and also the most microbiologically secure form of heat treatment. The completely filled closed pack is put into a tunnel pasteuriser. The treatment is via water spray at various controlled temperatures. The pasteuriser is divided into zones. First there are a heating zone and a 'superheat' zone; these raise the temperature of the container and product to the desired temperature. Next there is the pasteurising zone, where the product is held at the pasteurising temperature for the specified time. Finally the product is cooled to below 30°C. To verify correct pasteurisation a detector is sent through the machine regularly. This consists of a temperature sensor mounted within a 'dummy product' and a recorder to produce the time–temperature profile.

A special case of in-pack pasteurisation is a retortable process in which the packs can be treated to elevated (above 100°C) temperatures in a retort and cooled.

8.4.4 Aseptic filling

The processing for aseptic filling is a special case of flash pasteurisation (often using a higher temperature profile). The plant is not equipped with a hot divert as this allows the forward flow to stop when the divert is actuated. Should pasteurising conditions be lost (flow or temperature) then the plant is stopped and resterilised. Great care is needed with any equipment downstream of the holding tubes as it must be microbiologically impervious to maintain asepticity (e.g. valves should have steam barriers on shaft seals) and should be kept to an absolute minimum.

The aseptic processor either feeds the filler directly, with excess flow returning to the balance tank (and positive flow maintained under all circumstances), or feeds directly to an aseptic storage tank, which acts as a buffer between the processing and filling operations.

For successful aseptic filling, clean containers, clean product, clean head space and clean closures need to be brought together in an environment that prevents recontamination. This operation normally takes place in an enclosure overpressured with sterile air. The air is sterilised using either heat or filtration of 0.2 μm. (The filter will need sterilising periodically using either steam or chemicals.) It is recommended that the filling equipment is located in a separate room that is in turn overpressured with clean filtered air (typically 2–4 mm water column) and has 10–15 air changes per hour. The air quality is defined in terms of the number of particles per cubic metre, that is, a class 100 room would have 100 particles per cubic metre.

The cleaning regime specified for the containers will depend on their initial level of contamination. Normally heat (steam or hot air) or chemical means (hydrogen peroxide or oxonia) are used; sometimes these are used in combination with ultraviolet radiation.

Laminate board packs are made either from flat reels of material or from preformed blanks. These systems are generally ‘tied’, that is, the laminate supplier also makes the filling equipment. The fillers are well proven microbiologically and do not need to be placed within a special environment.

Bottle systems are more varied, whether for glass, polyethylene terephthalate (PET) or other plastic. Bottles are rinsed with oxonia solution and then sterile water prior to filling. The filler is generally of a non-contact type (it does not touch the bottles) and product is either weighed in or measured volumetrically. Caps are also chemically sterilised (unless a foil closure is used) and applied on a capper monoblocked with the filler, enclosed in a high efficiency pure air (HEPA) filtered enclosure. The filler and final rinser are in a class 100 room and the operator wears full protective clothing to prevent infection of the product.

There are some more specialised systems for PET bottles, cans, other plastic bottles, form–fill–seal packs and returnable PET or glass bottles for still and carbonated drinks (generally high acid). These are material-dependent solutions and each arises from the limitations and properties of the materials and the way they are formed into containers.

PET bottles are made from resin chips in a two-stage process (even if made on one machine). A preform is injection moulded and is sterile when formed; this is then blown into the final shape. If the machine is enclosed in an HEPA-filtered environment and the bottles blown with sterile air, the machine produces a sterile bottle. The bottle can be transferred to the filler within an enclosed sterile air conveyor and filled without further treatment, thus eliminating the need for rinsing and chemical treatment. Carbonated products can be filled if the carbon dioxide is sterilised.

Cans can be sterilised easily with super-heated steam or hot air as they are stable above sterilising temperatures.

Other plastic bottles can be blown on a sterile bottle blower and be supplied closed (i.e. material seals the bottle above the screw thread or neck). The bottles are rinsed on the outside before entering the aseptic filling zone, the excess material is cut off and the bottle filled. (This process is not suitable for PET bottles because of the need to form the screw thread as part of the injection moulding process.)

Form–fill–seal packs are also suitable for aseptic production. The pack is blown aseptically in a mould and filled through the soft neck. Once filling is complete the fill tube is retracted and the neck sealed with heat and pressure.

Returnable bottles are generally subjected to a three-part process. Bottles are washed in a bottle washer, as they would be for non-aseptic production; however, the final rinse is with hot water, which has an initial bacteriocidal effect. The bottle rinser and filler are enclosed in an HEPA-filtered chamber; the rinser uses hot water, maintaining the decontamination; finally the filler injects steam into the bottle just prior to filling. The total process leads to a high log reduction in bacterial contamination, producing commercially sterile products. This process has been used for carbonated drinks.

8.4.5 Chilled distribution

If products are filled cold into clean bottles on clean (but not aseptic) fillers, stored in refrigerated warehouses and sold to the customer from chill cabinets then quite long shelf lives can be obtained. The product is generally flash pasteurised to ensure its cleanliness prior to filling.

8.4.6 Summary

Many products will go through a combination of these processes depending on the product, preservative system, preferred packaging and shelf life requirements. Some products will develop a cooked taste if exposed to a full in-pack pasteurisation and if for marketing or legal reasons preservatives cannot be used, aseptic filling is the choice. However, the capital cost of aseptic equipment is high and so it is only used for products at the premium end of the market. Some packages cannot be exposed to elevated temperatures (e.g. PET) and some are not suitable for carbonated products (e.g. laminated cartons). The choice of the packaging and process is an important technical decision and requires full understanding of the limitations of materials while still meeting the customer's need.

8.5 Control of process plant

In the last 10–15 years tremendous strides have been made in improving the control of process plant; however, fewer improvements and innovations have been made in the field of hygienic instruments. Ideally these should be non-invasive and should be cleanable in place. All controls should be defined in a functional specification of how the plant is to work. Each part of the process must be described in detail, defining its route, start, stop and alarm conditions and its cleaning procedures.

8.6 Factory layout and operation

In addition to process design it is essential to design the drinks factory such that it can be operated economically and cleanly. It is also important that its design is of minimal capital cost and consistent with production to desired quality standards.

Prior to considering the factory layout the designer must understand the risks to the production environment. These come from factors external to the plant, transmission media into the plant and internal factors resulting from operations within the plant. Table 8.1 details some examples of risk factors. Measures must be designed into the factory to eliminate or control all the risks to acceptable levels.

A sensible layout can be achieved through consideration of the main flows within the facility. Material flow, both ingredients and packaging, is important and should follow in a logical order. Clean operations should be separated from dirty operations, and wet operations from dry. Figure 8.2 shows a typical process flow for materials; as can be seen there is an area where the risk of contamination is high. Generally, unwrapped packaging or processed material and the operations of mixing, processing and filling are high risk and care should be taken to ensure appropriate levels of cleanliness. Another flow within the factory is people; they can be a source of contamination or cross-contamination from an unclean operation to a clean area. Care must be taken to ensure that people can

Table 8.1 Examples of risk factors in the production environment

External factors	Transmission media	Internal factors
Birds	People	Unpacking materials
Insects	Raw materials	Processing
Rodents	Packaging	Generation of waste
Other vermin	Pallets	People movement
Site ground	Engineering parts	Material movement
Surface contamination	Canteen materials	Air movement
Surrounding atmosphere	Other consumables	Drainage
		Cleaning
		Maintenance
		Exterior packing

only access high-risk areas and operations through adequate and appropriate routes via cleaning and changing areas. A useful tool in understanding required people movements is a bubble diagram (Figure 8.3): each bubble represents an area of the factory and they are connected by arrows representing required access for personnel. The third important flow is waste. The process flow (Figure 8.2) shows waste being generated by a number of operations. The flow of waste and its exit from the factory needs careful consideration and its flow should not cross incoming material flows to avoid cross-contamination. The ideal factory layout can be developed from these flows.

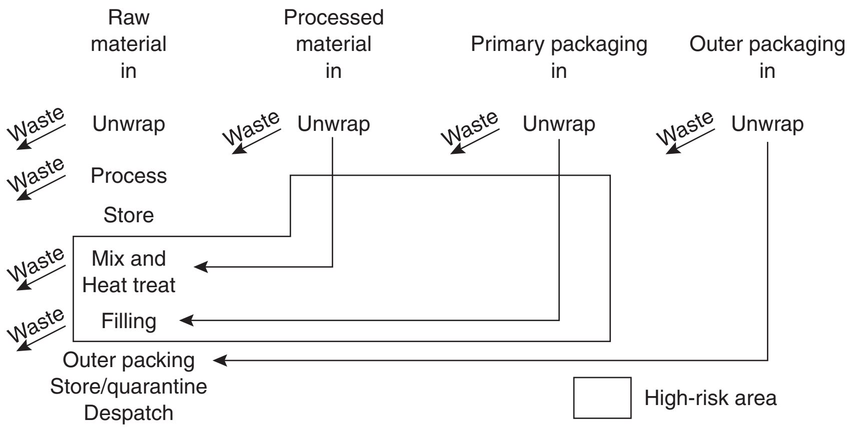

Figure 8.2 Typical process flow.

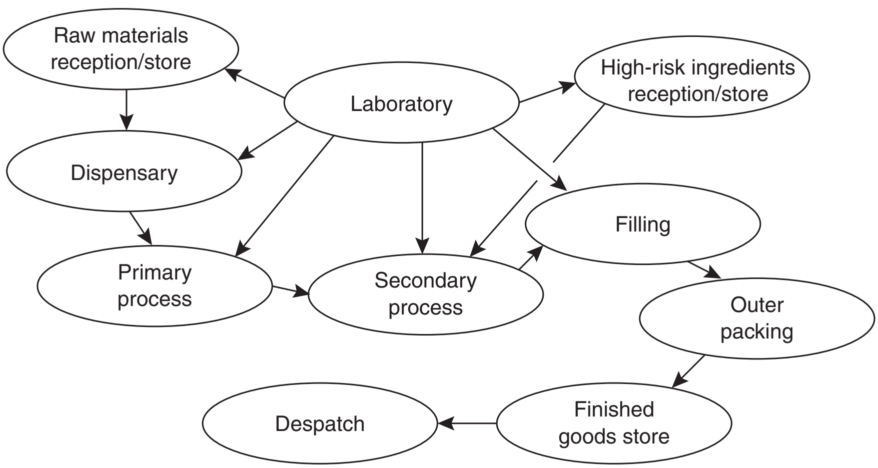

Figure 8.3 Bubble diagram.

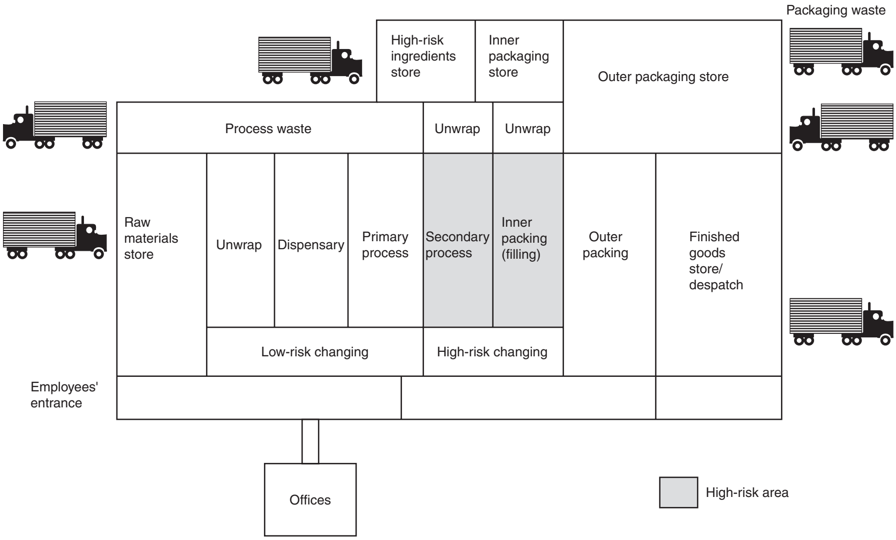

Figure 8.4 Typical factory layout.

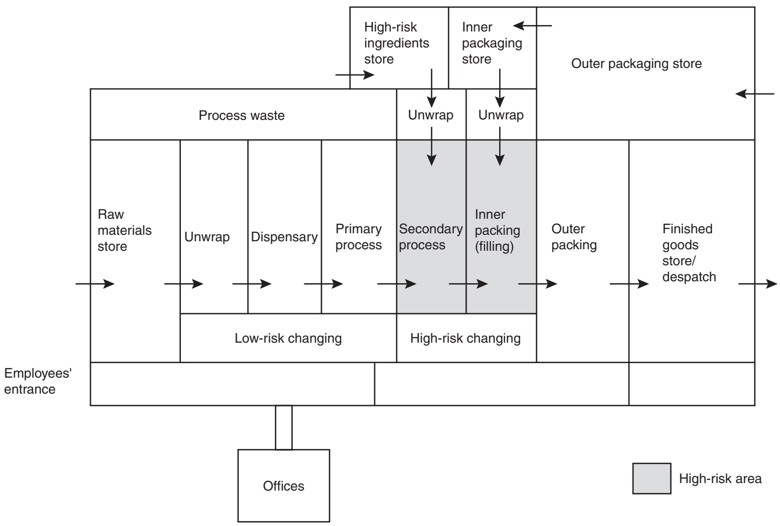

Figure 8.5 Factory layout showing material flow.

Figure 8.4 shows a layout suitable for a complex aseptic production facility; each of the operations is carefully controlled to minimise the risk of cross-contamination. The material, people and waste flows have been superimposed on this layout in Figures 8.5–8.7. In many cases this layout is too expensive and

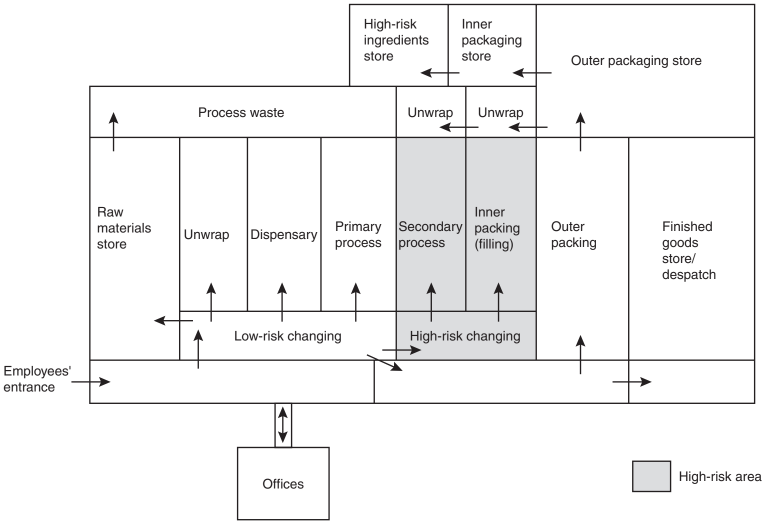

Figure 8.6 Factory layout showing people flow.

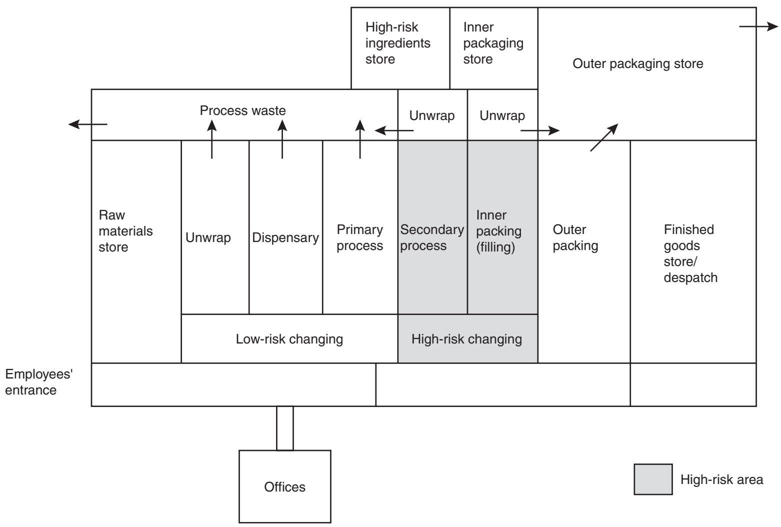

Figure 8.7 Factory layout showing waste flow.

elaborate for the production of preserved high-acid beverages and many of the separations between operations can be eliminated. However, for some products these precautions are essential. It should be noted that high-risk ingredients are those that are added to the product without further processing (e.g. aseptically packed fruit materials that can be added aseptically to the process flow; further treatment, although microbiologically desirable, might affect the flavour).

8.7 Hazard Analysis Critical Control Points

Once the process has been specified and the layout designed, the system should be subjected to a Hazard Analysis Critical Control Points (HACCP) study. This is an analytical tool to ensure continual cost-effective product safety. The steps involved in the operation are systematically assessed and those that are critical to the safety of the product are identified. Operational targets and corrective actions are defined with monitoring procedures. Any design modifications can be easily made at the planning stages of the factory; the operation should have had a HACCP study and once the factory has been built a further study should be conducted to ensure compliance. Any planned changes to the use or operation of the facility should also be analysed. The HACCP concept allows movement to a preventative approach to quality issues whereby potential hazards are identified and controlled prior to causing product failure. As with any steps in the validation of a process the HACCP study should be fully documented and linked with verification procedures and supplementary tests.

8.8 Good manufacturing practice

The plant should be run under the principles of good manufacturing practice (GMP). These are defined as all those management activities, including the effective planning, design, maintenance and control of buildings, equipment processes and personnel, which together ensure integrity of the product in compliance with the law.

In the United Kingdom adoption of HACCP and GMP can be used to show that due diligence has been exercised in terms of the Food Hygiene Regulations 1990 or trade standards such as those required to meet the British Retail Consortium (BRC) criteria. Other countries have similar legal requirements. To date due diligence has been used to show mitigating circumstances in the event of a breach of the regulations rather than as a defence to a prosecution in court.

All equipment should be designed and constructed in a manner that prevents the retention of moisture (unless it is designed to deliberately stay full of sterile water), ingress and harbourage of vermin and soil and to facilitate inspection, servicing, maintenance and cleaning.

The following need special attention:

- materials of construction and cleanability
- surface finish
- joints and fasteners
- drainage

- internal angles and corners
- dead spaces
- bearing and shaft seals
- methods of cleaning.

8.9 Cleaning in place

Cleaning in place (CIP) is a method of cleaning plant by circulating detergent solutions and thus obviating the need for stripping down and manual cleaning. There are two main types of plant to be cleaned within a drinks plant: pipelines and vessels. For pipeline cleaning hygienic design is vital and correct CIP velocity is critical. This is typically 1.5 m/s, an empirical value based on achieving turbulent flow to achieve a scouring action (i.e. Reynolds number above 2500). For vessel cleaning, two systems are available depending on the soil to be cleaned: high pressure/low flow, which relies on jet impingement to remove soil, or low pressure/high flow (e.g. spray balls), which relies on there being a continuous film of detergent solution on the vessel walls. Cleaning solution must be continuously removed from the base of the vessel (known as scavenging).

A typical CIP cycle consists of:

(1) pre-rinse to remove loose soil and reduce the work of detergent;
(2) detergent recirculation to remove remaining soil – there are three main variables to be considered: chemical strength, cleaning temperature and exposure time;
(3) intermediate rinse to remove detergent with fresh water until a neutral pH is achieved;
(4) sanitisation to destroy remaining microbial contamination using hot water above 90°C recirculating for more than 20 min.

In addition to internal cleaning of the equipment, effective external cleaning of the plant and environment is required to remove pathogens and spoilage organisms introduced during normal operation. This must include regular cleaning under the equipment and cleaning the drains. Gel cleaning is the only real innovation in plant cleaning since the introduction of foam cleaning in the 1970s. Its main advantage is the prolonged contact time. The detergent increases viscosity on dilution; hence the neat product is easy to handle and contact times of 45 min are possible compared with around 10 min for foams. Additionally, reduced application pressure reduces the production of aerosols on impact with the surface to be cleaned; these can spread contamination over a wide area.

Automatic fogging systems are available to disinfect rooms. A dense fog is quickly achieved by atomising disinfectant solution through nozzles.

Some construction materials, for example, polyvinylidene fluoride (PVDF), are bacteriologically resistant and it has been shown that simple rinsing without detergent is capable of cleaning them to a state suitable for further drinks production. Such materials are suitable for the production of pipes, coating vessels, etc. and will probably be used more widely in the production of process plant in the future.

8.10 Packaging

Packaging machinery is as varied as the many packages available on the market: some kinds of machinery form generic types and have fairly wide applications, others are very specific. Figure 8.8 shows a typical line layout for a bottling line. The principles of this layout are described here together with some of the more common variations. Bottles are supplied oriented on pallets and are unloaded on to the empty bottle conveyor system. Alternative layouts can be constructed for plastic bottles arriving in large bulk bins and being unscrambled (oriented) on to the line or (especially for PET) blown direct on to the line from preforms. Returnable bottles go through a decrater, which unloads them from crates, then to a bottle washer which cleans them, and finally to a bottle sorting area to remove those that do not belong. De-cappers and de-labellers may also be required. The bottles are then transferred in bulk (many bottles across a conveyor) towards the filler. Bottles are usually rinsed (the rinse water quality is important and it is often treated with ultraviolet light to disinfect it), filled and capped on one machine (really three machines monoblocked together with a

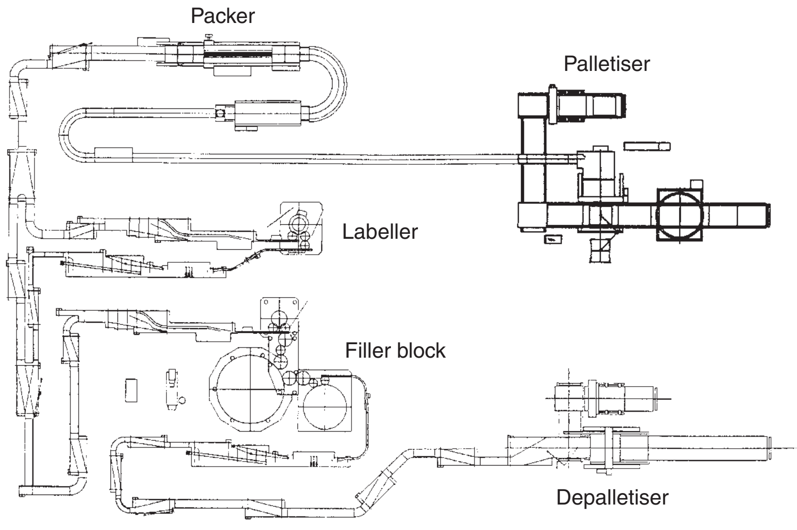

Figure 8.8 Typical packaging line layout. Scale 1 : 200.

common mechanical drive). This provides better bottle handling and control at high speeds, leading to better efficiency. In some cases, a wet glue labeller is also blocked with the filler.

Fillers are often equipped with two cappers for two different capping duties (different types of cap) and can handle a range of bottles by changing parts. Filling speed is dependent on the number of valves and the physical properties of the liquid (different manufacturers' valves will perform differently and also affect speed); some fillers are vacuum assisted to speed the filling rate. Filling machines are now very sophisticated in terms of operation and can be equipped to steam a bottle to cleanse it or pre-evacuate it to reduce oxygen levels, fill from the bottom of the bottle to reduce fobbing using a long tube valve, take a bottle and fill a precise weight, fill to a level, fill a precise volume, etc. (Another benefit of the long tube filling valve is that it eliminates the risk of the bulk product in the filler bowl being affected by contaminated return air from the bottles.) The choice of filler will depend on the image required by marketing, the cost of the product in terms of 'give aways' or overfilling, and the demands of the package or product.

Another factor affecting the choice of filler is its ability to be thoroughly cleaned. The simpler the valve the easier it will be to clean, and the more small passages, springs or complicated seal arrangements there are the more difficult cleaning will be. Most filler manufacturers will test fill and test clean products in their laboratories to demonstrate the cleanability and filling performance to be expected from their machinery.

One popular misconception concerns the counter-pressure filler. It is often stated that the overpressure of gas in the filler bowl pushes the product into the container, but this is not the case. Once the valve is open the pressures in the bottle and the filler bowl equilibrate and product is filled by gravity. (Note: a counter-pressure filler is usually used for carbonated beverages but it can successfully fill still products with an inert overpressure.)

Ultimately, the speed of a filling machine is a function of the engineering limits of metal machinability. The valves are mounted on a ring that has to be machined to very tight tolerances and mounted such that it rotates evenly, hence the overall diameter of a filler has a maximum value and so the number of valves that can be mounted on the pitch circle has a maximum.

There are many decoration options for bottles but the most common is the wet-glue labeller, which is described here, although self-adhesive labellers, sleeving and even a form of bottle printing using heat to transfer the print from a backing paper to the bottle are all available, together with machinery of application.

In wet-glue labelling, labels are stacked in a magazine and removed by preglued pallets that apply a controlled quantity of glue to the reverse of the label. A thin glue film is applied to the pallet, which in turn removes the labels from the magazine. The glued label is then removed from the pallet by a transfer cylinder usually using gripper fingers to hold the leading edge of the label and a vacuum pad to control the label's orientation on the cylinder. The label is now held glue-outwards, which is suitable for application to the bottle. Once firmly transferred to the bottle the dress is completed by brushes that smooth the label on to the bottle. Some simpler machines will transfer a simple label directly from the glue applicator to the bottle using a comb or similar device. Systems exist for cold-glue or hot-melt labelling, wrap-around labelling (where glue is applied only at the overwrap area), over-the-cap labelling (to provide tamper evidence), application of decorative foils (to give a premium image) and a host of other operations. A quick look along a supermarket shelf will show the many options available, some of which are very complicated in that a sequence of successive operational steps is required to achieve the final result.

Intricate label shapes can be applied but generally labels with an aspect ratio greater than 1 : 5 present problems with consistency.

The speed of labelling is generally a function of the bottle handling; the labelling stations described above can handle labels far faster than the technology of bottle handling can present bottles to be labelled.

The next machine on the line is the packer, which collates the bottles into the required sales unit (often 12 or 24 bottles). Sales units are generally shrink-wrapped trays, cardboard cartons or crates for returnable bottles. These are stacked on pallets for ease of distribution and storage.

Bottles are normally coded with a 'best before' date and often a closed code giving the batch or production record for tracing purposes. These are applied by laser printer or ink jet onto the label, cap or neck of the bottle. There is a variety of on-line inspection devices available for ensuring the quality of the product, for example, empty bottle inspectors, hydrocarbon sniffers (to check that returnable bottles have not been used for storage of paraffin, etc.), fill height detectors, label application detectors and date code verifies. All of these can be found on the modern bottling line.

To gain maximum efficiency from a production line it is normal for the machinery upstream and downstream of the filler to run faster than the filler and to provide accumulation between machines so that minor stoppages (for example changing the shrink-wrap reel) can be accommodated without stopping the filler. Figure 8.9 illustrates this for the factory layout shown in Figure 8.8.

Canning lines are very similar to bottling lines except that the container is sealed with a can end, which is rolled on by a seaming machine. There are also many specific machines for special filling operations in use in beverage packing, for example, laminated cartons, foil pouches, etc.

8.11 Conclusion

To ensure that good quality, consistent, wholesome product is delivered to the consumer is a complex operation consisting of a number of sequential steps. It is essential that these steps are well understood. The best way of achieving quality

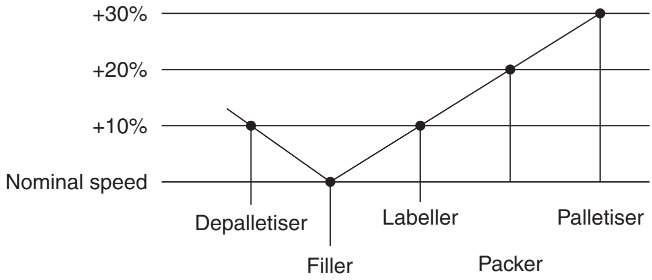

Figure 8.9 Line equipment reference speeds (relative to filter nominal speed).

is to design it into an operation from the beginning and maintain it through the adoption of the principles of good manufacturing practice.

To summarise, a plant will only be effective and the product safe if all the following aspects of design are taken into account:

- the nature and cleanliness of the raw materials
- the selection of materials of construction
- the nature and cleanliness of the packaging
- the process design of the plant
- the functional specification of the plant
- the correct operation of the plant
- the good management of the plant.

9 Packaging materials

G.A. Giles

9.1 Introduction

The importance of packaging in the soft drinks and fruit juices market – be it to preserve, protect or promote the product – cannot be over estimated. Every product will need to be packaged in some form throughout the supply chain.

Many in marketing will consider packaging to be the 'silent salesman'. Research has shown that the decision to purchase or not is made in a fraction of a second, during which time the package has to shout for attention, look and feel appealing and be right for the moment.

In the marketplace, competition is very strong and brand equity is used to establish the position of a product. The type and size of a drinks container, its shape and pack graphics are all used to help differentiate one product from another. Those involved in the development of drinks products need to be aware of the marketing requirements and commercial opportunities to ensure that their development efforts align with the brand strategy, costs and lead times.

It is not possible to cover all aspects of such a significant topic as packaging in a single chapter. The purpose here is to give readers a flavour of the opportunities; they should explore packaging requirements further, with their own in-house packaging technologists (if they have them) or with their packaging suppliers, national packaging institutions or other trade associations relevant to the beverage industry. Some potential contacts are given at the end of this chapter.

9.2 Commercial and technical considerations

9.2.1 General considerations

Soft drinks and fruit juices compete with many other beverages – water, milk (natural or flavoured), tea, coffee and so on – for our attention and the '% share of throat'. Whether they are still, carbonated, sweetened, unsweetened, energy-boosting or complement diets, all will claim to be needed in our hectic lifestyles.

Packaging formats and materials will all play their part in appealing to the consumer. It is important to consider whether the product is to be in a multi-serve or single-serve pack, purchased in a supermarket as part of a weekly shop or as an impulse purchase in a fast-food outlet, garage forecourt or corner 24–7 shop. This may affect the pack size, material format and hence the product processing conditions available to the drink development technologists.

Whether the annual sales volume will justify a customised pack or require a standard format be used, relying on the decoration to provide product differentiation, will be considered during the development phase. The timing to introduce the product in a stock pack will be faster than if a new pack has to be developed, new tooling produced and the pack evaluated.

Often the closure is last to be considered. We call it a ‘closure’, and it is one of the last component processes on the filling line; yet removing it is one of the first actions taken by the consumer. Thus, it might equally well be termed an ‘opening’ for the very significant role it plays for the consumer. Easy access can enhance the product experience (often without the consumer even thinking about it). If the consumer cannot unscrew a cap or breaks a nail tearing open a closure, the whole experience is ruined and a customer may be lost.

Another consideration is how long the consumer will interact with the package. If the pack is a multi-serve, stored in a refrigerator, and the product is poured into a glass or cup, then the interaction time is minimal. If the pack is a single-serve size, but with a ‘drink-from’ closure (now called a ‘sports caps’), then the interaction time will be longer and the tactile properties of the package may have more importance.

When considering the introduction of a new product formulation, the first point to take into account is whether the packaging format already exists. If a new packaging design is being requested for a relaunch it must be handled on the existing filling line, and it must be compatible with the same filling processes. For example, it must be decided whether changes in the packaging size, shape or closure would prove prohibitive. The use of aseptically filled packs, particularly bottles, is capital intensive and would have to be balanced against other process and pack configurations in the context of market opportunity. There may be only certain sizes with which the bottle filler can easily cope. The existing equipment may be a glass or plastic bottle filling system, or may have been purpose built to deal with both. Generally, it is best to have one material running on one line and not have a dual-purpose line. Fillers may be designed for still or carbonated products and may not be able to handle both cold- and hot-fill products. Often a new juice product will need a higher-performance package (e.g. if it is to be hot-filled or in-pack pasteurised); however, with new developments it is often possible to reformulate the product to be cold-filled without special package treatment.

A further consideration is the type of closure system available for use on the filling line. Closure systems are designed to cope with internal pressure caused by carbonation, the pressure created during in-pack pasteurising or the vacuum resulting when the product cools after hot-filling. Most soft drinks closures are standardised and few commercial systems can be customised, other than for colouring, embossing or printing. If there is sufficient volume demand, special sizes could be produced, based on a standard sealing system, but a balance would have to be achieved between the required investment, development and validation programme and the commercial opportunity. By adopting an existing sealing system it should be possible to collate data from other markets on closure performance to make the validation a faster process and achieve a suitable launch target.

Another opportunity for change might be decorative. If labels are used and need to be retained then the method of printing should be discussed with the label manufacturer and examined in terms of print effect versus cost. The label substrate can help to create an impression of quality through the use of plastic, either in conjunction with paper in laminate form or in monolayer form. Whether the labels are pre-cut or reel-fed will also impact on whether the label can be a shaped patch label or is wrapped around the package.

Shrink-sleeve packaging is a very popular decoration process used to increase the graphic area of the package and to convert a standard material pack (e.g. a high-density polyethylene (HDPE) bottle) into an eye-catching pack. This process allows three-dimensional decoration, which changes a 'labelled' bottle into a 'decorated' package: a complete, whole product. It is then possible to change the graphics, colour and so forth on one base bottle design to create packaging for more than one brand. Even bottles made in quite bland and low-valued materials, such as HDPE, can appear to be of premium quality in full-length shrink-sleeve decoration. The flavoured milk market has made use of this technology to great effect. This form of structural package standardisation allows for more than one product or brand to be launched quickly and with minimal extra cost. The money saved can be used either to enhance the quality of print decoration or to increase the level of advertising spend (an example of the packaging process supporting the commercial process).

Pre-decorated packaging has been available for many years, particularly on metal cans. Sleeving can also be used on metal cans. As two-piece beverage can technology is geared to high volume, the use of sleeved cans allows shorter run lengths or special promotions with specific graphics.

In summary, deciding on a packaging process requires consideration of the commercial opportunities, the product processing requirements and the packaging options available, the flexibility of the filling line and the packaging materials and their ability to withstand the processing requirements and shelf-life demands. It is also important to consider the decoration formats and their ability to match the container type, carry the necessary information and withstand (if pre-decorated) the processing itself while maintaining the brand quality. If all this is put together successfully then the brand will benefit from the added value created by packaging.

9.2.2 Packaging materials

The range of packaging materials covered in this chapter includes those for still and carbonated products, some of which can be cold-filled and others that need to be heat-treated. Heat treatment can either be within the package or by pre-treating and filling hot or aseptically into a pre-sterilised package to ensure an acceptably long shelf life to suit market requirements. The role of packaging is to contain, protect and preserve the product from producer to the end user, that is, to present the product in the condition intended by the producer for the enjoyment of the consumer. The packaging materials must, therefore, meet the processing requirements and be capable of running through the production lines smoothly and efficiently as well as meeting the necessary economic constraints so vital in a competitive market.

The packaging must be capable of being collated into suitable formats and must withstand both the distribution requirements and those at the point of sale. It also performs a marketing role in promoting brand positioning through package shape, material and decoration. It must enable identification and also provide selected information (nutritional and statutory). The packaging must remain secure in terms of providing tamper evidence and microbiological hermetic seals. In performing all these functions, it has additionally to be capable of meeting environmental legislation requirements for packaging waste at an economical cost.

Although much of the packaging specification will be determined by the product and process requirements, the pack size, material, neck size and closure will be determined by the market and commercial requirements.

Polyethylene terephthalate (PET) bottles have significantly changed the face of soft drinks packaging since their introduction into the world market in the late 1970s. However, glass and metal cans have continued as important packaging materials; they offer excellent process conditions, high-speed filling and very good barrier properties, thereby resulting in acceptable shelf lives in the smaller, single-serve sizes. Laminated board drinks containers provide a continuing development platform in formats that are well understood, and accepted, by the consumer. These well-established systems are not only ideal for the home market but are also capable of global distribution for brands with a wider appeal. Most countries around the world have drink carton filling capabilities – important for global branding and distribution.

PET packaging has allowed drinks companies to move into in-house bottle production and to operate more independently from packaging manufacturers. In turn the packaging manufacturers have had to offer a more comprehensive service, from managing in-house operations to training customers' operating personnel for fully owned operations. This has allowed drinks companies to drive their own packaging developments and product portfolios.

9.3 Processing

The processing of a product plays an important part in the choice of packaging material and format. This section looks at the following processes and their impact on packaging:

- cold-filling
- in-pack pasteurising
- hot-filling
- aseptic filling.

9.3.1 Cold-filling

Many soft drink products are formulated with ingredients that do not need to be heat-treated in order to be microbiologically stable over the required shelf life. Still products can be filled into the full range of packaging materials, with the limiting factors being the barrier properties (i.e. oxygen transfer rate) of the more flexible materials. Carbonated products need to be packaged in more rigid materials or in structural designs that can withstand internal pressure, for example, PET bottles. Special 'footed' designs in the base of a PET bottle and smooth shoulder contours enable it to withstand the required pressures and impact if dropped. Carbonated containers are circular in cross-section in order to resist the internal pressure. Any embossed features, particularly in a PET container, will require careful design as the features may be distorted by the effect of the internal pressure.

9.3.2 In-pack pasteurising

Some soft drinks and juices need to be heat-treated to provide a microbiologically stable product. One method considered very reliable and robust is in-pack pasteurising (IPP). This is particularly suitable for treating carbonated products as they cannot be hot-filled. Whether still or carbonated, it is normal to fill the products cold, although some still juices are heat-treated first, filled warm and then in-pack pasteurised as this allows for a different temperature regime in the IPP cycle as well as giving a better balance of internal/external pressures during and after the IPP treatment. This method is suitable for certain plastic containers and metal cans.

Once filled and capped, the bottles (or packs) are taken through an IPP unit, which warms them up to the pasteurising temperature (in about 20 min), holds them at that temperature for the required period (normally 20 min at 70–75°C) then cools them down. The whole cycle takes about 60 min. The actual pasteurising temperature and time cycle will be determined by the drinks technologists.

The packaging materials (including heat-shrink sleeves, if pre-decorated, and the printing on cans) are all subjected to continual dowsing in water, warm then hot then cold, over a long period of time. Care thus needs to be taken, and materials tested, when selecting the appropriate packaging format.

For still products there is a wider choice of materials, all of which must be resistant to heat and high humidity. For carbonated products the package must also be resistant to the buildup of internal pressure as the contents are heated to the correct pasteurisation temperature. The choice of bottle closure is also critical since leakage is possible during the cycle because of the internal pressure and differential shrinkage of the closure and bottle materials. For bottles with wide-mouth neck finishes (35 mm and above), it is standard to use metal roll-on (RO) closures as they are more stable at high temperatures and can resist the internal pressure better than plastic closures.

Care should be taken in selecting the appropriate cap-sealing compound specification and even the thread-finish profile as these will have a part to play in giving consistent performance. When selecting these components it is sensible to discuss performance requirements with the packaging suppliers.

9.3.3 Hot-filling

Still products can be hot-filled into a wide range of packaging materials (provided they are heat resistant). Juices are normally hot-filled at 85°C and the package sealed. In order to sterilise the whole of the internal surface of the package, it is usually turned on its side during the early part of the post-filling cycle. It is normal for reasons of microbiological safety that the temperature is not allowed to fall below 80°C during the first 3 min after filling. After this time the packages are either immersed in a cold water tank or dowsed in cold water until cool enough to prevent the product from ‘cooking’, thereby affecting the desired flavour profile. Care must be taken that the packaging is dry when packing these products for distribution. If they remain damp and the secondary packaging is corrugated board, there is a real risk that the board will soften and the stacked products collapse.

9.3.4 Aseptic filling

There are several machine systems available for aseptically filling glass and plastic bottles for still juices. (Aseptic filling of drink cartons is covered in Section 9.7.) Carbonated drinks are not aseptically filled. There are two main aseptic filling workflows, with a third workflow used less frequently. The first system sterilises the container, fills and seals it; the second takes a sealed, precleaned bottle, removes the seal in a sterile environment, fills and re-seals the container. The third system blows a bottle and while it is still sterile fills it and then seals it, all within the same machine, this is known as a ‘form–fill–seal’ (FFS) system.

In the first system the inner surface of a pre-made, open-necked bottle is sterilised. The sterility is maintained while the bottle is transferred to the aseptic filler. The layout of this type of filler is shown in Figure 9.1. The pre-made

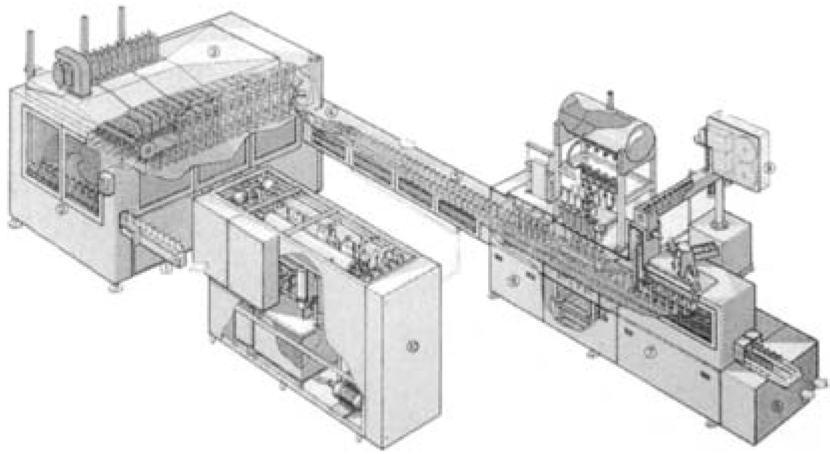

Figure 9.1 Aseptically operated filling and closing lines for bottles and wide-mouth containers made of glass and plastics. (1) one-lane feed transfer; (2) in-feed into bottle sterilisation unit; (3) bottle sterilisation unit; (4) discharge; (5) two-lane transport of bottles to filler; (6) aseptic 10-up inline filler; (7) closing machine; (8) discharge for closed bottles; (9) lid punch and in-feed; (10) aseptic module.
Source: Reproduced with kind permission from Robert Bosch.

bottles (1) are fed into the sterilising unit (2, 3) and the bottles are then kept sterile until after closing at (7).

It is normal to fit a foil seal before the main closure or dust cap is applied because it is easier and more reliable to sterilise a foil using heat or hydrogen peroxide (H_2O_2) sterilant than to sterilise a pre-formed threaded closure (Figure 9.2). Heat-resistant materials can be steam-sterilised but plastic materials usually need H_2O_2, which then needs to be flushed off using hot air. It is difficult to ensure that all the sterilant has been flushed away from the thread form or sealing system. If a foil is used as the primary seal then the plastic closure can be of a simpler design as it is only used to re-close the bottle once opened.

The bottles used in this process are all wide mouthed as the sterilant has to be driven out of the bottle before filling and this becomes more difficult the bigger and taller the bottle and the narrower the neck (Figure 9.3). Since heat is used to flush out the H_2O_2, (plastic) bottles have to be heat-stabilised even though the fill itself is at ambient temperature or under chilled conditions. Care should be taken with the specification for the bottle and its shape in order to counter distortion when it is subjected to heat. It is recommended that if this filling system is used, a joint development team is formed, including both the bottle and filling system manufacturers. If the bottle is to be pre-decorated with a heat-shrink sleeve, the sleeve manufacturer should also be involved because special heat-resistant specification films will be required.

Figure 9.2 Polyethylene terephthalate (PET), heat-stabilised bottles for aseptic filling.
Source: Reproduced with kind permission from Robert Bosch.

Figure 9.3 Wide-mouth bottles.
Source: Reproduced with kind permission from Robert Bosch.

This issue has been addressed by the second type of aseptic filling system in which a polyethylene bottle (which can be multi-layered to add a gas or light barrier) is moulded under sterile conditions. While the bottle is still sterile, and the polyethylene is soft, the trim material above the neck finish (moil) is crimped closed to seal the bottle. The sealed, sterile, empty bottle can then be packed and transported to a filling line, which may be in another location from the blowing operation. On the filling line the external surfaces of the bottle are sterilized, which is easier than sterilising the internal surfaces of an open-necked bottle. The closed, sterile bottle is then passed to a trimming station where the moil is removed and the bottle is filled aseptically. Again, a foil seal is used to close the filled bottle and an overcap of a suitable design is applied to protect the foil during transit, giving the bottle a 'normal' image and providing a re-closure during use. As this system relies on the moil being used to seal the bottle, the manufacturing process requires an extrusion blow-moulding operation and a material that will impact-weld while it is still hot. The polyolefin family of materials fits this requirement.

PET and glass bottles are made without moils and have open necks, so they are filled by the open-necked aseptic filling process. The moulding operations for PET and glass produce clean bottles, and several methods have been trialled over the years to seal the open neck after forming, while it is still clean, and then re-open it under sterile conditions. These methods have not usually been successful, but one PET bottle manufacturer has now linked up with an aseptic bottle manufacture to offer an in-line blow-moulding and filling system for juice products. There are pros and cons for this system versus a more conventional open-neck aseptic filler. These centre on capital investment, volume off-take and bottle design flexibility.

A third system is the FFS system. Two package formats are available for this system. The first is the thermoformed container route. Normally this takes the shape of a tapered pot with a foil seal, very much like a yoghurt pot. One of the benefits of this system is that it has been used for many years and it is well established for package materials such as toughened polystyrene and high-contact-clarity polypropylene. If barrier properties are an issue then a multi-layer specification laminate should be used. The alternative format is an FFS extrusion blow-moulding system, normally used for sterile products in the pharmaceutical or medicine markets. Since the bottle is sealed by impact welding while still in the mould, the materials are usually from the polyolefin family (polyethylene or polypropylene). These bottles are typified by the use of an integrally moulded, twist-off, winged 'nib' to open the bottle. However, they do not seem to have taken off in the mainstream drinks market, possibly because of the pack image or because the FFS system is relatively slow for volume use and is more suited to high-added-value markets, selling therapeutic benefits rather than packaging imagery.

9.4 Bottles

9.4.1 Glass

When choosing a bottle it is important to consider not only the intended market and package size but also the product to be contained, the shelf life and the filling requirements. Glass bottles can offer an excellent shelf life as the material is impermeable to gases, preventing both oxygen ingress and the loss of carbon dioxide from sparkling drinks.

Since many fruit drinks require heat processing to ensure microbiological stability, consideration must be given to the packaging material and its tolerance to elevated temperatures. If the product is also carbonated then the choice of packaging material and closure system becomes even more critical because of the required heat processing. As the heat builds up so does the internal pressure. Typical IPP conditions are 65–75°C for 20 min, with a warm-up and cool-down time of 20 min for each phase.

Another important factor is the volume of unfilled headspace designed into the bottle. Typically, this is not less than 5% of the total volume for an IPP carbonated product and ideally it is more like 7%. This is because as the product expands during pasteurising, the headspace becomes squeezed, and the smaller the headspace volume, the higher the internal pressure becomes. This could blow the closure off the bottle or, more typically, cause leakage of carbon dioxide or product or both. The loss of product gives rise to uneven fill levels, which could cause consumer concern over apparent low fills or, more seriously, draw attention from trading standards officers because of short volume fills.

The profile of the bottle shoulder and neck diameter are also important factors. The smaller the diameter of the closure and the more tapered the neck/shoulder, the more exaggerated the change in fill level becomes. Also, in order to create a 5–7% volume of ullage or vacuity the fill level height will be much lower, which can give the appearance of a short fill or deceptive volume.

The larger the diameter of the closure, the more force will be exerted on the closure during heat processing, which again could lead to product or gas leakage or, more seriously, to closure detachment during processing. During heating the metal closure is likely to expand more than the glass neck, and although the reverse is true on the cooling cycle the pressures will still be higher and the compound lining softer. A careful choice needs to be made for the compound specification and cap application. Assistance can be sought from closure suppliers and capping equipment specialists.

For carbonated products, for which heat treatment is not required, the level of carbonation at the time of consumption is likely to be important to the mouthfeel and flavour profile. Therefore, measuring carbonation levels during a real-time storage trial or even a shortened trial made at elevated temperatures (i.e. 35–40°C) will be an important part of the package validation protocol.

One reason why metal closures (mainly roll-on pilfer-proof, ROPP) are used on glass is because the dimensional tolerances of glass are such that it is vital to form each closure to fit each individual bottle neck (the ultimate form of customisation) and this can be done with ductile metal. In recent years, the use of plastic closures on glass bottle has grown. Consumers usually find the tactile properties, ease of grip and safety (from metal edges) of plastic closures superior to ROPP metal caps.

In order to create a satisfactory seal on a glass neck, some closure suppliers have developed single-piece plastic screw caps with spigot seals. Despite the dimensional tolerances of the neck finish, the spigot will always be gripped by the bore, which is usually a more controlled, smooth, parallel section of the neck finish (only for a defined length, due to the glass blowing process). As the molten glass shrinks on cooling, the thread detail can shrink away from the thread-forming part of the mould cavity; but a metal spigot (the blowing pin) is positioned in the neck bore section during the blowing process. Thus the glass can shrink only on to the fixed-diameter pin, which helps to control the bore.

Care has to be taken over how the sealing system works to retain the carbonated gas since the internal pressure has to be released before the closure releases itself from the thread finish. Otherwise, the closure can release with some force. Most plastic closure systems used on glass and other bottle materials now incorporate a liner seal that releases the internal pressure soon after the consumer begins to open the closure, thereby ensuring that all the gas has been released by the time the closure detaches from the bottle. The liner also creates a more flexible seal, accommodating a broader range of glass finishes and tolerances from more than one glass manufacture even if they are all at the same nominal diameter.

PET now dominates the multi-serve carbonated drinks market for products requiring only cold-filling. However, glass has continued to enjoy success in the single-serve still and carbonated juice markets. For small single-serve (i.e. 180, 250 and 500 ml) bottles, the shelf life of carbonated products is an issue because of the surface-to-volume ratio, particularly in the case of PET.

For products in a long or varied distribution chain, such as cash-and-carry, corner shops or garage forecourt retail outlets, the issue of shelf life is important. Major brands can often achieve sales within the timeframe of the shelf life offered by PET. Other brands that may have to satisfy the requirement of a longer supply chain have found that glass offers the appropriate tolerance to the required processing conditions and provides a long shelf life as well as a good premium image. Decoration using heat shrink sleeves has added to this premium image.

9.4.2 Polyethylene terephthalate

PET represents one of the most significant recent changes in terms of the packaging materials available to the soft drinks and fruit juice markets. Introduced to the UK market in commercial volumes in the late 1970s, PET has driven the most important market opportunity since the introduction of aseptically filled, single-serve juice drinks cartons. As mentioned above, PET has allowed the drinks industry to develop into more flexible and larger supply chains. PET offers the trade a package with superb clarity, unbreakability, consistent neck finish, recyclability and, in some countries, the option of refillability.

In considering PET for a particular application, thought needs to be given to the process and shelf-life requirements. PET is at its best in cold-fill, multi-serve applications with carbonated products, where re-sealability and bottle strength are an advantage. The carbonation pressure holds the bottle rigid, making it ideal for palletisation and distribution. PET's unbreakability means that only minimal secondary packaging, such as shrink film, is required.

As with all plastics, PET is permeable to gases, letting carbonation out and oxygen in. The shelf-life requirements, therefore, need to take into account the ambient temperature conditions, both in distribution and at the point of sale. A general rule of thumb for PET is that the shelf-life performance is halved for each 10°C rise in ambient storage conditions. Improvements in the barrier performance of PET have addressed this, as has the trade learning to move product through the supply chain using just-in-time (JIT) systems.

The barrier performance of PET can be improved by the addition of other plastics to the base material or, probably better, by adding discrete inner layers of high barrier material such as ethylene vinyl alcohol (EVOH) or nylon. These layers are usually added to the PET preform. PET bottles are produced in two stages. First a preform, similar in appearance to a test tube, is produced by injection moulding. This preform contains the neck finish and it is the body of the preform which is stretched into the final bottle shape. It is this stretching process that imparts the vital properties to a PET bottle. The stretch ratios of preform diameter to bottle diameter and preform length to bottle length are used by the bottle manufacturer to calculate the ideal stretch to ensure maximum barrier performance. The stretching imparts strain-induced crystallinity, mainly into the side walls of the bottle, which have a thickness of around 0.25–0.3 mm for a 2 l bottle. The unstretched portions contain amorphous PET, which, although thicker than the sidewalls, has a lower barrier performance per unit thickness than the stretched, crystallised material. ('Crystallinity' refers to material in which the molecular structure is aligned in a regular (crystalline) fashion; material in which the molecular structure remains random is termed 'amorphous'.) Preform and bottle design therefore play a vital role in determining bottle performance, as does the surface-to-volume ratio. Hence, small, single-serve PET bottles will have a shorter shelf life than their multi-serve equivalents.

As designers have come to understand the importance of material stretching and its impact on bottle performance better, so bottle weight has decreased. In turn this has added to the shelf life by reducing the amount of carbonation gas absorbed, and held, in the mass of PET used to make a bottle. PET is recyclable as polyester fibres or as resin to be reused in the production of food-grade bottles. The weight reduction development has been a good environmental message.

The level of crystallinity plays an important part in the level of heat stability of a PET bottle. Again, the evolution of preform designs and grades of material has increased the level of heat stability. In order to extend this stability to the temperatures used for hot-filling (typically 85°C), additional methods have been required to increase the level of crystallinity in PET bottles. Success has been achieved by effectively 'heat-setting' the bottles during the normal blowing process by heating the moulds to temperatures high enough to provide the required hot-fill tolerance. Since the heat-setting process increases moulding time there are commercial implications that limit application of this process to those products for which heat treatment is critical for microbiological stability and those markets that require an unbreakable bottle. An example is a sport drink made with a high juice content.

When a product cools, it contracts. This distorts the normal shape of the PET bottle panel. Hot-fill bottles are made with panels in the body section and patented features in the bottle base that collapse in a consistent way so that the bottle can be handled and distributed when cold.

The vacuum that creates the bottle panel has to be contained by the closure, and so hot-filled bottles have a specific closure design and applicator that are different from standard carbonated closure designs (see Section 9.5)

9.4.3 Polyvinyl chloride

Polyvinyl chloride (PVC) is used for some drinks bottles, mainly for dilutable juice drinks and cordials. PVC is easily moulded into more interesting shapes than PET and is relatively rigid, although it has significantly less impact strength than PET. There is an environmental lobby against the general acceptability of this material for food (particularly drinks) use, and so there has been a steady decline in the use of PVC containers. As PET has gained ground, the technology has advanced and in-house operations have grown, PET has made further inroads into the PVC drinks bottle market. One advantage PVC continues to enjoy is that, cut-through handels can be included because bottles are made by the extrusion blow-moulding process; this is something which PET bottles, made by the injection–stretch blow-moulding process, have not been able to replicate in a one-piece design. This makes PVC convenient for the larger 2 l concentrate bottles. Companies producing drinks for export may find their export market closed to them if they use PVC. This has driven even more standardisation to PET.

PVC is generally not suitable for carbonated juice and soft drinks products because of its creep strength and barrier properties.

9.4.4 High-density polyethylene

High-density polyethylene (HDPE) bottles in multi-serve and single-serve sizes are used for juice drinks that appear mainly in the chill cabinet (short shelf-life distribution) section. This is probably more to do with the product process technology required to produce longer-shelf-life bottled products than the material itself, which is not hot-fillable, although oxygen permeability leading to oxidised off-flavours becomes an issue for products with extended shelf-life.

9.4.5 Polypropylene

Polypropylene (PP) bottles have the advantage that they are inherently hot-fillable (at 85°C) and retortable to 120°C. The bottles are normally extrusion blow-moulded, and can also be made with multi-layering to include barriers against oxygen permeability. This technology is commonly used for sauce bottles and juice products. Since most of the bottles are of an oval shape, product contraction resulting in volume reduction is countered by allowing the bottle to become more oval under the effects of vacuum.

PP usually has a milky appearance (particularly when empty). However, the contact clarity is really quite remarkable, making a filled bottle look attractive. Full shrink-sleeve decoration changes the whole visual impact if total clarity is an issue and PET proves not to be suitable (e.g. for oxygen-sensitive products).

9.4.6 Plastics permeability data

Gas permeability depends on many factors including

- *Temperature*: gas permeability increases with increasing ambient/storage temperature.
- *Humidity*: atmospheric gas permeability increases with humidity in polymers that absorb moisture. This is an issue when EVOH is used as an oxygen barrier layer in a liquid drinks bottle.
- *Crystallinity*: gas permeability decreases with increasing crystallinity.
- *Orientation*: in-plane biaxial orientation decreases gas permeability.

Table 9.1 provides a summary of gas permeability data.

9.5 Closures

The choice of closure materials is generally dependent on the required processing conditions and the nature of the contents and closure system (i.e. the application and sealing method).

9.5.1 Metal roll-on or roll-on pilfer-proof closures

Metal roll-on (RO) closures are used mainly on glass bottles, where the neck finish tolerance makes it very difficult to produce a pre-threaded cap that will

Table 9.1 Gas permeability data for various plastics

Material	Oxygen (cm^3 mm/m^2/day)	Nitrogen (cm^3 mm/m^2/day)	CO_2 (cm^3 mm/m^2/day)	Water vapour transmission (g/m^2/day)
EVOH	0.01–0.04[a]	0.0015–0.003[a]	0.01–0.08[a]	0.5–2[b]
Nylon (amorphous)	1.5	0.5	3	1
Nylon 6	0.6	0.35	3	3
Nylon 66	0.6	0.3	3	5
Polycarbonate 100	13	500	3	
PEN (polyethylene naphthalate)	1.5	—	—	0.75
PET	2	0.3	6	0.6
HDPE	100	20	200	0.2
PP (oriented)	60	17	200	0.1
PVC (plasticised)	140	70	900	5
PVDC (polyvinyl dicholoride)	0.05	0.1	2	0.1

Note: For gas permeability the partial gas pressure gradient across the film is 1 atm. For water vapour transmission the humidity gradient is 90%.
[a] Increases with ethylene content.
[b] Decreases with ethylene content.
Data source: RAPRA Technologies Ltd.

ensure a reliable seal every time. The capping machine drops a plain shell (usually made from aluminium, and lined with a sealing compound) over the (filled) bottle neck. The capping head applies a downward force sufficient to create the primary seal and then roller wheels deform the side walls of the closure shell to the shape of the neck thread and follow the thread form over the length of available neck thread. Each closure is therefore effectively custom-made for its bottle. This places a great deal of dependence for closure reliability on the bottler and the capping machine. For those drinks which are sugar based, any spillage or breakage will affect the capping heads, which require ongoing maintenance and quality checks. The advent of more accurate necks on PET bottles has allowed the increased use of pre-formed plastic closures and simplified capping heads. In addition, as plastic closure technology has improved, it has been possible to fit plastic closures on to a wider range of glass bottles and the metal RO closure has suffered a demise in the soft drinks market.

RO metal closures come in two forms: tamper evident (TE) and non-TE. There are two TE formats: detachable TE ring and retained TE ring. If the bottles are to be returned and refilled it is important that the TE ring is removed at the time the bottle is opened. However, the retained type of ring has sharp edges, which may be an issue for some markets. The ring normally splits into six sections which look like wings when opened. It is these sharp edges that could be of concern. It is also possible to push the wings back into place and pass the bottle off as being unopened.

The retained TE ring is normally used for one-trip bottles. If the design intends the consumer to drink directly from the bottle, then importance will be placed on the height of the neck finish from the TE bead to ensure that the consumer's lips do not touch the sharp edge of the TE ring. If the product is carbonated and needs to be in-pack pasteurized, then consideration will have to be given to the thread form on the bottle (usually glass) neck.

There are also combination RO closures that have the metal shell and then a plastic TE band fitted into a holding collar shaped into the shell. This arrangement has the advantage that the plastic band can be a different colour from the shell and so can make it more evident whether the bottle has been tampered with.

9.5.2 Vacuum seal closures

Vacuum seal closures are generally of a lug seal design (Whitecap is a classic and synonymous with the term Twist-Off®) and made in tinplate. The designs usually require an internal vacuum within the package to maintain the seal and microbiological integrity. These closures can be found in a wide range of applications, such as juice drinks, sauces, condiments and baby foods. They are suitable for hot-, cold- and aseptic filling, pasteurisation or closed retort processes, and come in a range of sizes from 27 to 110 mm (Table 9.2).

Table 9.2 Closure style identification

∅	RTO	RTS	RTB	RSB	FTO	FTS	FTB	FSB	DTO	DTB (a)
(mm)	RTO	RTS	RTB	RSB	FAO	FAS	FAB	FSB	DTO	DTB (b)
27	•									
30								•		
38	•		•		•				•	
43	•		•		•		•			
48	•		•		•		•	•		
53	•	•	•	•	•	•	•	•		

(a) Carnaud Metalbox; (b) Whitecap.

Key: RTO, regular twist-off/open; RTS, regular twist-off/open with step; RTB, regular twist-off/open with safety button; RSB, regular twist-off/open with step and safety button; FTO, fluted regular twist-off/open; FTS, fluted regular twist-off/open with step; FTB, fluted regular twist-off/open with safety button; FSB, fluted regular twist-off/open with step and safety button; DTO, deep twist-off/open; DTB, deep twist-off/open with safety button.

Regular, regular twist-off/open cap with an overall height of approximately 10 mm. Step: cap with a step in the panel. Safety button: raised circular portion in the centre of the cap panel that acts as a tamper evident feature (visual and audible vacuum-indicating device). Fluted: cap with flutes or knurls on the skirt of the cap for better gripping. Deep: deep-drawn cap having an overall height of up to approximately 15 mm.

The closure is lined with a plastisol compound, which can be adapted for different product applications, including stacking and tray packing, where the top load exerted on the container may compress the seal.

In the filling process, the product, for example a still juice drink, is hot-filled (typically at 85°C) and the headspace flushed with steam in order to create a vacuum seal under the cap once the product cools and the steam condenses. In the application process, a hermetic seal is formed when the closure's preheated plastisol gasket is compressed against the glass sealing surface. At the same time, the lugs engage the glass threads and when sufficient vacuum is achieved the vacuum-indicating button is drawn down.

All the designs are made to standard sizes and a unique neck finish is required, as is specialised capping equipment. The closures require a rigid neck material as the closure typically has four points (lugs) of contacts. This means that a quarter turn of the closure removes it and reapplies it, which offers the consumer significant convenience of use. There are also closures with three, or six lugs, depending on diameter. (The 38–43 mm diameter closures, for example, have three lugs.) In some markets these closures have been used on plastic bottles. However, the lug seal can distort the flexible neck and the closure can be twisted to override the neck threads. Thus, care must be taken if using these types of closure for this application.

Tamper evidence is provided by one of two methods: by a circular portion in the top panel in the closure, which pops up on opening, or by a secondary plastic fitment. In the first method, a distinctive 'pop' is heard when the consumer releases the vacuum held by the closure and the vacuum-indicating button pops up, providing both an audible and a visual indication of opening. In the second method there are two formats for the secondary plastic fitment. The first format is the application of a heat-shrink sleeve over the sealed container neck, which the consumer has to remove before opening the closure in the normal way. The second format is the insertion of an injection-moulded plastic fitment into the closure by the closure manufacturer. This composite (plastic and metal) provides the necessary barrier properties and the same vacuum-indicating button and can be applied with the normal application speed for a twist-off closure.

The benefit of these closures is that they have two forms of tamper evidence: a vacuum-indicating button and a plastic tamper-evident band. They are ideal for hot-fill, cold-fill and retorted processes, and generally are available in the smaller 40–51 mm size range.

To improve appearance and differentiate a product from the competition a deep-drawn variant, the deep snap reseal, is available. The deep-drawn closure allows space for more graphics on the skirt of the cap, which can be seen from the store shelf. In addition, the deeper skirt allows easier removal and application of the closure.

If a vacuum seal is required on a more flexible material (e.g. PET), then a multi-start thread should be used. This allows a 360° grip on the neck. A number of juice and sports drinks bottles are made in heat-set PET for hot-filling. Neck sizes are typically in the 38–43 mm size range, although the 48 mm size is also made. The closures for these bottles are made in plastic (normally PP) and combine the capabilities of vacuum retention and tamper evidence. The seal is a PVC-free, FDA-approved gasket that is designed not to increase the torque retention on removal. The design of these closures enables them to maintain the same imagery, in PET, because they incorporate the same neck finish on glass.

9.5.3 Plastic closures

Plastic closures are now major components in the closures market, thanks to the inroads made by PET into the market share of glass. These closures are generally made with an integral tamper-evident ring. As the PET market has grown, so has the opportunity for plastic, pre-formed closures.

The first-generation plastic closures used for carbonated products were made mainly from PP in a one-piece design with a spigot seal. Given the quality of the seal and the superior concentricity of the neck of PET bottles, it was possible to remove the cap with ease. Typically, these closures have one-and-a-half turns of thread engagement with the bottle neck. Since the spigot creates its seal by entering the bore of the bottle neck, it requires a certain amount of closure rotation to remove the spigot from the neck and start venting the carbonation gases in the headspace of the product. By the time the closure disengages from the bottle neck the back pressure in the headspace must be reduced enough not to 'blow' the cap off the bottle.

Closures made with a liner (or wad)-type sealing system break the seal faster, thereby allowing more time to vent the carbonation gases. The technology of liners has developed significantly over recent years. This development has improved the reliability of the seal across a wide range of bottles, both glass and plastic, and offers the potential for an improved barrier in the closure. All this has meant that most closures in the soft drinks and juice market today have liners of one form or another. There are two types of liner: a loose liner pushed into the cap after the cap is moulded and a moulded-in liner, which is formed at the time the closure is made.

Plastic closures can be made by injection moulding, where the mould has two parts – a core which has the inside features of the closure and a cavity which has the features of the outside of the closure. Molten plastic is injected into the mould, which is water-cooled. The plastic solidifies, and the mould opens and the completed closure is ejected. If the closure is a two-piece design (i.e. fitted with a loose or flowed in-liner), then this is added later. Another technology for moulding plastic closures for the beverage market is compression moulding. A hot pellet of PP is positioned in a mould, similar to the cavity of an injection mould. As the mould closes, the pellet is squashed and deformed into the shape of the closure by the core of the mould. An advantage of this system is that a liner can be formed as part of the same operation: as the closure leaves the mould, a hot pellet of sealing compound enters the newly formed closure and another forming mould shapes the liner, which is then firmly attached to the closure.

As the plastic closure market has grown, designs have been developed that are also suitable for glass bottle necks (particularly in the returnable, refillable markets, where easy removal of the closure and the tamper-evident band is essential during the process prior to refilling). This increased flexibility of closures has led to a further decrease in the market share of metal RO closures in the soft drinks sector.

9.6 Cans

More than seven billion drinks cans are made in the United Kingdom each year. The real growth in beverage cans came with the development of the easy-open end in the 1960s, which made access to the product much more convenient. The first cans had the familiar ring-pull ends. As the technology developed, the response to market demand to have a retained ring made the market change from ring pulls. The ends are made from aluminium because it can be formed into complex shapes and offers good opening quality; the strength of aluminium can maintain pack integrity up to the point of consumption.

Steel easy-open ends have been tried for the beverage market several times in order to have a single material on steel cans. However, since the formability of steel is not as good as that of aluminium, there has not been much takeup of the steel designs offered to date.

In terms of market positioning, the beverage can is the epitome of effective, standardised packaging for carbonated and still drinks products, particularly those that are in-pack pasteurised. The format can be handled at very high speeds on filling lines to the extent that the makeup of the graphics plays an important part in can mobility because of the slip factor of various inks. A can's inherent strength when filled and its stackability make cans ideal for high-volume products sold either in multi-packs or through vending machines.

The main can format for the European soft drinks market is the 330 ml size, made either from aluminium or steel; a 250 ml size is also used. In US markets, the 12 and 16 oz sizes dominate. The core diameter is 206 for both soft drinks and beers, but in the last couple of years the market has progressively changed to the 202 diameter. (The designation 206 means a diameter of $2\frac{6}{16}''$ and 202 means a diameter of $2\frac{2}{16}''$.) The core diameter is not the can body diameter but the neck plug diameter (see Figure 9.4 for an explanation of the terms used for can dimensions). These diameters have been metricated to a nominal 57 and 52 mm for 206 and 202, respectively. The purpose of reducing the neck diameter was to reduce the size of the can's end, thereby saving around 17–20% on the amount of metal

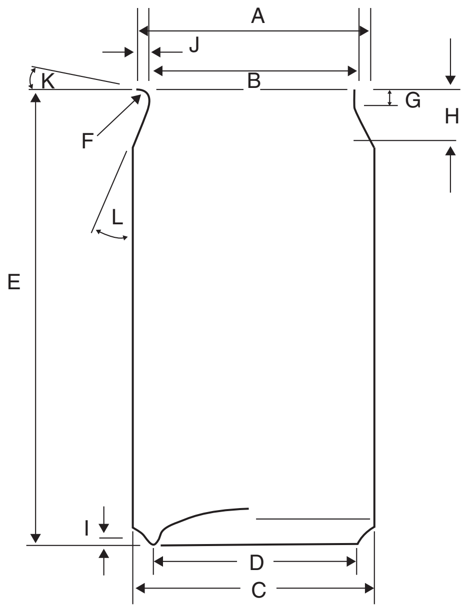

Figure 9.4 The can body. (A) neck diameter; (B) neck plug diameter; (C) outside body diameter; (D) stand diameter; (E) finished can height; (F) flange radius; (G) neck seaming clearance; (H) overall neck height; (I) nesting height; (J) flange width; (K) flange angle; (L) neck angle.
Source: Reproduced with kind permission from Continental Can Europe.

used in making the end. A great deal of development resource goes into reducing the amount of metal that goes into making a metal can as well as reducing the skeletal waste left from the can-making process. Body thicknesses have been reduced by wall-ironing techniques to the point where they are extremely thin. The can-making industry can be congratulated for continuing to make such progress for so many years.

The other can sizes used in the soft drinks industry are the 250 and 150 ml sizes, which use a 200 (50 mm) diameter end. These are the sizes used for trial or airline packs. Manufacturers of some premium drink products have also found the 250 ml format size attractive from a retail price-break point of view, and the tall, slim size is seen as an attractive profile, particularly if targeted at the female market.

Can bodies are made either from steel or aluminium, requiring the use of the advanced engineering and sophisticated technology of two-piece can-manufacturing equipment. The quality of the materials used is critical for the forming of metal beverage cans. For steel cans, a special grade of low-carbon steel is used that is coated on each side with a very thin layer of tin. The tin protects the surface against corrosion and acts as a lubricant while the can is being formed. For aluminium cans, the metal is alloyed with manganese and magnesium to give greater strength and ductility. Aluminium alloys of different strengths and thickness are used for the can body and end (i.e. the can 'lid' or closure).

The manufacturing process calls for the raw material to be specified at the smelting or furnace stage. The resulting output is large coils of sheet metal, which are then fed into presses to form shallow cups of a similar diameter to the finished can. Each cup is rammed through a series of carbide rings, the wall-ironing process, which re-draw the cup, thinning the side wall and increasing the height until the final can shape is developed.

The top of the can, which is irregular at this point, is trimmed to a precise height specification and the surplus material recycled. The can body is then cleaned, coated and printed. After this the can body has its open end reduced in diameter or 'necked in'. The top is then flanged outwards to accept the end or lid to close the can after filling.

The manufacturing of the can end is an even more complex process and an example of precision metal forming. The material is supplied in a coil of special alloy aluminium sheet that is fed through a high-speed press which stamps out thousands of ends (or 'shells') every minute. The edges of each end are curled and a compound sealant is applied into the curl. In the next step, the shells are fed into a sophisticated conversion index press, which carries out two processes with each stroke: the formation of the tab and the formation of the finished shell. The pull tab is formed from a separate, narrow coil of aluminium. The strip is first pierced and cut, with the tab being formed in two further stages before being joined to the can end. The shells themselves move through a number of stations to create the rivet to hold the tab, mark an identification on the end, form the panel and mark and score the opening or mouthpiece of the end. At the final position, the shell and tab are aligned and the rivet swaged to join the two together. The shape of the opening has changed over recent years to improve the pourability and ease of drinking directly from the can.

The conversion press uses some 6 stations and 22 stages to form the completed end. It runs at about 2,000 ends per minute. Figure 9.5 shows a can end illustration and typical dimension nomenclature.

The two-piece can format is widely used in the US and European markets, where high-volume throughput is assured. In smaller markets the drinks can is made using three-piece can technology (to form the body and two ends). The body is made from tinplate, as is the (plain) base end. The easy-open top end is made from aluminium. It is not unusual to see shaped three-piece cans in the beer or syrup markets, for example, Tesseire's Sirop in France. These cans are usually shaped mechanically by expanding the can internally using a rubber former against an exterior moulding shape. This process is relatively slow compared with the output speed of two-piece technology, which is a factor in the delay in market expansion.

Since two-piece can technology is used for high-volume output, the sizes are standardised for international use. It is only the volume can users (usually the

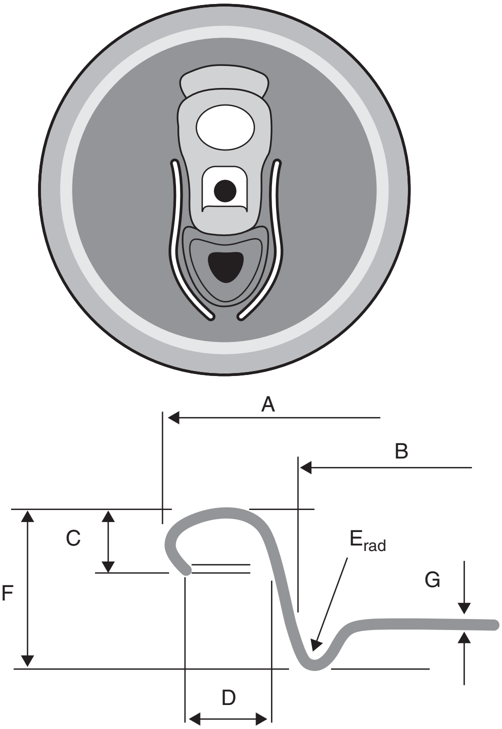

Figure 9.5 The can end. (A) curl diameter; (B) countersink diameter; (C) curl height; (D) curl opening; (E) countersink radius; (F) countersink depth; (G) nominal material thickness.
Source: Reproduced with kind permission from Continental Can Europe.

cola manufacturers) who can initiate customisation. Developments have indicated that plastic (PET) blow-moulding technology might lend itself to reforming lightweight metal two-piece cans since it uses high pressure (i.e. 40 bar) and produces small containers at high speed (up to 40,000 per hour).

Since three-piece can technology uses flat-sheet tinplate, all the high-quality printing options are available to the designers. This means they can use photographic-quality reproductions, for example, create surface effects by mixing areas of gloss and matt or use special effect varnishes that produce textured finishes. Since the sheet is printed when flat, close registration of the graphics is possible and this, when combined with surface effects, can produce a striking visual result. The sheet can also be printed in more than one pass.

Two-piece cans are printed 'in the round', in one pass, after they have been formed. First, the cans are coated externally with a clear or pigmented (normally white) base coat, which forms a good surface for the printing inks. After drying, the cans are printed with up to six colours, plus a varnish. The base of the can body is also protected by the application of a coat of varnish. Further protection is added by spraying the internal surfaces of the can with a lacquer to protect the can from corrosion and its contents from any possibility of interaction with the metal.

Protecting the can from the effect of corrosion is very important in order to achieve the very long shelf life that metal cans offer. Discussion with the can manufacturer will ensure the use of the appropriate grades of lacquers on the internal surfaces to prevent primary corrosion. The external surfaces must also be considered in order to prevent secondary corrosion, which will result in leakage. Handling on the filling line must ensure the smooth flow of cans and eliminate any sharp objects that may scratch or pierce them. A lot of moisture is present on the line because of the use of conveyor lubricants or from the pasteuriser, and it is important that cans are dried before being packed, particularly if they are to be shrink-wrapped. Cold cans, below the dew point, must be warmed to ambient temperature or else they will become wet when packed.

It is not only the can body that has to be kept free from corrosion; the easy-open end must also be protected. This end is scored through to create a locally thinned line of metal – the tear line when the can is opened. If this area of the can is subject to stress corrosion, then rapid leakage can occur.

Naturally, storage conditions such as daily temperature fluctuations and high humidity are also important.

9.6.1 Liquid nitrogen injection

Since beverage cans, particularly two-piece cans, are made with very thin side walls, their ability to resist vertical top loads is limited. It is not until they have been filled with carbonated product and the product has reached room temperature that cans achieve full top-load strength. For non-carbonated products this can be a problem; however, there are systems available which can inject a precise volume of liquid nitrogen into a filled can, just before the end is seamed on to the can body. As the liquid converts to a gaseous state, it expands. This helps to expel excess oxygen from the headspace, which may otherwise affect shelf life, and provides the internal pressure required for side-wall strength and package stability.

By adjustment of the injection time and duration, PET bottles too can be pressurised in this way when they are used for non-carbonated products. The advantage of nitrogen is that the gas does not go into solution with the product to change a still product into a fizzy drink.

9.6.2 Plastic cans

With the significant development of PET in the beverage market even the can format has been challenged; thus, for example, some UK mineral waters are packed in PET cans. Some years ago a significant effort was made to develop a PET two-piece can, using similar technologies to have for metal cans to produce high-output plastic cans. Unfortunately, the shelf life of such a small container with a high surface-to-volume ratio (and higher product cost) proved to be a costly weakness. However, other PET manufacturing technologies exist to produce smaller volume outputs to suit premium drinks markets for lightly carbonated products.

The real benefit of PET cans is not replication of a metal can but the ability to produce a shaped can in sizes not found in metal versions. The cost of an aluminium easy-open end for a metal can is relatively expensive compared with the 28 mm plastic closure used for a PET container. However, there is a market for which the can format proves to be attractive. The continuing development of the full-aperture easy-open end may, one day, result in a can end for drinks that, when combined with PET, would produces a 'glass' from which the drink can be consumed.

9.7 Cartons

Typical brick-shaped cartons (aptly named 'briks') are synonymous with the name of TetraPak and TetraBriks. Laminated board cartons have been available for 70 or more years for liquid products (Figure 9.6). Developed in the dairy market, they are a major packaging format for still drinks; some markets are dominated by the single-serve brik and others by the multi-serve version. The other significant factor for these drinks cartons is that the system is available worldwide, making global production of new products much easier.

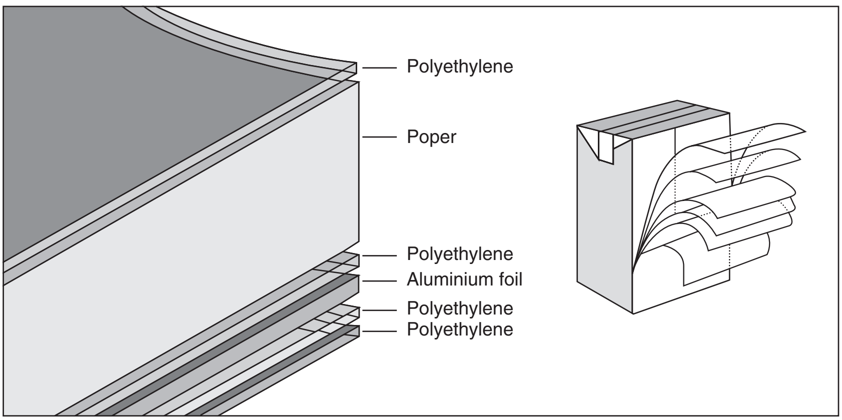

Figure 9.6 Structure of a laminated board carton.
Source: Reproduced with kind permission from TetraPak.

The development of aseptic technology has radically opened up new possibilities for efficient and economical handling of perishable liquid products. Aseptically processed and packaged juices will keep for months at room temperature in sealed packs. The key advantages of aseptic cartons are that

- they can be stored and distributed without refrigeration;
- they keep for a long time with no added preservatives;
- the quality of their contents remains outstandingly high.

The packaging material is a laminate made from layers of paper, plastic (polyethylene) and aluminium foil. The particular composition for a given product is chosen according to its specific requirements. This affords the best protection to each product and keeps it in peak condition until consumed. Polyethylene is laminated to the board to make it impermeable, and an aluminium foil (of 10 μm or less) is added to increase the barrier properties against external hazards such as sunlight and atmospheric oxygen, which would cause deterioration of product quality.

The entire surface of the laminate is sterilised with hydrogen peroxide before it is filled and shaped into packs. This has proved to be a very efficient and safe technique. The filling takes place in a sterile environment which is small and has few moving parts. These are important factors that contribute to the integrity of the system. The packs are sealed through the liquid, which means they are completely filled and the contents are thus fully protected against oxidation. The brik format makes very effective use of materials and economical use of bulk volume in palletisation and distribution: 93% is product and only 7% is primary and secondary packaging.

Each product can be given its own distinctive profile by choosing a particular combination of package volume, shape and printing technique. The general printing methods available are flexography, rotogravure and offset. In general each aseptic filling machine can handle a fixed-base footprint profile. Heights can then be changed to suit requirements. However, each machine will accommodate only a limited range of heights; thus one might will produce a single-serve-size range, another might produce a mid-size range and another the larger multi-serve sizes. This ‘inflexibility’ causes problems for some manufacturers, particularly those who want to launch a new product in a small (200/250 ml) single-serve size and in a large (1 l) multi-serve size, since they would need two machines. This would require either high capital entry costs or two co-packers. The makers of the new generations of filling machines have recognised this opportunity for flexibility and use modular, changeable parts to offer a wider size range from one machine (Figure 9.7).

The multi-layer structure of cartons continues to be an environmental issue. However, much has been done to develop recyclable technology for drinks cartons. One output uses a chipboard-effect material of the type used to make furniture; at the other end of the lifecycle is incineration with reclaimed heat.

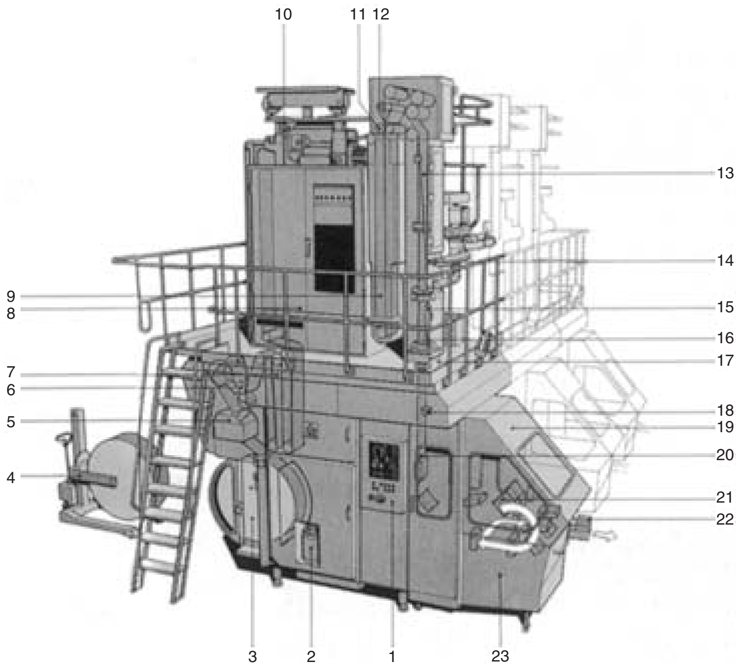

Figure 9.7 Machine functions. (1) control panel; (2) container for hydrogen peroxide, closed system; (3) reel of packaging material; (4) special trolley with hydraulic lift for handling packaging material; (5) automatic splicing equipment for packaging material; (6) date-stamping unit; (7) loops of packaging material, (these ensure a smooth, jerk-free feed and also allow continuous production when new packaging material is spliced in); (8) most of the machine's electrical system is located here; (9) the packaging material is sterilised in a deep bath of heated hydrogen peroxide; (10) strip applicator, which applies a plastic strip to one edge of the packaging material (later, at the longitudinal sealing stage, this is welded to the opposite edge, giving a tight and durable seal); (11) rollers, which remove the hydrogen peroxide from the packaging material; (12) nozzles for hot sterile air to dry the packaging material; (13) the packaging material starts to be shaped into a tube; (14) filling pipe; (15) element for the longitudinal seam that welds together the two edges of the packaging material; (16) 'short-stop' element that completes the longitudinal seam when the machine restarts after any brief halt in production; (17) the TBA/8 is designed so that two or more machines can be linked to form compact production units sharing a common platform; (18) photocells, which control the machine's automatic design correction system; (19) casing, which can be raised and lowered, covering the automatic external cleaning system and the final folder, where the top and bottom flaps are folded over and sealed on to the package; (20) the packages are sealed beneath the surface of the liquid using induction heat (the heat comes from jaws that also shape and cut off the packages); (21) in the final folder the top and bottom flaps are sealed on to the package in two lines; (22) discharge of the finished packages; (23) bath, which fills with water and detergent automatically for external cleaning of the machine.
Source: Reproduced with kind permission from TetraPak.

In the first step of the formation process a web of laminated material is fed through the filling machine, which sterilises the material at 70°C in a bath of hydrogen peroxide (H_2O_2). It is then dried with sterile hot air and is completely dry when it reaches the filling zone. The web is kept in a sterile area as it passes through the machine, where it is formed into a continuous tube (Figure 9.8). The liquid to be filled is poured into the tube, which is then sealed through and below the level of the liquid, using heated bars. This produces a continuous flow of sealed 'pillow pouches' (Figure 9.9) that are completely full, with no headspace. The machine then uses formers to shape the brik container along predefined creases in the laminate. Finally, the top and base 'ears' are folded down and stuck to hold the brik in shape.

The correct positioning of the artwork on each package is guaranteed by a sophisticated design-correction system (Figure 9.10). Photocells monitor the position of the artwork and transmit information to the design-correction system. If necessary, this system corrects the artwork positioning during production.

For products that need to be shaken before opening, the pouch cannot be filled completely; a headspace must be left in the pouch. This is achieved by sealing the package through the product flow before the pouch is completely filled.

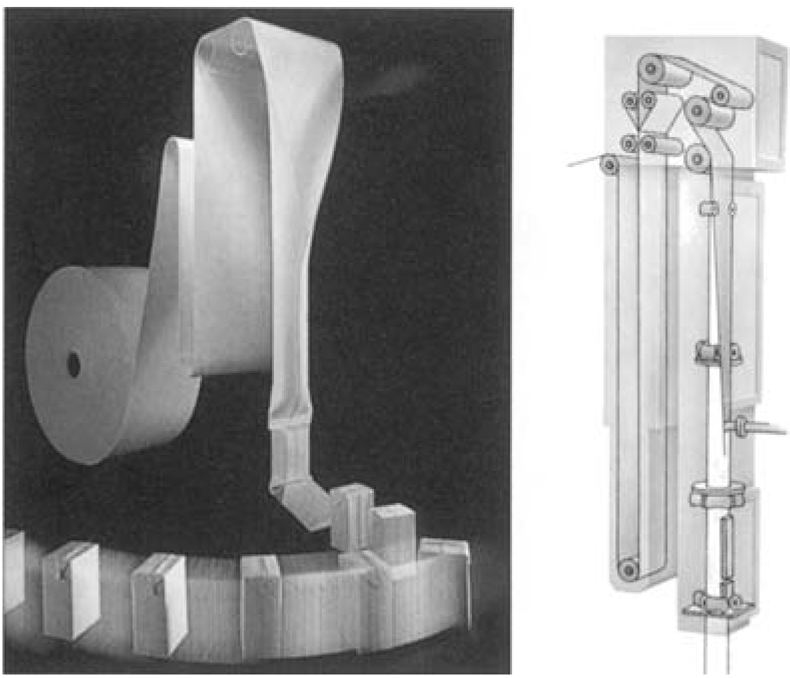

Figure 9.8 Formation of laminated cartons.
Source: Reproduced with kind permission from TetraPak.

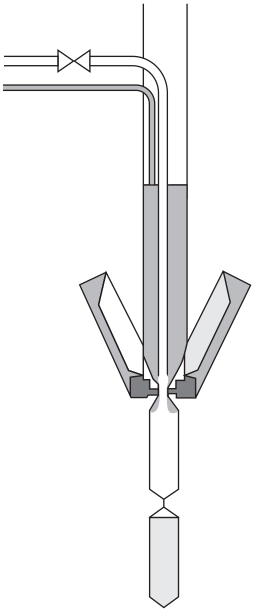

Figure 9.9 Filling laminated cartons.
Source: Reproduced with kind permission from TetraPak.

Figure 9.10 Photocells monitor the position of artwork on packages.
Source: Reproduced with kind permission from TetraPak.

Pack openability has been an issue over recent years, and suppliers have done much to include plastic closures to give ease of access to multi-serve sizes as well as some form of resealability. Packages can be cut open or opened by tearing along a perforation. Two types of perforation are possible: wave-shaped and high-fin. Both produce a positive spout for pouring and the high-fin perforation makes it easier to reclose the package once opened. This opening is the simplest from a construction point of view and is suitable for multi-serve packs remaining in one environment, that is, a fridge.

Often a pair of scissors is needed to open the package along the perforations (Figure 9.11a); a better alternative is a foil pull-tab, which opens a keyhole pouring hole (Figure 9.11b). This type of opening is easy to use, although not

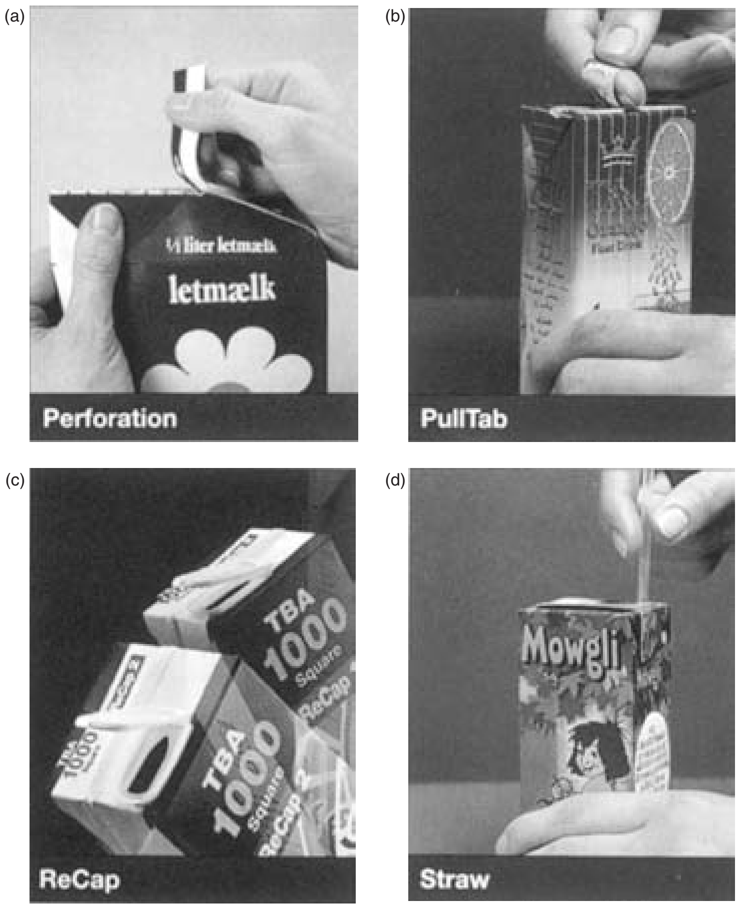

Figure 9.11 Examples of types of openings: (a) perforation; (b) pull-tab; (c) recap; (d) straw. *Source*: Reproduced with kind permission from TetraPak.

resealable, and can be found in a wide range of package sizes. As reclosure or resealability is an attractive feature in the larger package sizes, recent developments have added a plastic resealable cap for convenience (Figure 9.11c). The primary seal remains the foil, which is either removed or ruptured by pressing down an integrally moulded feature in the cap.

An obvious opening feature used in the single-serve pack sizes is the drinking straw (Figure 9.11d). A small hole is made in the outer surface of the carton to reveal the foil barrier layer, and a plastic drinking straw is attached to each package. The consumer removes the straw from the carton and its own sealed wrapping and then punctures the package using the angled end the straw and pushes the straw into the package. The size of the hole in the carton is such that it holds the straw tightly and minimises leakage. Even if the package is tipped over very little product leaks out through the straw. This is a simple and effective package; however, it does require the consumer to be aware of how to handle a less rigid package while attempting to gain grip to press home the drinking straw.

The gable-top carton has become identified with 'fresh' juices. The 1 l pack has a very good display area for graphics, fits the fridge door easily and is easy to hold and pour from. Some filling machine developments have meant that gable-top cartons can be filled aseptically.

Packaging of products in aseptic drinks cartons make retail handling and display easier and more economical. They do not need to be refrigerated, so they can be placed anywhere in the store. There are many packing options for distribution, and the printed design of the package can be developed to help sell the product whether it is displayed on shelves or in whole pallet loads. A number of collation systems are standardly available for drinks cartons for collection in threes, sixes, nines or larger numbers.

9.8 Flexible pouches

Flexible pouches offer new opportunities for many still juices. Essentially a cold- or hot-fill system, pouches can be made in a variety of sizes without a large capital cost because most pouch fillers support the pouch by the neck and therefore one machine can be used for all sizes. Since the film laminate is decorated while flat, most printing systems are available to the designer, as are the lamination formats widely used for other pouch or sachet types. The main material used is metallised PET laminated to aluminium foil as well as low-density polyethylene, which is used as the sealing medium. For high-temperature retort processing, a polypropylene-laminated film is also available. A free-standing pouch (Doy type) is used for one-trip usage. This has a straw to pierce a specially formed area of the pouch.

In Europe, the Cheerpack (made by Guala of Italy) is a pouch pack derived from a Japanese system. It is a free-standing pouch pack made from four pieces of film joined at the edges. An injection-moulded neck is sealed into the top portion of the pouch. This produces a resealable pouch which has found good acceptance in the sports drinks market as it is unbreakable and fits easily into a kit bag. Schools also find these packs acceptable as they are easy to dispose of when empty.

If packaging waste legislation is based on the weight of packaging, then plastic laminate packs will be an interesting format to study. Although the laminate itself may attract a higher levy, the actual pack weight (around 9 g for a 330 ml pack) may incur only a low tax.

9.9 Secondary packaging

Secondary packaging plays an important role, often underestimated, in collating, protecting and providing identification for and about the primary packaging it supports. In wholesale outlets such as (the cash and carry) the secondary package becomes the primary package and therefore it must work at that level, that is, be eye catching, functional and represent the added value of the consumer units it carries.

The trade outer is often forgotten about at the time the primary package is designed. In fact, the reverse practice should be followed and the outer should help in determining the major dimensions for the primary package: ensuring efficient loading through the supply chain can be a good cost-saver.

For many plants that supply a pan-continental market, pallet sizes are often a source of complexity because collated primary packs are not modular for the whole pallet range. Within Europe it is not unusual for multiple retail outlets to specify the minimum pallet utilisation level to be achieved. It is interesting that many drinks carton dimensions have been chosen to be modular, and this should also be done for other pack formats. By taking secondary packaging into account early in the packaging design and material selection process, a speedier and more economical result will be achieved.

In many multiple-store distribution networks, pallet loads are broken down into smaller, mixed-product units called ‘dollies’ to be distributed to individual stores. This means that trays or cartons of primary packs may be stacked (possibly unevenly) above or below heavier items. Thus, case care must be taken in selecting the secondary format. If the primary pack is unbreakable, then the role of its corrugated tray should be closely examined: is it there just to collate packs prior to shrink-wrapping or does it provide a valuable feature at the point of sale? The secondary packaging may need to be a sales enhancement feature in its own right or it may have to be unobtrusive enough not to distract from the decoration and marketing message on the primary package. It is disappointing to see a well-decorated primary package half hidden by a deep, plain corrugated tray. On the other hand, a well-selected and highly decorated tray can provide a visually attractive point-of-sale item.

There are several software packages that will calculate pallet layouts for various secondary pack formats, as well as offering suggested specifications for corrugated or board outers. They will also calculate pallet utilisation and the subsequent pallet layouts for sea containers, even for mixed loads, and for hand-balling cases into the container.

Filling lines have case or tray erectors that will handle only certain sizes or formats. It is important to understand which of these are available when undertaking the feasibility and capability phases in the product development cycle.

9.10 Pack decoration

This section covers package decoration formats rather than the detail of their printing. Packaging that is delivered pre-printed, such as cans and drinks cartons, is covered in Sections 9.6–9.8. Decoration formats include labels that are stuck on to packages and heat-shrink plastic sleeves, which may be applied through a labelling technology or as a pre-made sleeve and heat-shrunk into place.

Labels (made of paper or plastic) are usually applied to the filled package by a labelling machine. If the package has been filled below the dew point, then the package is likely to be wet by the time the label is applied. Special adhesives are therefore used and suitable advice can be provided by adhesive manufacturers.

Labels must be applied to a flat or two-dimensional surface. Compound curves result in the label creasing and looking unsightly. A label can be a patch, which may be square or rectangular or shaped into a particular profile. Shaped labels are usually used for products with a premium positioning.

Label application can also be reel-fed. As the name implies, the labels are straight edged and are supplied to the labelling machine in a continuous reel. The labelling machine applies the adhesive, cuts the label from the reel and applies the label to the package. Newer reel-fed labelling technology allows a plastic label to be applied to a contoured package and heat-shrunk into the contoured shape. A typical application for this is on beverage cans, which are predominantly cylindrical, so that the label only has to shrink in at the top and bottom of the can.

Pre-made heat-shrink sleeves are widely used on plastic beverage bottles for all-over decoration that can make a plain bottle look attractive. This also allows one bottle shape to be used for more than one product or more than one flavour. When considering sleeves care needs to be taken over whether they should be applied to the container before filling or after filling. Application before filling means that the filling operation can be completed at normal line speeds. However, if heat-processing of the filled container is required, this should be discussed with the sleeve supplier as special specifications may be required. Sleeving after filling will require the filling operation to include a heat-shrink tunnel and sleeve applicator. This can have an impact on the overall line speed and capital investment for a new product.

9.11 Environmental considerations

The environment impact of packaging is an emotive, controversial and yet important topic for discussion. Readers should make themselves aware of the local issues that can affect the product and package in question. Are there issues affecting the packaging material itself, or the packaged product, or the resultant waste or litter? Are there issues which affect the use of land-fill, or the recycling of the original packaging material back into the original packaging format or the conservation of renewable resources? Returnable/refillable packaging can have applications where there is a closed-loop supply chain on ensuring an effective (>30%) rate of return of empty containers and effective use of energy in returning the containers and cleaning them ready for reuse. Such containers, which are available in some European and US markets, are usually much heavier than single-trip containers and there have to be sufficient numbers in the pipeline to service the total loop. Glass is often used for recyclable containers. It is a very good material for reuse, however, glass is not always the best material for beverage products in sensitive locations such as sports and swimming areas or the beach, where broken glass can be a significant hazard. Aluminium is recognised as a good material for recycling, with many schemes in the world supporting the recycling of aluminium beverage cans. A local scheme in the United Kingdom in 2004 offers to plant a tree for every one tonne (1,000 kg) of aluminium cans collected. The scheme that claims that 'recycling aluminium saves 95% of the energy used in making it from the raw materials. Aluminium can be recycled again and again and, because it is such a valuable material it is such a waste to throw it away. Yet much of it gets thrown away in the waste bins.' However, in Denmark all metal drinks containers are effectively banned (see www.bottlebill.org/geography/worldwide/denmark). Even drinks cartons claim to be an effective use of wood-based materials, which come from renewable sources, before the wood energy is reclaimed in heat recovery incinerators. A report published in the United Kingdom in 2004, commissioned by the Industry Council for Packaging and the Environment (INCPEN), suggests that many people could be wasting their time by recycling: 'consumers are led to believe that recycling materials and choosing certain types of packaging is of significant benefit to the environment'. The report suggests that these benefits are minor when compared with the energy-use implications of the type of car we drive and the way we heat our homes. Packaging is indeed a controversial topic. Whatever one's point of view, packaging must be used effectively to preserve and protect the product. The energy used to produce packaging materials is much less than that used to produce the product they contain and that used to distribute the product (often to far places around the world). For that product to become unfit for sale due to the inappropriate use of packaging would be a far greater waste of energy.

Packaging has been on the environmental agenda for many years within Europe. A number of European countries have been very active in legislating against certain plastic materials (PVC) and one-trip packaging. European Union legislation is now having an impact on all member countries, and since 1 January 1997 all businesses in the European Union that manufacture, fill or sell in excess of 50 tonnes a year of packaging or packaging materials have been subject to certain regulations. The official name given to this legislation in the United Kingdom is the Producer Responsibility Obligations (Packaging Waste) Regulations. These regulations stem from the requirements of the EU Packaging and Packaging Waste Directive.

The purpose of the directive is to stop countries favouring one type of material or type of packaging in order to protect their local businesses. This 'single market' objective is the main reason for the directive, but it also aims to raise the level of recovery and recycling of packaging waste. Some countries still have discriminatory measures in place (and others are introducing them); so the main objective has not been realised while industry has paid significant sums to increase recycling to a level of over 50% across Europe. In the United Kingdom, businesses will have to meet certain targets based on the weight of packaging they handle. In the regulations, packaging is defined as 'any product which is used or is intended to contain and protect goods, or to aid their handling, delivery or presentation'. This covers all stages of packaging production from manufacture and distribution to delivery to the final user or consumer.

The regulations set a target of a minimum of 60% of packaging to be recovered by 2008. For the purposes of these regulations 'recovery' includes a number of processes that result in a net benefit being derived from used packaging. It includes recycling, energy recovery from heat generated during burning and composting of packaging. 'Recycling' is a more precise term for the reprocessing of used packaging so that the material produced can be reused for its original or another purpose. Composting (organic recycling) is counted as recycling.

Reusable packaging is exempt from the regulations. Reusable packaging might be defined as a package that is used at least three times (this definition is not part of the published regulations; clearly a single trip does not count as reusable, so three might be a minimum). Evidence has to be provided that the required trippage has been made. Within Europe, there are a large number of returnable/refillable bottles, mainly glass. However, PET has made inroads into this market. Unlike glass, PET is permeable and rigorous checks have to be undertaken on returned bottles to ensure that the bottles have not been filled with noxious products or other drinks with strong flavourings that will be retained by the PET. Filling lines have automatic 'sniffers' that search for the exact product with which the bottle was originally filled. If this 'smell' is not detected, the bottle is rejected. Mineral water bottles suffer high levels of rejection and are recycled as soft drinks bottles.

More interesting information can be found from the Container Recycling Institute's Bottle Bill Resource Guide at www.bottlebill.org, and also in the chapter on environmental considerations by Gary Parker in the *Handbook of Beverage Packaging*. INCPEN produce some very good details about packaging waste and are worth contacting for more current information.

9.12 Conclusions

Packaging for liquid beverages is a large and complex area. This chapter covers the topics in an informational way and is not intended to be a comprehensive document. For more details it is recommended that the reader contacts the various manufacturers, The Institute of Packaging (IOP) in the United Kingdom, the Institute of Packaging Professionals (United States) or PIRA International. The IOP runs very good packaging courses leading to a certificate or diploma in packaging and membership of the institute.

Some suitable institutes and trade associations are

The Institute of Packaging
Syonsby Lodge
Melton Mowbray
Leicestershire LE13 0NU, UK
Tel: +44 (0)1664 500055
Fax: +44 (0)1664 564164
www.iop.co.uk

Industry Council for Packaging and the Environment (INCPEN)
Soanepoint
6–8 Market Place
Reading RG1 2EG, UK
Tel: +44 (0)118 925 5991
Fax: +44 (0)118 925 5993
www.incpen.org

PIRA International
Randalls Road
Leatherhead
Surrey KT22 7RU, UK
Tel: +44 (0)1372 802000
Fax: +44 (0)1372 802244
www.pira.co.uk

Metal Packaging Manufacturers Association (MPMA)
Elm House
19 Elmshott Lane
Cippenham
Slough
Berkshire SL1 5QS, UK
Tel: +44 (0)1628 605203

British Bottlers Institute
PO Box 54
Gosport
Hampshire, UK
Tel: +44 (0)23 9252 5018
Fax: +44 (0)23 9252 5018
www.bbi.org.uk

The Institute of Packaging Professionals
1601 North Bond Street
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Tel: +1 630 544 5050
www.iopp.net

RAPRA Technologies Ltd
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Tel: +44 (0)1939 250383
Fax: +44 (0)1939 251118

The Can Makers Information Service
1 Chelsea Manor Gardens
London SW3 5PN, UK
Tel: +44 (0)20 7351 2400

British Soft Drinks Association
20–22 Stukeley Street
London WC2B 5LR, UK
Tel: +44 (0)20 7430 0356
Fax: +44 (0)20 7831 6014
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Further reading

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10 Analysis of soft drinks and fruit juices

D.A. Hammond

10.1 Introduction

Although marketing and 'image' have a lot to do with consumers' desire to try a product, if they do not like the flavour they will not purchase it again. In addition, if the quality, odour or flavour of the product is variable, it will not live up to consumers' expectations and it is again likely that they will not be regular consumers of the product. This means that it is critical to control the flavour of the product. This can be done in a number of ways, including sensory assessment and the analysis of certain key ingredients, such as sweeteners, acidity, colour and, in a carbonated soft drink, the level of dissolved carbon dioxide.

If a formulation uses high-intensity sweeteners, preservatives or colours, it is critical to ensure that the levels of these materials in the product do not exceed the statutory limits. Although the fortification of some products has been common practice for many years, the addition of vitamins to soft drinks has only recently become common. In the United Kingdom, this is likely to be attributable in part to the phenomenally successful launch of the Sunny Delight brand, which has prompted the launch of a wide range of 'me too' products. Here again there is an analytical need to ensure that the vitamin levels claimed on the label are met at the end of shelf life.

There is also a growing interest in drinks with associated special characteristics. These products will often contain sugars for energy, caffeine for stimulation and vitamins, amino acids and maybe herbs which impart some unique selling property as a health benefit. A very good example of this type of product is Red Bull, which is described, according to its website, thus: 'Red Bull is much more than a soft drink – it is an energy drink. It was made for moments of increased physical and mental stress and improves endurance, alertness, concentration and reaction speed. In short: it vitalizes body and mind.'

The great success of this brand since its launch has led to the production of a wide range of other products based on the same concept of stimulation and improved alertness. These more complex drinks formulations can pose a significant analytical challenge. This is especially so if the product contains a mixture of herb extracts and there is a requirement to confirm that each one is present.

Thirty years ago there would have been a limited set of equipment in the quality control laboratory of the majority of fruit juice and soft drinks factories. This would probably have consisted of a refractometer, a burette, a spectrophotometer, an instrument to measure the level of carbonation and some equipment for checking the microbiological integrity of the product. However, with the move to larger and more efficient factories with faster bottling lines, it has become critical to ensure that a bottling formulation is correct before a product is packed. These developments mean that in today's factory if a product's formulation is prepared incorrectly and this is not detected it will be a very expensive mistake to rectify after the product has been packed. Complementary to this is the fact that general analytical equipment, such as high-performance liquid chromatography (HPLC) systems, has become more reliable, requires less-skilled operators to use it and is also relatively cheaper than it was 20 years ago. These developments mean that it is now common to see sophisticated equipment in a factory's quality assurance laboratory so that a bottling formulation can be checked for the basic parameters such as Brix, acidity, high-intensity sweeteners and preservatives before packing.

Since the publication of the first edition of this book, a few more validated methods for the analysis of soft drinks ingredients have been documented. When the first edition was published in 1998, only a handful of methods for the analysis of soft drinks ingredients had been collaboratively tested in the Association of Official Analytical Chemists (AOAC) official methods manual, and only two of these were modern HPLC approaches. At that time, no methods could be found in the British Standards catalogue. Inspection of the British Standards website (http://www.bsi-global.com) now shows that there are two standardised approaches for the analysis of high-intensity sweeteners in soft drinks, both of which use HPLC. This overall lack of standardisation of methods is probably because a soft drink's matrix is relatively straightforward, without many of the problems associated with other areas of food analysis, and so the industry has not felt the need to standardise the test methods.

Although there are only a limited number of methods that have been validated specifically for soft drinks, there are around 80 validated methods available for the analysis of fruit juices, most of which would work equally well for soft drinks. These methods are published in the International Fruit Juice Union (IFU) handbook of analytical procedures, which offers the best reference collection of methods for the analysis of fruit juices in the world, with new methods added on a regular basis (Anon, 2004a). The IFU's collection of analytical methods covers most of the main procedures required to assess the quality and authenticity of fruit juices and nectars. The methods are listed on the IFU's website (http://www.ifu-fruitjuice.com); at the time of writing they cannot be purchased directly from there, but they can be obtained from the Swiss Fruit Union, Zug, and details of how to do this are given on the website. It is possible that at some time the methods will be made available directly from the website.

A number of these IFU methods have provided the basis for those published for fruit and vegetable juices by CEN, the European standardisation organisation, which have been adopted by the member states of the European Union as national standard methods. There are 24 listed on the British Standards website (http://www.bsi-global.com). They can be purchased from this website and should also be available from other standardisation organisations within the European Union and affiliated states such as Switzerland. They are also likely to be adopted as Codex methods when the standard for juice is finally approved and published. Although some of these IFU procedures, such as titratable acidity in a carbonated product, require modification for use in soft drinks systems, most are equally appropriate for the analysis of soft drinks.

This chapter will cover the analysis of the different key elements of a soft drink formulation or a juice, for example, sweeteners and preservatives, and the procedures for their assessment. When both soft drinks and juices contain a particular ingredient or component, their analysis is discussed together. Where there is no overlap, soft drinks and juices are covered separately.

Although it is beyond the scope of this chapter to discuss in any great detail methods used to assess the authenticity of fruit juices, a brief summary of this area is given at the end. In the first edition of this book, an introduction to HPLC was given to assist newcomers to the field, but it is now felt unnecessary as this has become such a standard procedure.

10.2 Sensory evaluation

The sensoric assessment of soft drinks and fruit juices is discussed briefly here for completeness.

As the flavour and odour of a soft drink or fruit juice are very important elements of a product they should be closely controlled. This is generally carried out by trained panellists who have been screened to ensure that they have an aptitude for this type of assessment; they are often quality assurance personnel or workers from the factory. Sensory assessments should be carried out in surroundings where the panellists can concentrate without distractions.

Every batch of finished product, should be checked to ensure that it tastes ‘normal’ (i.e. is free from off-tastes). Although the tasters should be familiar with the product’s flavour and odour they should always be provided with an approval control for reference purposes. Incoming raw materials such as sugar and water should also be assessed to ensure that they will not impart any off-tastes to the finished product.

10.3 Water

In a soft drink, or a fruit juice reconstituted from concentrate, the quality of the water is an essential element. Checking the water quality includes assessment to ensure that it does not contain any off-tastes or odours. It also involves checking that any water-treatment processes have been effective and have not introduced defects into the water. The water should also be assessed to ensure that it does not contain materials that are likely to precipitate from the product on storage. Such precipitates are often called ‘flocs’.

The most common cause of floc formation in a clear soft drink is microbiological growth (yeasts). If this occurs the yeasts can be identified by allowing the floc to settle in the container, or precipitating it by centrifugation, and very gently decanting the liquid or syphoning off the product. The precipitate can then be placed on to a microscope slide for examination. The yeast cells will be seen as large (*c.*5 μm) ellipsoids.

Another cause for a floc, particularly in the autumn, can be the presence of algal polysaccharides in the water. Although these polysaccharides are soluble in water at neutral pH values, they will precipitate from solution when the water is made acid. To check for the presence of polysaccharides in the water the following test should be carried out.

To a sample of raw water (200 ml) concentrated hydrochloric acid (2 ml) is added to ensure that the pH is below 2. The water is held at 608C for 12 h and then examined against a dark background under strong illumination to detect any floating particulate materials. The heating period can be extended for a few days to ensure there is no precipitate.

On a less frequent basis, and as part of good manufacturing practice, samples of water should also be analysed more thoroughly for a wider range of components, including the heavy metals (e.g. arsenic, copper, chromium, cadmium, lead, mercury, selenium and zinc) and polyaromatic hydrocarbons (PAHs), some of which are known to be carcinogens. The levels of fluoride and nitrate ions in the incoming water should also be checked periodically. The heavy metals and PAH analyses are both very specialised and are better left to specialist laboratories. However, analysis for fluoride and nitrate levels can readily be performed using ion exchange chromatography linked with conductivity detection. A typical example of this is given in Dionex application note 25 (Anon, n.d.a) and a chromatogram of a standard for this type of separation is given in Figure 10.1.

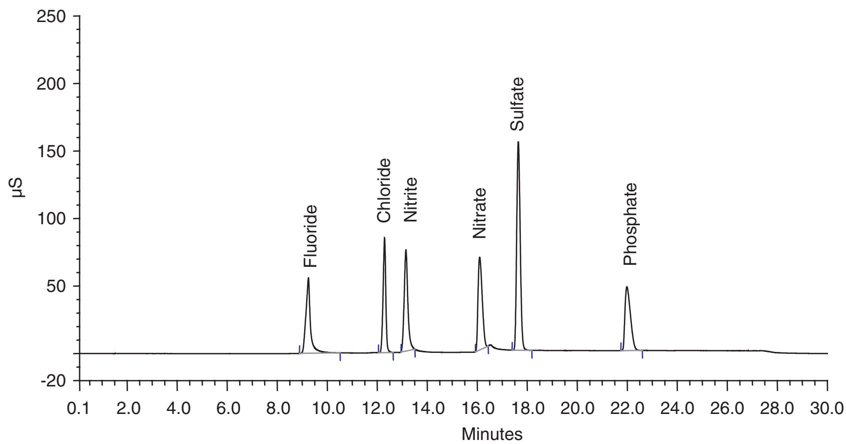


Figure 10.1 Ion-exchange chromatographic separation of main anions found in water using a Dionex HPLC and AS-11 HC column. Conditions: column: Dionex AS-11 HC 250 × 4 mm; solvent Milli-Q water and 3 mM NaOH for 6 mins then to 30 mM NaOH over 15 min; flow rate 1.5 ml/min; suppressed conductivity detection.

10.4 Sweeteners

After water quality, sweetness is probably the most important feature of a soft drink. In fact, until 1995 in the United Kingdom it was essential that a soft drink contained a minimum level of sugars. This level was set at 45 g/l unless the product was listed as a 'low-calorie' soft drink (Anon, 1964; UK Soft Drinks Regulations 1964). These regulations have been revoked (Anon, 1995a), and it is now possible to make a soft drink with or without added sugars, if required, provided the product is appropriately labelled.

The sweeteners used in soft drinks can be divided into two main categories. These are the natural sweeteners, such as sucrose, invert syrups, corn-derived syrups and honey, and the high-intensity sweeteners (artificial sweeteners) such as saccharin, aspartame and acesulfame K. In most fruit juices and many soft drinks, except diet varieties, sugars are a major component of the product.

All fruit juices contain glucose and fructose and some sucrose, but the proportions of these sugars will depend on the actual fruit. In soft drinks the type of sugar used in a formulation will often depend on where the product is being produced. In EU countries, where the price of sugar is adjusted to support local production, the sugar used to prepare soft drinks will often be sucrose or a sugar syrup prepared from beet or possibly cane. However, in the United States most soft drinks are prepared from syrups derived from starch, such as a high-fructose syrup prepared from corn (HFCS), as these syrups are cheaper than sugar derived from cane or beet.

High-intensity sweeteners are used in diet formulations but they also often appear in regular soft drinks. Their use depends on the requirements of the manufacturer and also on the relative prices of sugar and the high-intensity sweetener(s) involved. This substitution was particularly common in the United Kingdom in the 1970s, when part of the sweetness of a product was often provided by the high-intensity sweetener saccharin to control the cost of a soft drink's formulation.

10.4.1 Analysis of natural sweeteners

The natural sweeteners used in soft drinks formulations are generally the same as those found in fruit juices, that is, sucrose, glucose and fructose. As sugars are generally the second largest component in a soft drink or fruit juice, one of the quickest and simplest ways of assessing if a product is within specification is to measure its refractive index. This can be achieved using a simple hand-held refractometer costing a few pounds or a sophisticated temperature-controlled refractometer, which costs several thousand pounds.

The assessment of the refractometric solids content, generally referred to as the Brix value, is one of the basic tests that is carried out in today's soft drinks and fruit juice factories to assess the soluble solids content of an incoming sugar syrup, a soft drinks bottling syrup before dilution, fruit juice concentrate or a diluted finished product. This measurement of soluble solids content is only an estimate as it assumes that everything in solution has the same refractive index as sucrose. Because of this assumption, it is normal practice to adjust the refractometric solids content of citrus juices for their acidity by application of a correction factor. This correction factor can be calculated using the following equation:

$$\text{acid correction to °Brix} = 0.012 + 0.193\,m - 0.0004\,m^2$$

where m is the total acidity obtained by titration to a pH of 8.1 expressed as anhydrous citric acid in g/100 g. In the case of soft drinks this is frequently the limit of analytical testing for sugars. However, it is common to check the levels of the individual sugars in a juice to assess the authenticity and quality of the product. Today most practitioners in this field use either HPLC or enzymatic methods to achieve this.

A wide range of HPLC methods has been used to detect sugars, and the literature includes a long list of papers on the separation and quantification of sugars using HPLC. As the sugars used in soft drinks formulations, except fructose, do not exhibit strong light absorbance in the ultraviolet region they are generally detected using a differential refractometer, which is a non-specific detector with a relatively low sensitivity (typically in the region 0.01–0.05%). This technique is ideally suited for the analysis of sucrose, glucose and fructose at the levels detected in soft drinks and fruit juices, which are usually in the region of 0.5–5%. The separation of the sugars can be performed on a range of different columns; only three are discussed below and any one of these would be appropriate for soft drinks analysis.

The first method employs the use of an amino-bonded silica column. On this column the free hydroxyl groups on the silica particles have been reacted and replaced by amino groups. This type of column, sold by most column manufacturers, will separate sucrose, glucose and fructose in about 10 min, as illustrated in Figure 10.2.

As with all methods, this approach has some limitations: it uses acetonitrile, which is toxic, and the separation of glucose from fructose can sometimes be problematic after extended use of the column. However, sample preparation is easy since it requires only dilution to the required level (often 1 : 10) and filtration prior to analysis to remove particulate materials, which protects and extends the useful life of the column. The degradation of the resolution between glucose and fructose is caused by the partial inactivation of the column by materials in the matrix, but this resolution can be recovered by reducing the acetonitrile concentration in the solvent. The same column can also be used to assay the level of ascorbic acid (vitamin C) in a soft drink or fruit juice, although different detection and solvent systems are used.

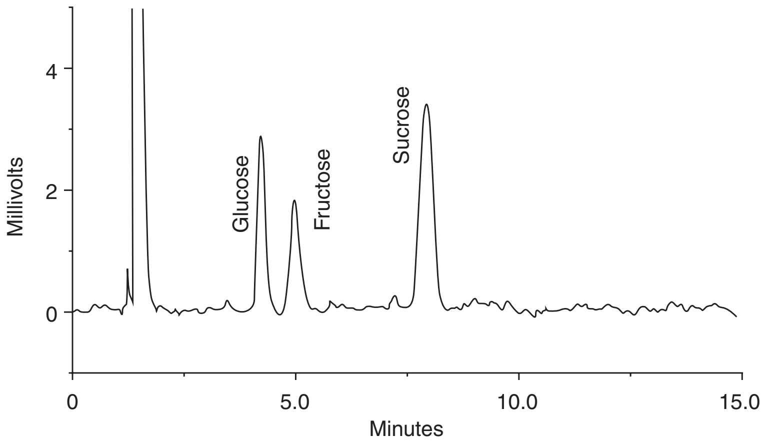


Figure 10.2 Separation of the main sugars found in orange juice using an amino-bonded silica column. Conditions: column: Highchrom 5 μm 250 × 4.5 mm amino-bonded silica; solvent = acetonitrile/water (8:2); flow rate 2 ml/min; refractive index detection.

The second method, using refractive index (RI) detection, is carried out using a resin-based polymer column. Sucrose elutes first from this column, followed by glucose, fructose and then sorbitol. This type of column is generally more robust than the amino-bonded column and if handled well will last much longer; however, it is around three times more expensive. The method has been collaboratively tested for the analysis of sugars and sorbitol in fruit juices by the IFU. The HPLC conditions are given below.

HPLC conditions: column: benzene divinylstyrene sulphonated resin in the calcium form 300 × 7.5 mm; solvent = 0.1 mM Ca EDTA in HPLC-grade water at 0.6 ml/min; at 80°C using RI detection.

The third HPLC method for analysing sugars uses a very different detector system, a pulsed amperometric detector (PAD). If sugars are analysed using a conventional electrochemical detector they very soon 'poison' the electrode and reduce its sensitivity. This problem is overcome with the PAD system, where the electrical potential applied to the cell is varied to automatically clean and recondition the electrode. This type of detector was pioneered by the Dionex Corporation in the 1980s for the quantification of sugars and other oxidisable materials. Figure 10.3 shows a chromatogram of an adulterated apple juice using this approach; the four major peaks that are visible are, in order of elution, sorbitol, glucose, fructose and sucrose (Anon, 1992). The column used for this analysis is a mixed-bed resin column containing both sulfonated and amino-bonded resin beads (PA-10). From the size of the sorbitol peak it is clear that this apple juice contains undeclared pear juice.

Sucrose (Bergmeyer & Brent, 1974a), fructose (Bergmeyer & Brent, 1974b) and glucose (Bergmeyer *et al.*, 1974) can also be quantified by the use of an

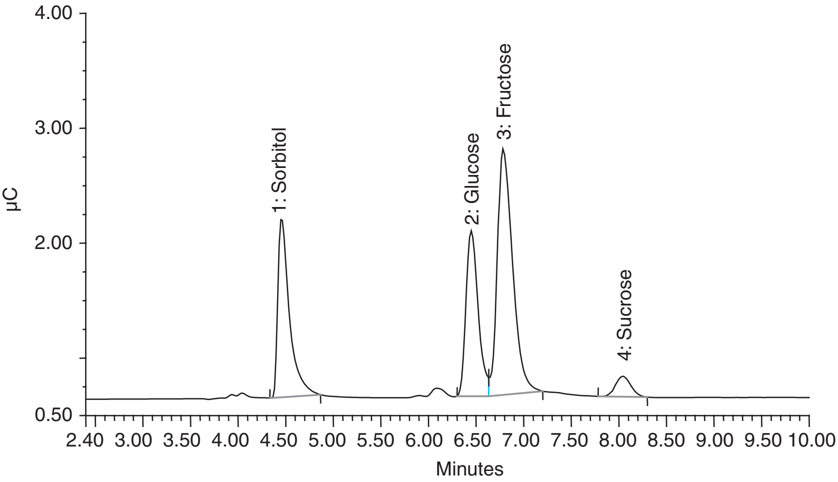


Figure 10.3 HPLC trace of a suspect apple juice run on Dionex HPLC using PA-10 column and PAD detection. Conditions: column: Dionex PA-10 250 × 4 mm; solvent = 100 mM sodium hydroxide in HPLC water; PAD detection; sample diluted 1:100 in water prior to filtration and analysis.

enzyme-linked assay. This type of approach is most commonly used in some areas of Europe (e.g. Germany) for the analysis of sugars in fruit juices or in laboratories that have a spectrophotometer but no HPLC equipment. The kits, developed by Boehringer Mannheim, for the enzymic analysis of sucrose, glucose and fructose are now sold by r-Biopharm. It is also possible to buy the individual chemicals and enzymes to carry out the assay (Anon, 1987a). These methods have been collaboratively tested for the analysis of fruit juices (IFU compendium of methods – see Anon, 1985, 1998a, b).

Generally, in fruit juices the enzymatic methods are slightly more reproducible for glucose and fructose than the HPLC procedure (results from IFU analytical commission ring tests). However, the HPLC methodology gives slightly better quantification of sucrose because the enzymic procedure uses a two-stage process that introduces more analytical variation. In the absence of an automatic enzyme analyser, it is quicker to use HPLC to quantify the sugars in fruit juices. If glucose, maltodextrin or inulin syrups are being used in a soft drinks formulation the total sugars may not add up to a value close to the soluble solids. This is because the higher oligosaccharides that these syrups contain will not be quantified in these procedures. If this is an important feature, then Dionex HPLC can be used to quantify these higher oligosaccharides. However, in this case a sodium acetate gradient is used to elute off the higher sugars from the column (Dionex application note 67 (Anon, (n.d.b)). A typical example of a soft drink containing a maltodextrin syrup is given in Figure 10.4. Maltodextrin

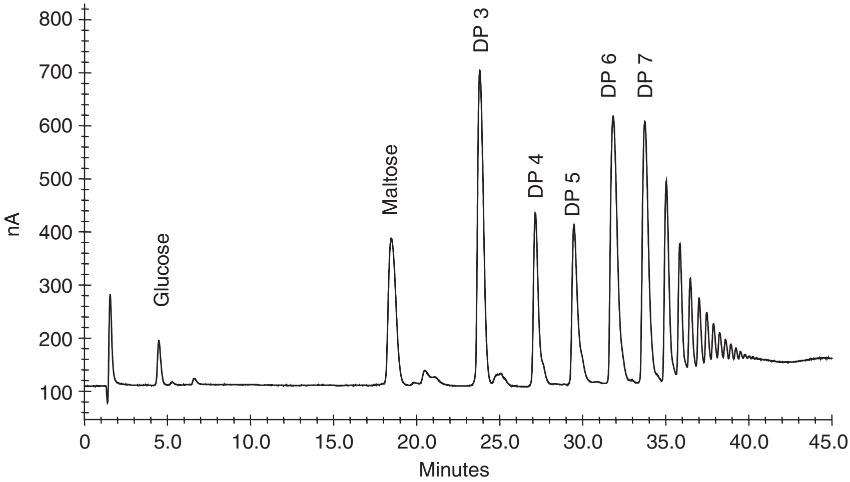


Figure 10.4 Separation of oligosaccharides by Dionex HPLC. Conditions: column: PA-100 5 μm 250 × 4 mm; solvent = 0.002 M sodium acetate in 0.1 M sodium hydroxide; flow rate 1 ml/min; solvent B1M sodium acetate in 0.1 M sodium hydroxide; flow rate 1 ml/min; using pulsed amperometric detection, time 0 = 100% A then a gradient to 46% B over 50 min using curve 9.

syrups are often added to soft drinks to control the osmolality of the product, such as in 'sports' drinks. As the proportion of the different oligosaccharides varies from one manufacturer's product to another's it is impossible to determine the exact level of addition of the maltodextrin syrup unless the actual material used in the formulation is available to the analyst.

10.4.2 Analysis of high-intensity sweeteners

Although sugars are found in juices, the high-intensity sweeteners are not. However, they are often used in juice-based drinks or nectars, and here it is important to ensure that there is resolution of the sweeteners of interest from the compounds naturally present in fruit juices. This can be a particular problem in drinks with a high juice content as naturally occurring polyphenolic materials in the juice can disturb the analysis.

It is common to use one or more high-intensity sweeteners together with sugar in a soft drink's formulation. If some or all of the sugar is replaced with a high-intensity sweetener it becomes important to check whether it has been added at the correct level. This is important not only to produce a consistent product for the consumer but also because in most countries legal maxima are set for these materials. This applies not only to the high-intensity sweeteners but also to the preservatives, bittering agents and other additives that will be discussed below. In the United Kingdom, these limits are given either in the Sweeteners in Foods Regulations 1995 (Anon, 1995b and its various amendments), which were introduced into law in 1996 to enact EC directive 94/35/EC (Anon, 1994), or the Miscellaneous Food Additives Regulations 1995 (Anon, 1995c and its various amendments), which were introduced into law in 1996 to enact EC directive 95/2/EC as corrected (Anon, 1995d).

Saccharin is a high-intensity sweetener that has been commercially available for about 100 years and has probably been one of the most widely used low-calorie sweeteners. The traditional approach to quantifying saccharin involves its extraction into diethyl ether from a strongly acidic solution, removal of the solvent and quantification of the saccharin by titration against sodium hydroxide using bromothymol blue as an indicator (Egan *et al.*, 1990a). Although this is a reliable procedure, it is rather time-consuming and is unlikely to be used extensively today.

With the wider use of HPLC, it is now much more common to assay most sweeteners and some preservatives. A wide range of procedures has been published for the analysis of saccharin in soft drinks using this technique. There is one validated method for the analysis of saccharin in soft drinks in the AOAC manual and also one in the British Standards catalogue. The AOAC procedure separates saccharin, caffeine and benzoic acid using a reverse-phase column and was published as AOAC method number 978.08 (Woodward *et al.*, 1979). A reverse-phase C18 column is used with acetic acid/water/propan-2-ol as the mobile phase and detection at 254 nm. The level of propan-2-ol is set between 0 and 2% (v/v) to ensure that there is an appropriate resolution between saccharin, caffeine, benzoic acid and any other compounds, such as sorbic acid or colours, in the soft drink's formulation. The British Standards method uses a phosphate buffer and acetonitrile mixture as the solvent with a C18 column (Anon, 1999a). This method actually allows the separation of a range of other components in soft drinks and foods, that is, preservatives plus the degradation products of aspartame, although the chromatographic resolution of the method between acesulfame K and phenylalanine, which elute very early in the run, is not baseline.

There are a large number of other published procedures for the separation of a number of sweeteners and preservatives at one time; these are all based on reverse-phase HPLC. Perhaps one of the most startling is the method published by Williams (1986). This uses a small particle size (3 μm) C8 column and allows the separation of a range of colours, sweeteners and preservatives in less than 5 min. The materials separated were amaranth, quinoline yellow, quinine sulphate, sunset yellow, caffeine, aspartame, saccharin, vanillin, sorbic acid, benzoic acid and green S.

HPLC conditions: column: 3 μm Spherisorb RP8 100 × 4.6 mm, solvent = 17.5% acetonitrile, 12.5% methanol, 70% buffer (0.85% sulphuric acid in HPLC-grade water containing 17.5 mM KH_2PO_4 *at pH 1.8); flow rate 1.35 ml/min; UV detection at 220 nm.*

Two methods have been published which were designed to analyse a range of sweeteners and preservatives in one run. The first method, published in German by Hagenauer-Hener *et al*. (1990), describes the analysis of aspartame, acesulfame K, saccharin, caffeine, sorbic acid and benzoic acid in soft drinks and foods. The method relies on a similar system to that given above but with a less complex solvent system (Figure 10.5). The solvent system has been modified to include a gradient portion to elute the preservatives more quickly.

The second method was specifically developed for the analysis of diet soft drinks and food products by Lawrence and Charbonneau (1988) at Health Canada in Ottawa. The method describes a procedure for the separation of a broad range of sweeteners including cyclamate, alitame, sucralose and dulcin. This technique again uses reverse-phase HPLC with an acetonitrile/phosphate buffer system but in this case, as the compounds have a wider range of polarities, a gradient elution is used. Aspartame, saccharin, alitame, acesulfame K and dulcin were detected using ultraviolet absorbance at 210 or 200 nm. Sucralose, which is becoming more popular as a high-intensity sweetener, was measured using RI detection.

HPLC conditions column: C18 250 × 4.6 mm; solvent = 0.02 M phosphate buffer/acetonitrile from 97 : 3 at pH 5.0 to 80 : 20 at pH 3.5 using a linear gradient.

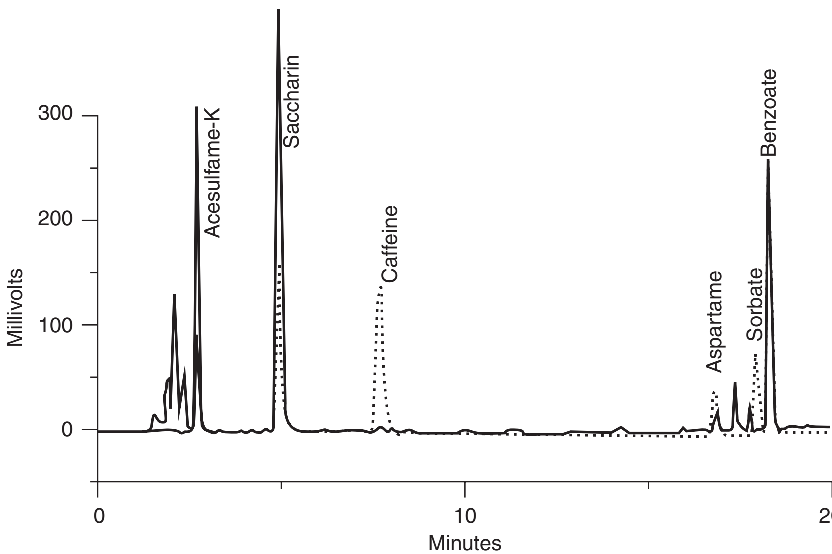


Figure 10.5 Separation of soft drinks ingredients using the method of Hagenauer-Haner *et al.* (1990). Solid line is orange squash; dotted line is mixed standard. Conditions: column PR8 5 μm 150 × 4.6 mm; solvent = 0.02 M phosphate buffer/acetonitrile (90 : 10); flow rate 1 ml/min; using UV detection 220 nm, time 10: 10% to 70% acetonitrile over 15 min.

Aspartame was introduced as a high-intensity sweetener for use in foods in the mid-1980s and has been widely adopted for use in soft drinks. It does have one limitation in that it is not very stable at the pH of normal soft drinks and it is gradually lost by hydrolysis and side-reactions. However, analysis of aspartame and its decomposition products is quite straightforward using HPLC. Tsang *et al*. (1985) used a reverse-phase separation for the analysis of aspartame, whereas Argoudelis (1984) used a strong cation exchanger. The European Norm (EN) method (EN12856:1999) will also separate aspartame and its decomposition products (Anon, 1999a).

Acesulfame K was introduced as a high-intensity sweetener at around the same time as aspartame. It too is much sweeter than sucrose but is also stable under the low pH conditions of soft drinks. Its analysis in a soft drink is relatively straightforward and an HPLC procedure is given by Grosspietsch and Hachenburg (1980).

Although the use of cyclamate was banned in the United Kingdom in the late 1960s, after concerns about its safety, it was re-approved for use in foods in the European Union in 1996 after re-evaluation of toxicological data. A number of the traditional methods for its analysis are given in *Pearson's Analysis of Foods* (Egan *et al*., 1990b) and a number of these involve the oxidation of the molecule to give sulphate ions, which are then measured gravimetrically or colorimetrically.

As cyclamate was banned in a number of countries before HPLC techniques were fully developed, there have not been many methods published for its analysis using modern procedures. The substance offers a challenge to the analyst as it does not have a useful chromophore in the ultraviolet region and its detection by a change in refractive index would be difficult at the levels used in soft drinks (a maximum of 400 ppm).

One paper published in the mid-1980s described the analysis of cyclamate using indirect ultraviolet detection (Herrmann *et al*., 1983). Here the solvent is chosen so that it has a strong ultraviolet absorbance and as the substance elutes from the column it reduces this absorbance and so becomes detectable as a negative peak. A further paper described the analysis of cyclamate using capillary electrophoresis (Zache & Gruending, 1987). There is also a European standard method for its analysis using HPLC (Anon, 1999b). In this case the cyclamate is reacted to form a derivative, *N*,*N*-dichlorocyclohexylamine, prior to its analysis by HPLC using UV detection at 314 nm. The separation is conducted on a C_{18} column and the analysis run takes about 12 min. The European Parliament has recently reviewed the status of cyclamate and reduced the level at which it can be used in soft drinks, which may reduce its usage in beverages.

10.5 Preservatives

There are three main preservatives used in soft drinks in the United Kingdom and Europe: benzoic and sorbic acids and sulphur dioxide. In some countries *para*-hydroxybenzoates are also used. The analysis of these ingredients will be covered in two sections: the first will examine the analysis of benzoic and sorbic acids and the *para*-hydroxybenzoates and the second will discuss the analysis of sulphur dioxide.

10.5.1 Benzoic and sorbic acids and para-*hydroxybenzoates*

One of the older methods used to detect the presence of preservatives in soft drinks and juices is thin-layer chromatography (Woidich *et al.*, 1967). This provides a useful method to detect benzoic and sorbic acids as well as the substituted benzoic acids. The first stage involves the extraction of the preservatives with diethyl ether prior to their chromatographic separation on polyamide plates. Although it is difficult to use this procedure to quantify the level of these preservatives in a sample, it is not impossible. This approach can still be used today by a laboratory that does not have access to HPLC.

Extraction using diethyl ether has also been used in another traditional method to determine the level of preservatives in a sample. In this case, benzoic acid can be extracted from a product at low pH using diethyl ether. By adjusting the pH of the product, and hence the ionisation of the acids themselves, it is possible to quantify benzoic acid in a soft drink in the presence of saccharin. After extraction the benzoic acid can be assayed spectrophotometrically or by titration (Egan *et al.*, 1990c).

Benzoic and sorbic acids are now normally assayed using HPLC. As discussed in the section on the analysis of sweeteners, some of the HPLC methods developed for soft drinks actually allow the separation of both sweeteners and preservatives in one run, for example, Williams (1986), Hagenauer-Hener *et al.*, (1990) and the EU method for sweeteners (Anon, 1999a), although the preservatives were not included in the collaborative trial of the method. The separation of benzoic and sorbic acids can sometimes be difficult and care should be taken that the system will actually resolve these two preservatives if they are present; otherwise spurious results can be obtained. The pH of the solvent is a critical feature that allows the separation of these two preservatives.

A validated method (No. 63) for the analysis of benzoic acid, sorbic and *para*-hydroxybenzoic acids in fruit juices has been published in the IFU handbook (Anon, 1995e). In this method the resolution takes place on a C8 reverse-phase column. With this solvent system (methanolic/ammonium acetate, pH 4.55) sorbic and benzoic acids elute quite quickly and the less polar *para*-hydroxybenzoate esters elute much later. The method does stipulate that caution has to be taken by orange juice due to possible interferences by natural materials in the juice that elute close to benzoic acid. As sorbic and benzoic acids elute quite early in the chromatogram using this method, it would probably not be very useful for soft drinks containing some of the high-intensity sweeteners as they may present problems with interfering peaks.

HPLC conditions: column: 5 μm 250 × 4.6 mm RP8; UV detection at 235 nm; solvent = 0.01 M ammonium acetate and methanol (50 : 40) adjusted to pH 4.55 with acetic acid; flow rate 1.2 ml/min.

There is also a method in the AOAC manual for the analysis of benzoic acid in orange juice (994.11). However, this method was not designed to detect benzoic acid as a preservative. In Florida, when pulpwash was prepared benzoic acid used to be added as a marker, so that if the pulpwash was added to a juice it would be detected. However, it is believed that this practice has now been stopped.

10.5.2 Sulphur dioxide

Sulphur dioxide is a widely used preservative for foods and soft drinks, particularly dilutables, and in principle is quite easy to detect and quantify. Owing to the importance of sulphur dioxide as a preservative an extensive review of its chemistry in foods was undertaken in the mid-1980s (Wedzicha, 1984).

There is a very simple and quick method that can be used to detect the reducing power of sulphur dioxide, developed in the last century and often called the Ripper titration (Ough, 1988). In this method, sulphur dioxide is titrated against iodine or potassium iodate/potassium iodide solution in the presence of starch. When all the sulphur dioxide has been oxidised, a blue colour is produced by the reaction of free iodine with the starch. This is a very quick method but will give only an estimate of the level of sulphur dioxide as other reducing substances, such as ascorbic acid, will interfere; consequently, this method is not particularly appropriate for juices with high ascorbate levels.

To assess the level of sulphur dioxide in a fruit juice, or a soft drink containing juice, a steam distillation procedure is generally used. Here sulphur dioxide is driven out of acidified solution by heating. Acidification displaces any sulphur dioxide that was bound to other food materials. As sulphur dioxide is carried over by the steam it passes through a trap containing a hydrogen peroxide solution, where it is oxidised to sulphuric acid. The acidity contained in the trap is then measured by titration against sodium hydroxide.

There have been many modifications for different applications to the original method published by Monier-Williams (1927). However, for fruit juices it is best to use the Tanner modification (Tanner, 1963), which uses phosphoric acid rather than hydrochloric acid for the acidification as this method is less liable to interferences. The method has also been published in the IFU handbook method no. 7a (Anon, 2000) with collaborative ring test data.

Although some chromatographic methods have been published for the analysis of sulphur dioxide in solution (the sulphite anion), these have often involved a steam distillation to ensure that all the sulphur dioxide is liberated. However, as the distillation is a time-consuming stage rather than a simple acid/base titration, there is little point in using these procedures in systems that do not liberate large amounts of other acidic or sulphurous materials, such as garlic and cabbage, which interfere with the titration method.

Some newer methods have also been developed which will assay both free and total sulphur dioxide. These procedures rely on the separation of the sulphite anion from the other materials in the food using ion-exchange chromatography and measurement with an electrochemical detector. There is an AOAC approved method using direct current (DC) amperometric detection (Anon, 1995f). This employs an alkaline extraction medium (20 mM phosphate buffer containing 10 mM mannitol at pH 9) so that free and total sulphur dioxide can be detected. After extraction the sample is injected directly on to an HPLC system with an ICE-AS 1 column and DC electrochemical detection. The AOAC method suggests cleaning the electrode at the end of each run to maintain optimal detector sensitivity. However, Dionex has improved the original AOAC method by using pulsed amperometric detection (Anon, n.d.c). Using this approach, the detector response is much more stable, as the electrode is constantly cleaned during the analysis rather than at the end of each run as suggested in the AOAC method. Although at the time of publication of the first edition of this book it was thought that this modification might be collaboratively tested, formal verification is still awaited.

HPLC conditions: column: 250 × 4 mm Ion Pac ICE-AS1; solvent = 20 mM sulphuric acid; PAD detection using a platinum electrode.

10.6 Acidulants

After sweetness, the second most important feature of a soft drink is acidity and the balance of the sweetness to acidity (sourness), commonly called the Brix to acid ratio. An acceptable range for this ratio is often laid down in juice specifications. For European tastes in orange juice, a Brix to acid ratio might be set around 15 and 16.

The normal method used to assess the acidity of a soft drink or fruit juice is titration using sodium hydroxide. Traditionally, phenolphthalein was used as an indicator to detect the end-point of the titration. However, with the ready availability of pH meters, testing is now carried out to a final pH of 8.1. A major advantage of this technique is that it allows the titration to be carried out automatically and avoids problems with coloured products, such as blackcurrant juices, where the phenolphthalein indicator end-point, colourless to pink, can be difficult or impossible to detect. The ability to carry out the titrations automatically is also more time- and cost-effective.

In a carbonated product, carbon dioxide has first to be removed by boiling, as sonication alone is not enough to ensure that all the gas has been liberated from a soft drink. It should be noted that if dealing with cola products, which contain phosphoric acid, a titration taken to an end-point of pH 8.1 will not affect the final ionisation of the hydrogen phosphate ion (HPO_4^{2-}) to the phosphate ion (PO_4^{3-}). As the pKa of HPO_4^{2-} is 12.67, this final dissociation will not take place under normal titration conditions and should be allowed for when the acid content of cola products is being determined; for example, the acid only 'appears' to be divalent.

In the analysis of fruit juices, it is important to determine the levels of the individual acids to assess authenticity and quality. A range of these acids can be determined using an enzyme-linked assays and these procedures have been collaboratively tested and published in the IFU compendium of methods (citric no. 22, isocitric no. 54, D-malic no. 64, L-malic no. 21 and D-and L-lactic acids no. 53). r-Biopharm now distributes the Boehringer Mannheim kits to assess the levels of these acids. Similar kits are available from other suppliers.

Most acids can also be assessed with HPLC using either ultraviolet or conductivity detection. The following procedure was developed to assess the organic acid profile of cranberry and apple juices but has been found to work well for other organic acids (Coppola & Starr, 1986). It has been published as AOAC method no 986.13. A typical trace for cranberry juice run using this method is given in figure 10.6. The method recommends separation of the acidic materials from the sugars by prior treatment with an ion-exchange resin.

Separation of the organic acids is also possible using an ion-exclusion column, such as a Bio-Rad HPX-87H. However, in this case it is essential that the acids are separated from the sugars; otherwise a number of peaks co-elute, thus distorting the quantification. This method is particularly good for the analysis of fumaric acid in apple juice because no pre-treatment is required and the acid elutes late in the chromatogram, well separated from other components (Figure 10.7).

Fumaric acid has been found to be a good marker to detect the addition of D,L-malic acid to apple juice (Junge & Spandinger, 1982). With the advent of the enzymic assay procedure for D-malic acid the method fell out of use. However, in 1995, a number of samples of apple juice in Germany were found to contain elevated levels of fumaric acid, which was attributed to the addition of L-malic acid.

Organic acids can also be quantified using HPLC linked to a conductivity detector. This has one advantage over UV detection in that only charged species are measured, which means that the method is liable to fewer interferences. Depending on the actual approach chosen, it is sometimes possible to detect other anions, such as Cl^-, SO_4^{2-} and PO_4^{3-}, in the same run. If the conditions given in Dionex application no. 21 are used, this allows the organic acids to be separated without interferences from the fully ionised anions such as Cl^-, SO_4^{2-} (Anon, n.d.d).

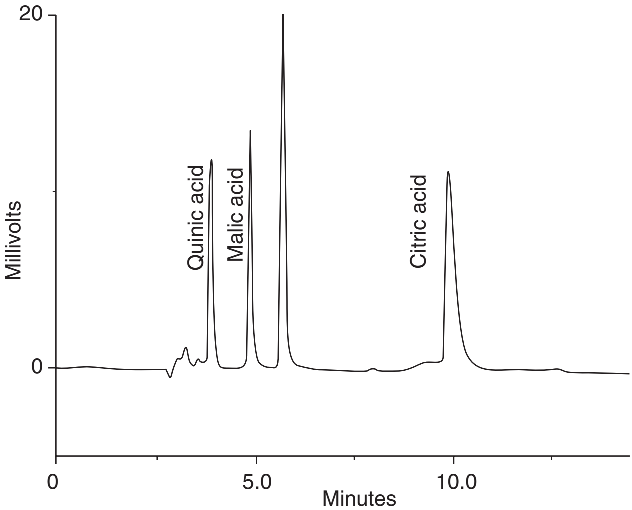


Figure 10.6 HPLC separation of the organic acids in cranberry juice using Coppola's method. Conditions: column: 5 μm ODS2 250 × 4.6 mm; UV detection at 210 nm; solvent = 0.05 M phosphate buffer at pH 2.5; flow rate 1 ml/min.

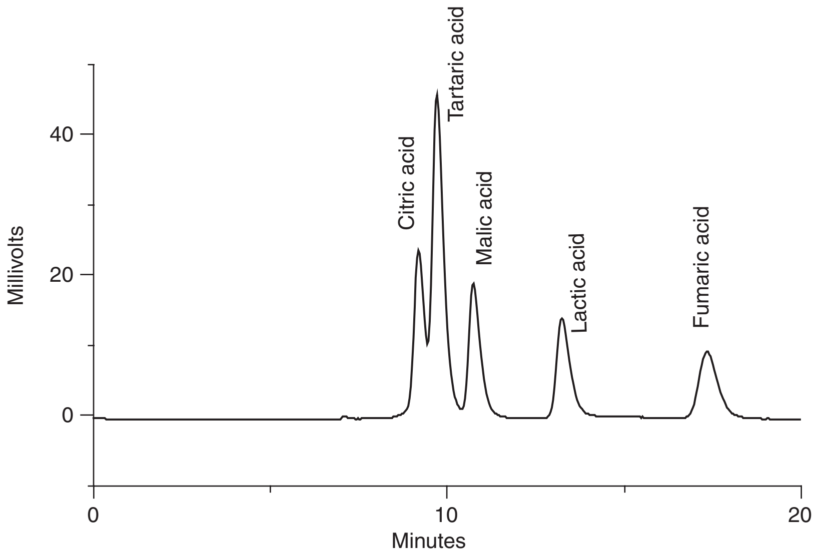


Figure 10.7 HPLC separation of organic acid standard by ion-exclusion chromatography with UV detection. Conditions: column Phenomonex Rezex monos 300 × 8 mm; UV detection at 210 nm; solvents 4 mM H_2SO_4 in Milli-Q water; flow rate 0.6 ml/min.

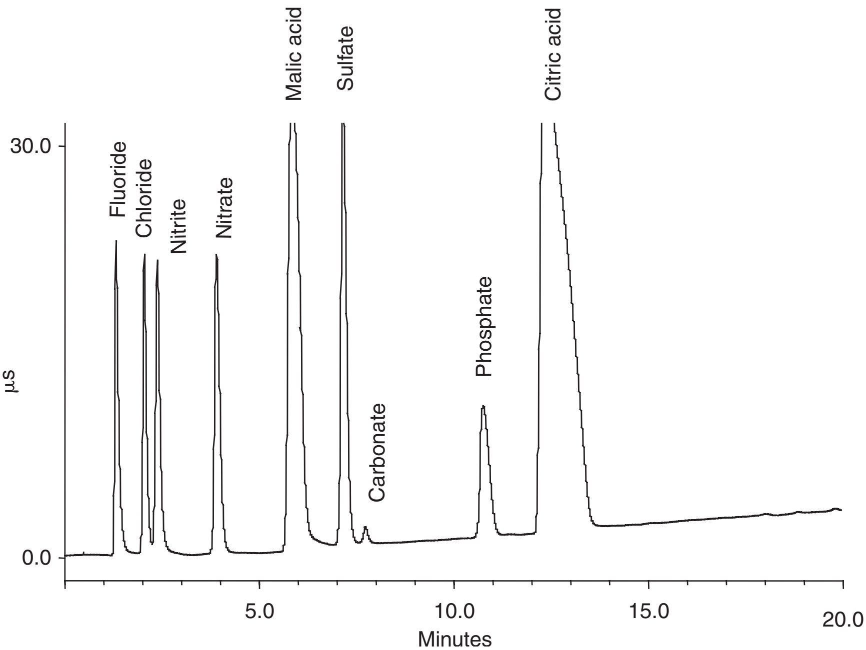


Figure 10.8 HPLC separation of organic acids and anions by ion-exchange chromatography and conductivity detection. Conditions: column Dionex AS-11 250 × 4 mm: suppressed conductivity detection; solvents 18 MΩ water and 0.3 M NaOH; flow rate 1.5 ml/min 2 mM NaOH to 33 mM NaOH over 15 min.

HPLC conditions: column: 2xICE-AS1 300 × 7.9 mm; conductivity detection; solvent = 2 mM octanesulphonic acid in 2% propan-2-ol; flow rate 0.5 ml/min.

If a method similar to Dionex application no. 123 is used, both organic acids and anions can be separated on the AS-11 column (Figure 10.8) (Anon, n.d.e). In fact, care has to be taken with this method as nitrate and malate ions elute very close to each other under these conditions. This can be a particular issue if the column is not operating well and there is a need to determine the level of nitrate in juices which contain high levels of malic acid.

10.7 Carbonation

For carbonated soft drinks the level of carbonation is a key parameter. If the level of carbonation is too low or too high the overall flavour balance of the product will be disrupted. The level of carbonation can be measured in a number of ways and four are discussed here. The first is a manometric method published by AOAC for beer (940.17); the second is also a manometric procedure, but is somewhat less complicated than the AOAC procedure. The final two methods use specialist equipment: one uses the electric conductance of carbon dioxide gas for the determination and the other method uses ion chromatography.

In the AOAC procedure, a container's cap is punctured with a strong needle. The gas is then shaken out of the product and collected in a gas burette. The pressure is finally adjusted to atmospheric before the volume is read off the burette. Carbon dioxide can also be removed by dissolving it in sodium hydroxide solution; any other gases in the container can also be measured in this way.

The second method is a similar procedure; the cap of the product's container (plastic, glass or can) is punctured with a needle. Carbon dioxide is expelled from solution by vigorous shaking, and the headspace pressure is recorded. From the pressure rise, the volume of carbon dioxide dissolved in the product can be determined. This method is probably one of the most commonly used procedures for measuring carbonation; however, the exact method used varies from one manufacturer to another. One supplier of this type of carbonation tester is Stevenson & Reeves of Edinburgh, which also sells a slide rule to convert pressure rise measurements into carbonation levels. Information about their products can be found on their website (http://www.stevenson-reeves.co.uk).

Caution. *Care has to be taken when measuring the carbonation level of products using this method in glass containers as there is a risk that the container might fracture. To remove this risk, special carbonation testers can be purchased in which the glass bottle is contained inside a shield so that if it does fracture no glass fragments can escape.*

The first of the more sophisticated approaches requires a specialised instrument, the Corning 965 carbon dioxide analyser.

The soft drink is initially cooled to 4°C in a fridge. The container is then opened and an aliquot of concentrated sodium hydroxide (40%) is added to 'quench' the carbon dioxide in the product (typically 10 ml of NaOH is added to 284 ml of product). The carbon dioxide is quenched by reaction with the sodium hydroxide to form bicarbonate and carbonate ions. An aliquot (50 μl) of the quenched product is removed and pipetted into the corning instrument's cell. The cell is closed and the solution acidified to release the carbon dioxide, which is then detected by the change in the thermal conductance of the vapour phase.

This is a rapid procedure and gives reproducible results similar to the pressure rise method and is extensively used in the brewing and soft drinks industries.

The final method uses ion chromatography and shows comparable results to the Corning method (Harms *et al*., 2000). Here the carbon dioxide is again converted to carbonate by the addition of sodium hydroxide. The treated sample is then analysed using HPLC linked with conductivity detection. Harms *et al*., found that the method worked well for both beers and soft drinks and that there were no significant differences between the results produced by this procedure and the data obtained using the normal Corning method discussed above.

HPLC conditions: column: ICE-AS1 150 $\times$ 4 mm; conductivity detection; solvent = 18 MΩ HPLC-grade water; flow rate 0.4 ml/min.

During the carbonation of water, the levels of oxygen and nitrogen need to be reduced to the minimum. This means not only that the overpressure of the carbon dioxide applied to the carbonator can be lower but also that fewer problems are seen with fobbing (rapid out-gassing) during filling. Fobbing causes loss of product when the container leaves the filler head. Oxygen and nitrogen gases are often typically driven out of solution by rapid agitation of the water as it is fed into the carbonator and during carbon dioxide sparging to ensure that the problem is kept to a minimum.

In non-carbonated products, oxygen can also be a problem as it can cause oxidative changes to the flavour and also reduce vitamin retention, particularly of vitamin C. Although headspace oxygen measurements can be made using, gas–liquid chromatography (GLC) this is not a straightforward analysis and requires skilled staff and specialised equipment (Anon, 1997a).

10.8 Miscellaneous additives

Two compounds that are used in some soft drinks formulations for specific purposes are caffeine, used in a range of beverages including colas for its stimulant properties, and quinine, used for its bitter taste. Traditional techniques for the analysis of these two compounds have often involved their extraction from aqueous solution into an organic solvent and then quantification by one of a range of methods.

Over the last 5–10 years, there has been a rapid growth in drinks with certain associated attributes. As highlighted earlier, Red Bull has been a great success based on its energy and stimulant properties. However, there have been a range of other drinks which have focused on different aspects of health, and these often contain green tea extracts, soy extracts and/or extracts of herbs.

10.8.1 Caffeine

Three spectrophotometric procedures are given in the AOAC compendium of methods (960.22, 962.13 and 967.11) for the analysis of caffeine, all of which have an extraction stage followed by a quantification procedure. There is also an HPLC method, discussed earlier, which was designed to measure saccharin, benzoic acid and caffeine at the same time (AOAC, 978.08). Again, the HPLC method, EN 12856:1999 (Anon, 1999a), can be used for the analysis of caffeine, but this analyte was not included in the collaborative study.

In *Pearson's Analysis of Foods* two methods are quoted for the analysis of caffeine. The first is a simple solvent extraction followed by quantification by ultraviolet absorbance at 273 nm (Egan *et al.*, 1990d) and the other is a GLC method.

10.8.2 Quinine

The level of quinine in a soft drink can be measured either by determination of the absorbance of an extract of the soft drink after making the product alkaline with ammonia or by detecting it directly spectrophotometrically or fluorimetrically (Egan *et al.*, 1990e).

As with the other additives used in soft drinks, caffeine and quinine can be, and often have been, detected using the same HPLC method used for other materials, such as in the method published by Williams (1986). This method separates most of the major additives used in soft drinks in a short time (4–5 min). Although some of the resolutions are not quite baseline, as would be expected in such a short analysis time, and not all of the synthetic colours are separated from each other, this is still a very impressive method.

10.8.3 Taurine

Taurine, or 2-aminoethanesulphonic acid as is it is correctly called using the IUPAC method, is one of the specialised ingredients often included in energy drinks or products that claim to stimulate the mind. It is similar to an amino acid except that the carboxylic acid group is replaced by a sulphonic acid residue. It is involved in a number of functions in the body such as digestion and is thought to be involved in brain development. It can be analysed using the liquid chromatographic method used for amino acids. This involves separation using an ion-exchange column and detection in the visible region after post-column addition of ninhydrin. There is a standardised method for the analysis of amino acids in fruit and vegetable juices (Anon, 1999c) which would be applicable for this type of analysis. However, this approach needs a dedicated machine and is costly to set up if not run on a regular basis.

There are also other pre-column derivatisation methods, such the Waters Pico-Tag (Cohen, 1984) or AccQ-Tag approaches (Anon, 1996a,b). Here the amino acids and taurine are treated with a reagent which gives the derivatised molecules either a UV (Pico-Tag) or fluorescent (AccQ-Tag) chromophore, and these derivatised molecules are separated on special HPLC column designed for the purpose. There is also a new method, recently published by Dionex, which requires no pre- or post-column derivatisation. Here amino acids are separated on an ion-exchange column (PA-10) which happens also to be used for sugars analysis, using a hydroxide ion or hydroxide/acetate gradient (Anon, 2003a).

10.8.4 Fibre analysis

There is a growing interest in the inclusion of soluble fibre into the diet to help improve health. This is extending to drinks as well, with such fibre being added to milk- and fruit-based products such as 'smoothies'. One source of soluble fibre which has attracted attention over the last few years is inulin or oligofructans. Inulin consists of oligosaccharides that are extracted from chicory or Jerusalem artichokes and that are claimed to improve colon function and to have prebiotic properties, enhancing the working of the gut. Inulin is a complex carbohydrate which can be assayed in a number of different ways. However, there are two published methods in the AOAC manual for its analysis (997.08 and 999.03).

In method 997.08, high-pH anion exchange chromatography linked with pulsed amperometric detection (HPAEC-PAD) is used to analyse the product for sugars at various stages of the analysis process. Initially the concentrations of the free sugars in the product are measured. The sugars are then determined after treatment of an extract with an amylase and finally an inulinase. From the levels of the sugars (sucrose, glucose, fructose and galactose if milk is present) found at each stage, it is possible to determine the level of inulin in the product.

Method 999.03 is slightly different and uses two enzymes to hydrolyse any sucrose or starch that the product contains. The glucose and fructose liberated in this step are then reduced with sodium borohydride to their corresponding alcohols. Finally, the borohydride-treated extract is mixed with a fructanase to degrade the inulin, and the free sugars that are liberated in this step are quantified using a colorimetric assay. Once again, from the level of free sugars liberated by the fructanase, the inulin content of the product can be determined.

There are a number of well-documented, validated methods for the analysis of total fibre in food products in the AOAC manual (993.19, 993.23, 991.42, 991.43 and 958.29). Most of these are gravimetric methods where the fibre is precipitated with ethanol and washed and the fibre content calculated after allowances are made for any protein and/or ash that the precipitate contains. These methods rely on precipitation of the fibre with alcohol for their quantification, but as inulin is soluble in mixtures of ethanol and water it cannot be detected in these tests. The most recent method (2001.03) includes an HPLC stage to allow for detection of the presence of maltodextrins, which are poorly digested in the body, but this will not detect inulin.

10.8.5 Herbal drinks

There is a growing interest in the health benefits that can be derived from taking herbal extracts, and this has led to the inclusion of such extracts in soft drinks. Although this is a relatively small and specialist market at present, it is growing in importance. The analysis of herbal extracts is a very complex issue and outside the scope of this chapter. However, it is incumbent on manufacturers to check the authenticity of the materials they are buying. This can be done in a number of ways. However, as with the authenticity of fruit juices, the analysis is often best left up to specialist laboratories that have a broad range of experience in this field.

There are some useful references in this area. The German Pharmacopoeia (DAB 10) has a large section on the analysis of herbal extracts; many of these analyses use high-performance thin-layer chromatography (HPTLC). Another useful reference is the book edited by Grainger-Bisset (1994) which gives a long list of herbs, the risk of their adulteration, pictures to assist in their authentication and some analytical procedures. There is also a small monograph which details a number of herbal extracts in the British Herbal Pharmacopoeia (Anon, 1996c). There is a growing level of interest in this topic in the United States and an increasing number of HPLC methods are being published in the US Pharmacopoeia.

If it is a challenge to confirm the authenticity of a herbal extract, it is even more of an analytical challenge in a complex finished product that might contain juice and other ingredients. This means that a manufacturer should check incoming raw materials and that they should be delivered with a certificate of analysis from a reputable laboratory that specialises in this area.

10.8.6 Osmolality

Although osmolality is not an additive, it is an important parameter to measure in sports drinks and isotonic products that are being sold for rehydration purposes. Such products require an osmotic pressure similar to serum, which is typically 285–295 mOsm/kg. Osmolality is measured using the colligative properties of solutions, which chemists cover in their first year of physical chemistry at university. These properties are dependent on the numbers of molecules in solution rather than their nature. Osmometers typically use either the depression of freezing point or the elevation of vapour pressure to measure the osmolality of a product. Two manufacturers who supply these types of instruments are Advanced Instruments Inc, Norwood, Massachusetts, USA (http://www.aitests.com) and Wescor Inc, Logan, Utah, USA (http://www.wescor.com). Analysis for osmolality typically requires no sample treatment; simply, a sample is introduced into the instrument and a few minutes later the value is printed out.

10.9 Analysis of colours used in soft drinks

The colours used in soft drinks can be split into two classes. The first class consists of the synthetic food colours that have been used to enhance the appearance of foods and beverages for many years. They are generally water soluble and include tartrazine, quinoline yellow, sunset yellow and ponceau 4R. However, over the last few years these have become less popular with the consumer because of the bad publicity concerning their possible links with hyperactivity in children. This strong consumer trend led to the reformulation of a wide range of products, particularly orange dilutables, in the United Kingdom in the mid-1980s to remove the azo-dyes such as tartrazine and sunset yellow, and replace them with natural colours or synthetic colours based around the carotenoid molecule, such as annatto, β-carotene and β-apo-8$'$-carotenal. This switch from the water-soluble colours to β-carotene or β-apo-8$'$-carotenal caused its own problems as these new formulations showed a tendency to neck ringing, forming unsightly red/orange rings at the top of the container.

Although the anthocyanin and carotenoid pigments are stable at low pH and provide a strong colour, they can in some cases fade due to oxidation. This is a particular limitation with the carotenoid pigments, but is generally overcome by the addition of an antioxidant such as ascorbic acid, which significantly improves their stability.

10.9.1 Assessment of colour

The colour of a soft drink or fruit juice may be assessed in a number of ways. If the product is clear it can simply be carried out by measuring the absorbance of the product at one or more wavelengths. The actual values chosen will depend on the particular colour of the product. For a yellow product, such as apple juice, wavelengths of 465, 430 or 420 nm are often chosen to assess the colour. These values can then be expressed in European brewing convention (EBC) units by multiplication by a factor of 25. The actual Brix value chosen to assess the colour depends on the country; however, levels between 11 and 12 are often taken as the norm. If dealing with a red-coloured product, then the assessment is generally carried out at 520 nm. Absorbance values are sometimes also taken at 420 nm in red or black juices to assess the brownness of the product. The two absorbance values are often used to express a colour ratio, which gives an indication of colour versus brownness:

$$\text{colour ratio} = \text{absorbance at 520 nm/absorbance at 420 nm}$$

Sometimes the absorbance is also assessed at 580 nm and a ratio calculated for the values at 520 and 580 nm, which is called the blue index. With the red/black juices, where the product may have to be diluted, the intensity of the colour is assessed by dilution in an acidic buffer to ensure that the true colour intensity is recorded. If this is not done the colour can be reduced by the shift in pH affecting the apparent colour of the product. The analysis of the colour in juices is detailed in IFU Handbook method No. 78.

In cloudy products, colour assessment is more difficult. The particulate material can be removed by filtration in some cases. However, this does not solve the problem in all cases as some colours, particularly the carotenoids and some anthocyanin pigments, are absorbed on to the particulate material/filters and their removal gives a low colour reading. In addition, the particulate material affects the opacity of the product, which affects the visual perception of the colour. It has been known for a number of years that the simple approach of measurement of a product's absorbance values at 420 or 520 nm does not fully address this issue because some products can have similar absorbance values at these wavelengths but will be perceived visually as very different.

One way in which this has been addressed is by the use of tristimulus colour measurement. In this method the colour is split into three separate primary components, which more closely match the way the eye perceives colour. In this method the absorbance or reflectance of a product is measured at a range of wavelengths in the red, green and blue areas of the spectrum. The values are taken and the components X, Y and Z are calculated. These X, Y and Z values are then transformed to split the colour up into three variables that address the colour or hue (red/green, yellow/blue) of a product, the opacity of the colour (how much light it reflects or absorbs) and finally the depth or intensity of the colour (deep purple/pale purple). This three-dimensional space can be represented by either L, a, b or H, C, L values, which are different conventions used to describe a colour in a conceptually similar manner. In the L, a, b system, used by Hunter Lab, the colour is defined in terms of its lightness (L), which is the extent of black or white it contains (from 0 to 100), and in terms of two variables a and b which define the colour hue and intensity. Red colours are situated in the $+a$ direction; green has $-a$ values. Yellow is in the $+b$ direction and blue has $-b$ values. The value is indicative of the intensity of the colour (i.e. the larger the absolute value, the more intense is the colour). In the CIELAB 76 convention, L again defines the lightness as above, whereas H (hue angle) defines what the colour is (red is situated at 0°, yellow at 90°, green at 180° and blue at 270°) and C (chroma) describes the intensity or depth of colour. This area was reviewed by McKaren (1980).

The different instruments (Hunter, Gardner, Instrumental Colour Systems, etc.) process absorbance and reflectance data in slightly different ways, which means that the values obtained can differ slightly from one instrument manufacturer to another. Therefore, a product's defined colour has to be qualified with a statement indicating the instrument used. Notwithstanding this limitation, it is not uncommon to find a tristimulus 'colour meter' in a manufacturer's quality assurance laboratory so that routine quantitative assessment of a product's colour can be made. This is particularly true for tomato-based products, whose nature makes conventional spectrophotometric assessment meaningless.

The discussion above has addressed the assessment of a product's colour or perceived colour in basic terms. In the next two sections, methods to determine which coloured compounds are present in a product will be addressed. For the purpose of this chapter, the section on synthetic dyes will cover the analysis of the water-soluble dyes, or so-called coal tar dyes, and the section on natural pigments will cover the anthocyanin pigments, such as grape skin extracts, and the carotenoid-based materials, even if they are of synthetic origin.

10.9.2 Synthetic colours

Synthetic colours are materials based around azo-sulphonic acids. They are water soluble and provide strong stable colours. There is an interesting chapter by Wadds (1984) that discusses some of the basic methods, including thin-layer, paper and high-performance liquid chromatography, which have been used in the past and are still used today to analyse for these colours.

When colours are added to a food system, their characterisation is often more difficult due to interferences from other materials in the food or difficulty with their extraction from the food. This is particularly the case for high-protein foods, which bind colours very tightly and can make their quantitative analysis very difficult. However, analysis for azo-dyes in soft drinks is generally straightforward using modern methods. There is less interference than in other food systems and as the colours are already in solution, and not bound to other materials, this makes the analysis easier. In some cases, the colours can be analysed without prior concentration and in others they have to be concentrated by solid-phase extraction methods, for example, C_{18} cartridges followed by elution with a small volume of methanolic ammonia.

Owing to the varied structures of various food dyes, they can often be differentiated from one another by their characteristic ultraviolet/visible absorbance spectra. Using HPLC coupled with a diode array detector (HPLC-DAD) it is possible to collect a compound's absorbance spectrum as it elutes from the HPLC column, which greatly assists in identification. At Reading Scientific Services Ltd (RSSL) this type of detector is routinely used in a range of analyses of such substances as patulin, a mycotoxin found in apple juice, and in the analysis of colours and vitamins, which allows a more certain assignment of a particular peak to a specific compound to be made.

A number of different approaches, all of which have both advantages and shortcomings, have been proposed for the analysis of water-soluble dyes. The separation of colours has been carried out using ion-exchange resins, reverse-phase HPLC coupled with ion-pair reagents and reverse-phase HPLC at low pH where the ionisation of the dyes is suppressed. The last of these is the technique used at RSSL and is also the method recommended by Wadds (1984). It offers the simplest approach to this type of analysis and a typical HPLC profile of

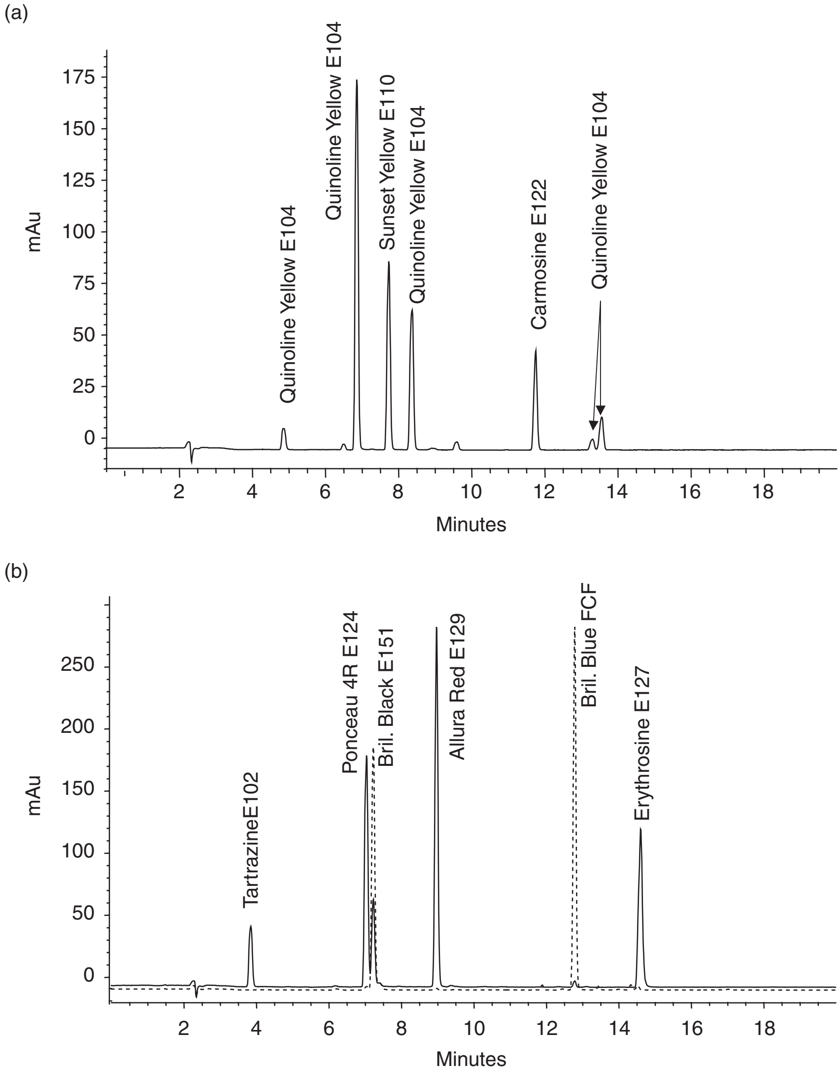


Figure 10.9 HPLC trace of standard pigments using gradient elution system. (a) standard mixture (1) monitored at 415 nm; (b) standard mixture (2) monitored at 490 nm and 590 nm. Conditions: column: 5 μm C18 150 × 4.6 mm, using diode array detection at 450 nm and gradient elution; solvent A = 0.02 M ammonium acetate, Solvent B = acetonitrile; gradient profile: 0 min 95% A, 20 min 50% A, 25 min 95% A; flow rate 1.0 ml/min.

some water-soluble pigments is given in Figure 10.9. This approach allows the identification of pigments and undeclared materials present in a product. However, the quantitative analysis of colours is often more difficult because of the lack of pure standards or pigments of ‘known’ purity. This area is further complicated by the fact that some pigments are made up from a mixture of a number of components rather than a single entity: for example, in Figure 10.9(a) several peaks are attributed to a single colour, quinoline yellow, which is made up of five different compounds.

10.9.3 Natural pigments

This section examines the analysis of two major classes of compounds:

(1) anthocyanin-based materials (e.g. grapeskin extracts and highly coloured juices);
(2) carotenoid-based materials (e.g. annatto, β-carotene and β-apo-8′-carotenal).

Whereas the quantitative analysis of the synthetic water-soluble pigments is sometimes difficult, it is often impossible with the natural pigments because of the lack of pure standards or their prohibitive cost. If they are available they can cost several hundred dollars per 100 mg. There is also a large natural variation between one plant extract and another. Quantitative analysis of these natural extracts is further complicated because they generally contain a range of different compounds that make up the ‘colour’. A concord grapeskin extract can contain over 30 different monomeric anthocyanin pigments plus some polymeric materials; all of these contribute to the colour but will not be readily quantifiable by HPLC. Quantitative analysis of anthocyanins, however, is often less critical than analysis of the synthetic pigments. In fact, the analysis of anthocyanin pigments is usually carried out to assess the authenticity of red and black fruit juices rather than to quantify the level of added colour. This is changing with the increased interest in the health aspects of polyphenolic compounds such as those seen in grapeskin extracts.

The analysis of the natural carotenoid pigments found in orange juice, for example, is extremely complex and should be left to an expert laboratory. However, the quantitative analysis of added β-carotene and/or β-apo-8′-carotenal to an orange drink or dilutable is much easier as pure standards are commercially available and these compounds can be readily separated using HPLC.

10.9.3.1 Anthocyanin pigments

As extensive research into anthocyanin has been conducted over the years, a large amount of information is available on which pigments are found in a particular fruit. This allows an analyst to assess whether a sample contains the expected pigments and to determine whether there are any added from another source or fruit. A very good reference book which details the anthocyanins found in various plants and also gives details of other phenolic materials found in fruits is Macheix’s book on plant phenolics (Macheix *et al.,* 1990).

Most anthocyanin analysis today is carried out using reverse-phase HPLC, often coupled with diode array detection, as this offers the best approach to assess these compounds. The use of reverse-phase HPLC means that predictions can be made about the elution order of compounds from the column, which is based on their polarity and makes the interpretation of the anthocyanin profiles easier. A couple of useful chapters that discuss the analysis of anthocyanin pigments more fully are those of Lea (1988) and Wrolstad *et al*. (1995). Both of these describe more fully the types of methods used and give examples of profiles found in different fruit types. The method used by Wrolstad *et al*., is similar to the procedure that is routinely used at RSSL for this type of analysis.

Another method of analysis that is used around Europe, and given below, was used by Hofsommer in the analysis of red and black juices as part of an EU-sponsored project in the early 1990s on methods to determine the authenticity of fruit juices (Hofsommer, 1994a). This method has been collaboratively tested by the IFU analytical commission and was found to give acceptable results (Anon, 1998b). The procedure does not involve any sample cleanup for juices; the product

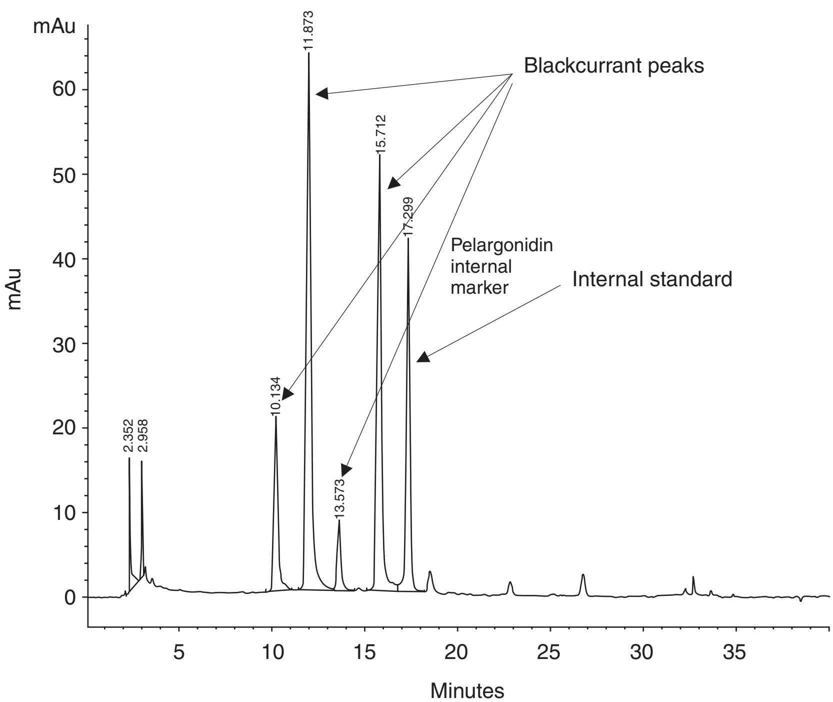


Figure 10.10 HPLC analysis of a blackcurrant juice using the IFU method. Conditions: column: 5 μm ODS2 250 × 4.6 mm; flow rate 1 ml/min; solvent A = 10% formic acid, solvent B = 10% formic acid in 50% acetonitrile and 40% water; gradient 0–1 min 12% B, 1–26 min from 12% B to 30% B, 26–35 min 30% to 100% B, 38 min 100% B, 38–43 min 100% to 12% B; detection at 518 nm using DAD.

is merely filtered and an internal standard added prior to analysis. However, for soft drinks it is often useful to employ a reverse-phase cartridge to concentrate the colours prior to analysis due to their low levels. Two examples of this method for juices are given in Figures 10.10 and 10.11. Figure 10.10 is an HPLC trace for an authentic blackcurrant juice, which shows four peaks for the juice plus the late-running pelargonidin internal marker. Figure 10.11 shows a chromatogram for an adulterated blackcurrant juice that contains elderberry. In this trace there are the same five peaks seen in Figure 10.10, but two others are also present (indicated by an arrow) and the intensity of the third peak of the blackcurrant juice (retention time 13.5 min) is enhanced by the co-elution of a pigment from the elderberry (also indicated by an arrow). This demonstrates the importance of this type of procedure, since adulteration was instantly detectable using this method. However, adulteration is not always as easy as this to detect. The judicious choice of materials can enable adulteration to be hidden. Assessment of the anthocyanin profiles of red and black juices should be part of any routine screening procedure designed to assess the authenticity of these products.

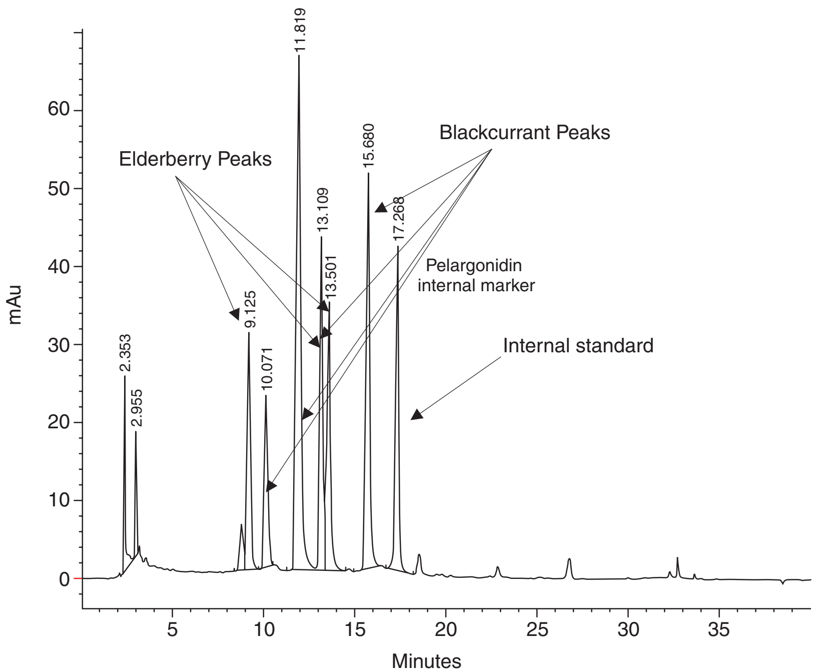
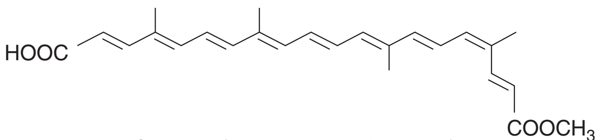


Figure 10.11 HPLC analysis of adulterated blackcurrant juice containing elderberry using the IFU method. Conditions: column: 5 μm ODS2 250 × 4.6 mm; flow rate 1ml/min; solvent A = 10% formic acid, solvent B = 10% formic acid in 50% acetonitrile and 40% water; gradient 0–1 min 12% B, 1–26 min from 12% B to 30% B, 26–35 min 30% to 100% B, 38 min 100% B, 38–43 min 100% to 12% B; detection at 518 nm using DAD.

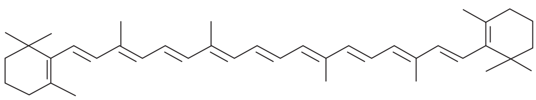
10.9.3.2 Carotenoid-type materials

For orange-based drinks, either flavoured or containing juice, carotenoid-type materials such as annatto, β-carotene and β-apo-8′-carotenal are often used as colours. Although the analysis of the natural carotenoid pigments found in orange juice is extremely complex because of the very large number of compounds present, the analysis of soft drinks for added carotenoid pigments is often much more straightforward. If β-carotene or β-apo-8′-carotenal is being used it can be extracted either by solvent extraction (ethyl acetate) or by precipitation of the pigments, using Carraz reagents followed by extraction of the pellet material with acetone. Analysis and quantification of these pigments in products with a low juice content is relatively straightforward because there are few interferences. Often a simple gradient or isocratic system using acetone/water, a reverse-phase column (RP18) and detection at 430 nm can be used.

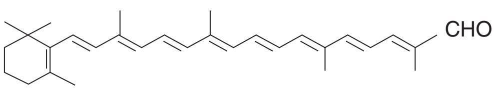
Another pigment that is sometimes used in soft drinks is annatto. This is extracted from *Bixa orellana* and contains a range of apocarotenoid materials based around a C_{24} dicarboxylic acid from the central portion of the carotenoid backbone, as shown in Figure 10.12. This figure also shows the structures of β-carotene and β-apo-8′-carotenal for comparison. After extraction, the extract is subjected to a basic hydrolysis to cleave some of the ester groups, giving the water-soluble 'annatto' colour. This is a mixture of free dicarboxylic acids and esterified materials and its analysis by HPLC gives a number of peaks because the product is a mixture of a range of closely related compounds.



Cis-bixin (a constituent of annatto)



β-carotene



β-apo-8′-carotenal

Figure 10.12 Chemical structures for annatto, β-carotene and β-apo-8′-carotenal.

A typical example of an HPLC method used to analyse carotenoids is given below and again comes from work sponsored by the European Union in the early 1990s (Hofsommer, 1994b):

Conditions: column: 5 μm ODS2 250 $\times$ 4.6 mm; flow rate 1 ml/min; solvent A = water pH 6.5 (with sulphuric acid), solvent B = methanol, solvent C = acetone; gradient: 0 min 20% A, 25% B and 55% C, 1 min 14% A, 24% B and 62% C, 22 min 7.2% A, 12.8% B and 80% C, 42 min 3% A, 7% B and 90% C, 43 min 100% C, 44 min 100% C, 45 min 20% A, 25% B and 55% C; detection at 430 nm.

As β-carotene also has some vitamin A activity it is sometimes added to products for fortification as well as colourant purposes. If this is the case, the methods outlined above can be used. A more recent reference has used a similar method to look at carotenoid isomers in carrot juices and fortified drinks (Marx *et al.*, 2000).

10.10 Vitamin analysis in soft drinks systems

The fortification of some foods has been common practice for years. However, the production of soft drinks containing added vitamins was much less common until about 10 years ago. A few fortified products had been available for a long while, such as Trink 10 in Germany, which is a mixture of ten juices fortified with a range of vitamins, but these were uncommon. Also the fortification of beverages can sometimes offer its own challenges, one being to mask the taste of the added vitamins. However, with the concentration on a healthier lifestyle, fortification is now more common, and the highly successful launch of Sunny Delight, which contains vitamins, in the late 1990s meant that there has been a lot of 'me too' activity. The introduction of the revised Fruit Juice Directive in the United Kingdom in 2003 (Anon, 2003b) has led to the possibility of fruit juices fortified with vitamins and/or minerals, rather than just fortified fruit juice drinks.

If vitamins are added to a product to make a nutritional claim, it is critical that shelf-life studies are undertaken to prove that the overages added are sufficient to ensure that the label claims can be met at the end of product shelf life. This is important as none of the vitamins are fully stable in a soft drink environment and some, for instance vitamin C, are very quickly lost in the presence of oxygen. The addition of the fat-soluble vitamins to a soft drink also offers a formulation challenge to ensure that they are fully dispersed and that there are no problems with neck ringing during storage. Trace metals, particularly the transition elements, can also have a deleterious effect on vitamin shelf life, and sometimes metal scavengers, such as EDTA or phosphate salts, are added to improve the shelf life.

Vitamins are generally assayed in one of two different ways. The first route uses a microbiological technique. Here, a specialist organism is used, the growth of which is dependent on the concentration of the particular vitamin of interest.

These methods are highly sensitive to interferences that limit the growth of the micro-organism and often take rather a long time to perform, as they rely on the growth of micro-organisms under stressed conditions, for example, limited levels of a critical vitamin. However, they do generally offer quite specific methods. Very special skills are required to run these tests effectively.

The second major, and growing, route is the use of HPLC and either UV or fluorescence detection. Here vitamins are extracted from the product and then assayed using HPLC. This is another very specialised area and is often best left to an expert laboratory, as there may be problems with interferences and losses due to such factors as oxidation during extraction.

A few examples of the typical HPLC methods are given below, but if the reader is interested to know more about this area there are two good reviews. One is by Ian Lumley (1993) from LGC in the United Kingdom, who gives good background on the area with information on how the methods have developed. The second is a book which covers all the different types of vitamin analyses using modern chromatographic methods and is edited by Leenheer *et al*. (1992).

It is not uncommon to have to include a hydrolysis step before vitamins are analysed. This can either be to hydrolyse ester groups to liberate the free alcohol, in the case of fat-soluble vitamins, or to remove phosphate groups with some of the B vitamins. There are a large number of references for the analysis of vitamins in foods but few of these were designed specifically for juices or soft drinks. Over the last 5 years, there has been a lot of CEN activity in this area and there are now 10 standard methods published in the British Standards collection for foodstuffs; it is likely that there will soon be more methods published for this type of analysis in foods. The present methods cover vitamins A, B_1, B_2, B_6, C, D, E and K_1.

Although there are a number of citations in the AOAC manual, most of these are once again for foods rather than beverages, but this should not preclude the use of these methods for fortified drinks. There is one reference for vitamin A in fortified milk products (2002.06) and a number of references for a range of vitamins in infant formulations.

10.10.1 Fat-soluble vitamins

If fat-soluble vitamins (A, D and E) are added to a product, the first step in the analysis will often be hydrolysis. In fortified products this converts any esters, for example, vitamin A palmitate or vitamin E acetate, to their free alcohols. The product is then extracted with a hydrophobic solvent, such as hexane or diethyl ethyl or a mixture of these solvents, prior to HPLC analysis often using a reverse-phase (C_{18}) column.

Carotenoids also have vitamin A activity and they can be analysed using the procedures discussed above. Another recent method will separate some of the carotenoids and tocopherol isomers (Schieber *et al.*, 2002) but this is similar to that of Hofsommer described above.

10.10.2 Vitamin B class

There are a number of vitamins in this case, B_1 (thiamine), B_2 (riboflavin), B_3 (niacin), B_5 (pantothenic acid), B_6 (pyridoxine) and B_{12} (cyanocobalamine), and their analysis is complex. Chromatograms are often displayed by column manufactures showing the resolution of the majority of these vitamins in one run. However, this is generally only for standards or premixes. When the vitamins are added to a finished product at the normal levels, the analysis of B_5 and B_{12} is very difficult/impossible by HPLC. The presence of colours in the beverage can also interfere with the analysis of these materials and give erroneously high results. This is where diode array detection (DAD) is quite critical for analysis using UV or visible detection, so that the analyst is able to confirm the integrity of the peak and check for co-eluting materials.

10.10.3 Vitamin C

This is one vitamin that most laboratories can measure. There are a number of old-fashioned approaches that use 2,6-dichloroindophenol in a titrimetric method such as AOAC 985.33. This works well in some systems but can give rise to false positive results if there are other reducing substances present. It will not detect dehydroascorbic acid (DHA) and so it may well underestimate the actual vitamin C activity if a product contains a significant level of DHA. However, even with for these shortcomings, it is often used as a quick and rough method. In the AOAC there is also a fluorometric method (AOAC 984.26) where ascorbic acid is oxidised to DHA and this is reacted with *o*-phenylenediamine to give a fluorometric compound which can be detected. This is a robust method that has general applicability.

Vitamin C can also be effectively detected in juices and soft drinks after treatment with a reducing agent such as dithiothreitol or cysteine and then quantified using HPLC on either a reverse-phase or an amino-bonded column. This type of approach was adopted in the recently published EU method (Anon, 2003c). Vitamin C is one of the vitamins that should be routinely assayed in a range of juices such as orange, grapefruit and blackcurrant that are all good sources.

Another vitamin which is of interest in fruit juices is folate, which can be found in low, but significant, levels in some juices, such as orange. However, the analysis of natural levels of this vitamin is very difficult and is best performed using microbiological methods.

Although microbiological and HPLC methods are the most commonly used methods for analysis of vitamins, there are some immunological methods available for the determination of some of these compounds. These are either a standalone enzyme linked immunosorbent assay (ELISA) method such as the method for B_{12}, folate and biotin which is sold by r-Biopharm (http://www.r-biopharm.de) or the use of a dedicated piece of equipment such as the Biacore. This is a sophisticated instrument that permits immunological assays; instead of using changes in the visible absorbance of a solution, detection is by changes to the response of the surface of a sensor chip. At present there are kits available for the analysis of vitamins which are more difficult to assay by normal HPLC methods (e.g. B_{12}, folate and biotin) and recently a method for B_2 has been launched. For more information on this type of assay go to the Biacore website (http://www.Biacore.com).

10.11 Methods used to detect juice adulteration

Over the past 30 years, extensive research has been carried out to find ways to detect the adulteration of fruit juices. The approaches have developed from simple procedures, such as measuring the potassium and nitrogen contents of juices, to the use of highly sophisticated and expensive equipment to detect the most recent approaches that unscrupulous suppliers may be using to extend their products. Such adulteration often involves the substitution of some of the fruit juice solids by sugars derived from beet, cane, corn or inulin, or the addition of cheaper juices or second extracts of the fruit.

The methods used to detect adulteration of juices have been reviewed by a number of authors and two examples are given here (Fry *et al.*, 1995; Hammond, 1996). With the changing methods of unscrupulous suppliers it is now common to use a battery of tests to ensure that a product is authentic. Although this is costly, it is the only way to ensure the authenticity of a product and protect company reputations. The array of tests will often include a number of the procedures described above, such as sugar and acid profiles, along with other methods such as stable isotopic and fingerprinting procedures.

A number of multi-component approaches have been used to assess fruit juices such as the German RSK system and the French AFNOR system. However, it is now common across Europe to use the Association of the Industry of Juices and Nectars from fruits and vegetables of the European Union (AIJN) Code of Practice (COP) (Anon, 2004b), which can be viewed at the association's website for a fee (http://www.AIJN.org). In fact it is mandatory in countries that form part of the European Quality Control System (EQCS) to use these guidelines to 'judge' the quality of 'juices' on sale within that market. The EQCS is a voluntary industry self-control scheme which has gradually grown out from the German system and now covers most of the EU states and also a number of the extension members of the Union. Its principles are to ensure that products on sale within the countries are all authentic and that there is fair and even competition between the various companies. In doing this, it also protects the interests of the consumer and the good and healthy image that fruit juices have in the eyes of the general public. It offers a number of independent, but interlocking, schemes which control the quality of the products in their markets and a network to alert other schemes to any problems that have been detected in their market.

All the compositional guidelines – RSK, AFNOR and AIJN – define ranges for a number of analytes within which a sample's data should fall. These are valuable approaches but they should not be used to the exclusion of other, newer procedures, which are sometimes more sensitive to the more sophisticated approaches that adulterators now have to use to extend their products with any hope that they will not to be detected.

Individual companies can do some of the analyses, including most of those laid down in the AIJN COP, to safeguard their own name, but very few companies have the time or money necessary to carry out all these tests or some of the isotopic procedures. This should not prohibit them from using such procedures as there are contract laboratories around the world that can carry them out. They should also, if possible, use a list of approved juice suppliers whom they have inspected and with whose production methods they are happy. This is expensive but could limit a company's exposure to bad publicity or prosecution if problems occur in the market, such as happened in 1995–6 when a large number of well-known companies were found to have purchased apple juice adulterated with syrups derived from inulin. This was detected using a new capillary gas chromatographic fingerprinting method (IFU recommendation No. 4, Low & Hammond, 1996; Anon, 2004c).

Another way of tackling raw material supplier audits is to subcontract them to an outside body or use Germany's Schutzgemeinschaft (SGF) International Raw Material Assurance (IRMA) programme, details of which can be found on its website (http://www.sgf.org). The IRMA scheme is a system set up some years ago by a wide range of suppliers who have the same aims as the SGF, for example, to produce high-quality products. These suppliers are open to unannounced audits by the IRMA team at any time to ensure that these standards are being met. Although the use of approved suppliers does not exclude the need for a company to test incoming materials, it does limit the risk and means that a lower level of testing is required than would otherwise be necessary.

Fingerprinting methods such as the anthocyanin methods and the Kirksey method for polyphenols (Kirksey *et al.*, 1995) offer good ways to check for the addition of other fruits in a product. As the adulterators have become more sophisticated in the approaches that they use to extend juices, there has been a need for more complex methods of analysis. This means that it is now not uncommon to have to use fingerprinting techniques and isotopic methods to detect the most sophisticated forms of adulteration. These sophisticated analytical methods can even involve detection of the isotope ratios within a class of compounds such as sugars (Hammond *et al.*, 1998). Using the RSSL ^{13}C-IRIS approach, which was developed with financial support from the UK Food Standards Agency, it was possible to reduce the detection limit for the addition of C_4-derived sugars to juices by about a factor of two.

Sometimes it is even important to look at the internal isotope ratios seen within a molecule such as malic acid in apple juice. Two groups, Isolab in Germany and Eurofins in France, found it was useful to look at the carbon isotope ratios at the C_1 and C_4 positions of malic acid extracted from apple juice. This allowed them to detect the addition of synthetic L-malic acid to apple juice at much lower levels than would be possible by other means (Jamin *et al*., 2000).

However, sometimes simple methods such as that developed by Low in the early 1990s using capillary gas chromatography, which is readily available in well-equipped laboratories around the world, can expose major problems of adulteration (Low & Hammond, 1996). Here a well-established technique, analysis of sugars by capillary gas chromatography, was able to be applied to detect two routes that were known to be used to extend juices, the addition of HFCS and invert syrups. The method also discovered the adulteration of apple juices with another type of sugar syrup that had only been proposed as a possible adulterant.

10.12 Methods used to assess the juice or fruit content of a soft drink

Under the UK Soft Drinks Regulations 1964 certain products had defined minimum juice contents or minimum fruit contents, for example, a fruit crush had to have a minimum juice content of 5% and a comminuted drink had to contain a minimum level of fruit (10%) before dilution. When these regulations were revoked in 1995 the various statutory descriptions such as ‘drink’ and ‘crush’ were removed but the need to define the juice content of a product was not. Under the EU regulations (Anon, 1997b) covering the labelling, presentation and advertising of foodstuffs, the so-called QUID (quantitative ingredient declaration) amendment, manufacturers have to declare on the label of a food the levels of characteristic ingredient(s); for example, in an orange juice drink a manufacturer will have to label the juice content of the product. The regulations allow the manufacturer to list the juice content based on the level of juice in the product formulation. Although this means that the producer can put a calculated value on the label, it will still be up to the regulatory authorities to check that this value is appropriate and they will have to assess the product to determine whether the label is correct.

A similar situation exists in the United States with the Nutrition Labelling and Education Act (NLEA). When this was introduced in 1990, it placed the onus on the manufacturer to label the product with the individual level of the component juices in, say, a 100% juice blend or the quantity of juice in a soft drink claiming to contain fruit juice on the packaging. The conditions for labelling, together with information concerning how the juice content should be assessed, including the minimum Brix level considered acceptable for a range of single-strength juices, are given in the Code of Federal Regulations (CFR) 21 CFR.101.30 (Anon, 1990).

The estimation of juice or fruit content is a difficult area and a number of approaches have been suggested. These have involved assessing the ash content, levels of potassium ions, phosphorous content, total level of free nitrogen by measuring the formol value, levels of specific amino acids and a range of other components. The simplest of these procedures, the assessment of the total nitrogen content, phosphorous and potassium levels (N, P, K), was proposed by Hulme *et al.* (1965) as a method to assess the fruit content of comminuted orange drinks. This type of approach can also work for other juices where the levels of these components are well documented. Useful references in this area are the RSK values (Anon, 1987b), *McCane and Widdowson's The Composition of Foods* (Holland *et al.*, 1991), Souci *et al.* (1981) and the AIJN Code of Practice (Anon, 2004b), which give compositional information on a wide range of juices that can be used to estimate the juice or fruit content of a product.

However, if only a simple approach to establish the juice content of a drink is used, such as assessing the levels of nitrogen, phosphorus and potassium, it is easy for an unscrupulous supplier to circumvent this method of assessment by the addition of potassium and ammonium phosphate salts, which enhances the nominal juice content of the product.

Alternative testing procedures have been suggested by other authors, such as the use of certain amino acids (Ooghe & Kastelijn, 1985) and organic acids (Wallrauch, 1995). However, because of the wide natural variation of all these components, any calculation of juice or fruit content should really be considered only as an estimate rather than an absolute value. The situation is even more complicated for a mixture of juices in a juice blend or soft drink. In this case, the parameters chosen for analysis, often potassium or phosphate, should be fairly constant between the juices within the blend, and if possible components that are characteristic of a particular juice, such as quinic acid in cranberry juice, or isocitric acid in orange, grapefruit or lemon, say, should be analysed. This type of specific marker can be used to assess the overall level of a particular juice in a blend, but not every juice has a unique marker.

10.13 Conclusions

It is essential for soft drinks companies to assess their products on a batch-to-batch basis to ensure that there have not been errors in the syrup formulation, for example. This is possible with HPLC systems that can be used near to the production line and now require less-skilled staff to operate them than was the case 10 years ago. In the future it may be possible to use in-line monitoring of the product using infrared or near-infrared spectroscopy to ensure proper product formulation. In fact, FOSS offers a system at present that can determine a wide range of parameters – ethanol, pH, volatile acidity, total acidity, levels of tartaric, malic, gluconic, lactic and acetic acids, reducing sugar content, glucose to fructose ratio and density – in wines and musts from one simple measurement (http://www.foss.dk), which is perhaps an indication of things to come.

Fruit juice companies can themselves do a lot of the testing required to assess whether a product is authentic. However, they may not have the expertise necessary to assess the fine detail and an analyst without sufficient breadth of knowledge in this field might miss some subtle deviations in the data for a sample that would be detected by an expert.

When setting up a scheme to control the authenticity and quality of raw materials, a range of tests should be used to assess samples as this will provide the best chance of detecting any problems that exist. This does not mean that every test conceivable should be carried out on every sample; a range of tests should be applied on a programmatic basis and this will give the best chance of detecting any deviations from normal. One of the most critical features in the authenticity area is to know your supplier; if this is the case you are less likely to have problems.

Cautionary note: *A number of the HPLC methods listed in this chapter use phosphate buffers as solvents. It should always be remembered that phosphate salts are very abrasive to piston seals and pistons. If using these systems, always remember to wash out the aqueous pump thoroughly with water at the end of a run. If this operation is not carried out, the piston seal lifetime will be significantly reduced and in some cases, if left too long, pistons can stick and possibly fracture, which will be expensive to repair/replace.*

In the early part of 2004 there was a problem in the UK caused by low levels of bromate in a branded bottled water. This arose from the presence of low levels of bromide in the water that was then disinfected by treatment with ozone. The bromate ions formed were at levels above the EU and EPA limit of 10µg/l for drinking water. The analysis of this anion at trace levels is demanding and should be left to a specialist laboratory. However, Dionex have published four methods that can be used for the analysis of bromate ions in water and the application notes (81, 101, 136 & 149) are available from the Dionex website (http:/www.dionex.com/)

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11 Microbiology of soft drinks and fruit juices

P. Wareing and R.R. Davenport

11.1 Introduction

Soft drinks and fruit juices represent an important market within the food industry. The increasing variety of products being released at a bewildering rate has altered the potential for spoilage problems. Soft drinks are generally nutrient-poor media that are spoiled by relatively few organisms – usually yeasts, and a few acid-tolerant bacteria and fungi. Carbonation shifts the spoilage flora to those organisms tolerant of carbon dioxide. Soft drinks enhanced by the addition of low levels of fruit juice tend to exhibit similar spoilage flora to fruit juices. The use of ever more exotic raw ingredients may lead to the discovery of unusual spoilage organisms in the future. Yeasts in general, and *Zygosaccharomyces bailii* in particular, remain the key spoilage organisms because of their overall physiology and resistance to organic acid preservatives (Stratford *et al.*, 2000).

Microbial problems within soft drinks and fruit juices can be divided into two groups:

(1) growth in, and deterioration of, the product by general organisms to produce spoilage;
(2) growth in, or contamination of, the product by pathogens to produce food poisoning.

There have been relatively few instances of food poisoning in fruit juices or soft drinks, but microbial spoilage is very common. The previous edition of this book contained an excellent review and practical guide to the identification of spoilage problems in the soft drink industry by the late Professor Davenport (1998). The present review will therefore keep key parts of the previous text, enhancing it with recent data, and simplifying some of the information. For example, some new information on acid-tolerant bacteria such as *Alicyclobacillus acidoterrestris*, and data on the pathogens *Escherichia coli* O157:H7 and *Salmonella* spp. within fruit juices, merit examination, as do some novel processing methods.

11.2 Composition of soft drinks and fruit juices in relation to spoilage

There is a bewildering variety of soft drink and fruit juices for sale, and many methods for their manufacture. Soft drinks can be non-carbonated, carbonated, with or without added fruit juice, often with the addition of organic acid preservatives. They can be filled on standard or clean fill lines. Fruit juices, fruit juice concentrates and fruit nectars may be fresh, unpasteurised and clean filled, or pasteurised, then hot, aseptic, or clean filled (Stratford *et al.*, 2000; Stratford and James, 2003). Recent technology using ultra-high pressure has been used to produce 'cold pasteurised' fruit juices. These have the advantage of a fresh juice mouthfeel, but with destruction of pathogens and the majority of spoilage agents, enhancing the shelf life of an essentially fresh product (Mermelstein, 1999; Zook *et al.*, 1999).

Simple soft drinks such as orangeade and lemonade are too acidic for the growth of most organisms, so that spoilage is generally by carbonation-resistant species such as *Dekkera anomala* (Stratford and James, 2003). Yeasts usually require a carbon source such as a hexose sugar, a nitrogen source such as amino acids or ammonium salts, simple salts (phosphate, sulphate, potassium and magnesium ions), trace minerals and vitamins. Some yeasts have particular sugar requirements; for example, *Z.bailii* and *Z.rouxii* cannot utilise sucrose (Pitt & Hocking, 1997; Stratford *et al*., 2000).

Sugars have a protective effect on the heat resistance of yeasts and bacteria; this is an important consideration at higher concentrations of sugar. Soft drinks are often nitrogen poor and thus the addition of fruit juice greatly enhances the potential for spoilage. Some yeasts, for example *Dekkera bruxellensis*, can use nitrate. Phosphate levels are often low, trace minerals satisfactory, particularly in hard water areas. The low pH value of soft drinks and fruit juices, pH 2.5–3.8 (Table 11.1), inhibits most bacteria, but leaves yeasts unaffected. In soft drinks

Table 11.1 Examples of fruit and vegetable juice pH and risk organisms

	Approximate pH ranges	Risk organisms
Fruits		
Apples	2.9–3.91	Yeasts
Grapes	3.20–4.51	Yeasts
Oranges	3.20–4.3	Yeasts
Raspberries	3.12	Yeasts
Blackcurrants	2.48–3.60	Yeasts
Pineapples	3.3–3.7	Yeasts and bacteria
Mangoes	3.95–4.50	Yeasts and bacteria
Tomatoes	3.80–4.80	Yeasts, bacteria and moulds/bacteria
Vegetables		
Carrot	4.90–6.44	Bacteria
Celery	5.7–6.1	Bacteria
Cabbage	5.4–6.0	Bacteria
Pea	6.65–6.77	Bacteria

and fruit juices, oxygen levels are usually low, and CO_2 levels either low or very high (in carbonated soft drinks). Spoilage is therefore due to facultative anaerobes, organisms that can grow with or without oxygen. In carbonated drinks, mould and bacterial growth is very unlikely as they are very sensitive to CO_2.

The recent trend towards polyethylene terephthalate (PET) packaging presents its own problems. Typically PET containers cannot be hot filled; around 50°C is the upper temperature, although some recent types can be heated to 85°C. They are also permeable to oxygen (Rodriguez *et al.*, 1992), which allows the growth of aerobic spoilage agents.

11.3 Background microbiology – spoilage

Many micro-organisms are found in soft drinks as environmental or raw material contaminants, but relatively few can grow within the acidic and low-oxygen environment. Yeasts are the most significant group of micro-organisms associated with spoilage of soft drinks and fruit juices. Spoilage will be seen as the growth and production of metabolic byproducts, for example, CO_2, acid, and tainting compounds. As noted above, most spoilage is therefore by yeasts and mould species, with yeasts most important, and some spoilage is by acid-tolerant bacteria (Hocking & Jensen, 2001; Jay & Anderson, 2001).

11.3.1 Sources

Fruit and fruit juices are commonly contaminated with yeasts and moulds, often from insect damage. Fallen fruit should thus be avoided where possible, for all of the risks outlined below. Sugars and sugar concentrates are commonly contaminated with osmophilic yeasts, for example *Z.rouxii*. Growth is slow in concentrated solutions, but one cell per container of diluted stock is enough to cause spoilage (Davenport, 1996). Flavourings, water and other chemicals are all potential sources of microbial contamination. Process machinery and filling lines are particularly problematic and strict hygiene is essential.

11.3.2 Yeasts

There are over 800 species of yeasts currently described (Barnett *et al.*, 2000), but only about 10 are commonly associated with spoilage of foods prepared in factories operating good standards of hygiene and using correctly applied chemical preservatives (Pitt & Hocking, 1997). Others are found if something goes wrong during manufacture; for example, incorrect preservative level, poor hygiene or poor-quality raw ingredients.

Davenport (1996, 1997, 1998) describes a simple classification scheme for yeasts causing spoilage in the soft drinks industry, which, for investigative purposes, is used in Sections 11.6 and 11.7 and Appendices 11.1–11.4 in this chapter. He found that the yeasts isolated could be divided into four categories: Groups 1–4. Group 1 yeasts are described as spoilage organisms that are well adapted to growth in soft drinks, able to cause spoilage from very low cell numbers (as few as one cell per container). The characteristics of Group 1 yeasts are osmotolerance, aggressive fermentation, resistance to preservatives (particularly weak organic acids) and a requirement for vitamins. *Z.bailii* is a typical example of this group, and this group corresponds closely with Pitt and Hocking's (1997) 10 key spoilage yeasts. Davenport describes Group 2 organisms as spoilage/hygiene types, able to cause spoilage of soft drinks, but only if something goes wrong during manufacturing, for example, too low a level (or absence) of preservative, ingress of oxygen, failure of pasteurisation or poor standards of hygiene. Group 2 organisms are common contaminants in factories, but can be severely restricted if good manufacturing practices (GMP) are adhered to strictly. Group 3 organisms are indicators of poor hygiene standards. These yeasts will not grow in soft drinks, even if present in high numbers, and are typical of the yeasts found in many factories; the higher the count, the worse the hygienic state of the factory. Group 4 yeasts, called 'aliens' by Davenport, are those out of their normal environment. An example would be *Kluyveromyces lactis*, a dairy spoilage yeast. Table 11.2 details the most common organisms in each group.

Table 11.2 Examples of yeast species found in soft drink factory environments

Group 1 – Fermentation and preservative resistance	Group 2 – Spoilage and hygiene	Group 3 – Hygiene	Group 4 – Aliens
Dekkera anomala	*Candida davenportii*	*Aureobasidium pullulans*	*Kluyveromyces marxianus* (dairy yeast)
D. bruxellensis	*C. parapsilopsis*	*Candida sake*	*K. lactis* (dairy yeast)
D. naardenensis	*Debaryomyces hansenii*	*C. solani*	
Saccharomyces cerevisiae (atypical strains)	*Hanseniaspora uvarum*	*C. tropicalis*	
S. exiguus	*Issatchenkia orientalis*	*Clavispora lusitania*	
Schizosaccharomyces pombe	*Lodderomyces elongisporus*	*Cryptococcus albidus*	
Zygosaccharomyces bailii	*Pichia anomala*	*C. laurentii*	
Z. bisporus	*P. membranifaciens*	*Debaryomyces etchellsii*	
Z. lentus	*Saccharomyces bayanus*	*Rhodotorula glutinis*	
Z. rouxii	*S. cerevisiae*	*R. mucilaginosa*	

Sources: Based on Davenport (1996); Stratford & James (2003).

Table 11.3 Names of the most important yeasts within the soft drinks industry, with synonyms

Current Name	Synonym
D. bruxellensis	*Brettanomyces intermedius/B.bruxellensis*
D. naardenensis	*B. naardenensis*
Saccharomyces cerevisiae	*Saccharomyces carlsbergensis*
S. bayanus	*S. uvarum/S.heterogenicus*
S. exiguus	*Candida/Torulopsis holmii*
Zygosacchaaromyces bailii	*Saccharomyces bailii*
Z. bisporus	*Saccharomyces bisporus*
Z. lentus	*S. lentus*
Z. rouxii	*S. rouxii*
Torulaspora delbreuckii	*S. delbreuckii/S. rosei /Candida colliculosa*
Pichia anomala	*Hansenula anomala*
P. membranifaciens	*Candida valida*
Issatchenkia orientalis	*C. krusei*
Kluyveromyces marxianus	*C. kefyr*
K. lactis	*C. sphaerica*
Debaryomyces hansenii	*C. famata*
Pichia guilliermondii	*C. guilliermondii*
Hanseniaspora uvarum	*Kloeckera apiculata*
Metchnikowia pulcherrima	*Candida pulcherrima*

One of the most confusing aspects of mould and yeast taxonomy is the frequency with which names are changed. Many yeasts and moulds have sexual and asexual stages that may have different names. Sometimes, the association of a yeast with a particular genus is recognised as wrong, and a name change then occurs. The current trend is to name the organism by the sexual name, with the asexual stage described as a synonym. Thus, *Candida famata* (asexual) becomes *Debaryomyces hansenii* (sexual). It is important to be aware of this variation when conducting literature searches. Names do not always reflect the spoilage potential – *Saccharomyces cerevisiae* is an important yeast for the brewing and baking industries, but a disaster for the soft drinks and fruit juice industry. Table 11.3 details alternative names for important yeasts.

11.3.3 Bacteria

Bacteria that have been associated with spoilage in the soft drinks industry include Acetobacter, Alicyclobacillus, Bacillus, Clostridium, Gluconobacter, Lactobacillus, Leuconostoc, Saccharobacter, Zymobacter and Zymomonas (Vasavada, 2003). Gluconobacter is a common spoilage agent of fruit juices; it is a strict aerobe, requiring free oxygen (Stratford *et al.*, 2000).

Alicyclobacillus spoilage is becoming an important issue in heat-treated fruit juices, and the bacterium is commonly found contaminating fruit in the field. Growth of the organism is associated with the production of antiseptic and 'smoky' taints within juice. The former is due to 2,6-dibromophenol (2,6-DBP), giving a 'TCP' flavour (Jensen & Whitfield, 2003); the latter is due to guaiacol (2-methoxyphenol) (Jensen, 1999). Heat shock, taint precursors, incubation temperature and oxygen are all important factors in the production of taints (Jensen, 1999).

Alicyclobacillus acidoterrestris is a thermotolerant, spore-forming aerobic organism that can survive fruit juice heat treatments to grow in the juice. Spoilage can occur from inoculum levels as low as 1 spore per 10 ml (Pettipher & Osmundon, 2000; Walls & Chuyate, 2000a). Growth minima and maxima at temperatures of 26–50°C, minimum pH 2.0, D_{95} of 2.4 min. Isolation methods have been compared (Pacheco, 2002; Pettipher & Osmundson, 2000; Silva & Gibbs, 2001, Walls & Chuyate 2000). Effective control can be achieved by rapid chilling of juice to below 20°C after pasteurization, use of sorbate or benzoate, removal of oxygen or addition of ascorbic acid (Cerny *et al*., 2000) or by using an appropriate pasteurisation regime (Silva & Gibbs, 2001). Surface disinfection of fruit using chlorine or 4% peroxide has also proved effective (Orr & Beuchat, 2000).

11.3.4 Moulds

Mould problems can be divided into two types: growth of a variety of moulds due to poor hygiene within the factory or field environment, and growth of heat-resistant moulds within heat-processed juices. The former type can cause tainting, discolouration and other general problems associated with gross mould growth. The latter type can result in slow growth of the mould within the processed product. There is some overlap between the two groups. Xerophilic (highly sugar-tolerant) fungi are likely contaminants if hygiene is poor.

Heat-resistant moulds able to cause spoilage of fruit juices and soft drinks include *Aspergillus ochraceus*, *Aspergillus tamarii*, *Aspergillus flavus*, *Byssochlamys nivea*, *Byssochlamys fulva*, *Paecilomyces variotii*, *Neosartorya fischeri*, *Eupenicillium brefeldianum*, *Phialophora mustea*, *Talaromyces flavus*, *Talaromyces trachyspermus* and *Thermoascus aurantiacum*. Others include *Penicillium notatum*, *Penicillium roquefortii* and *Cladosporum* spp. (de Nijs *et al*., 2000; Pitt & Hocking, 1997; Stratford *et al*., 2000). Mould spoilage within the factory is associated with poor hygiene. Various types of heat-resistant spores can be produced: ascospores, chlamydospores and sclerotia.

Many of the above moulds are found on fruits pre- and post-harvest. Most moulds require oxygen to grow. Growth is exhibited as surface mats, sometimes producing copious spores. Some moulds produce extracellular degradative enzymes, such as pectinases. Detection of heat-tolerant moulds is usually carried out by plating out a sample of heat-shocked juice. Mould identification is carried out by reference to standard morphological texts, and is difficult unless carried out by experienced personnel. An excellent scheme that combines basic physiology with simple morphological attributes was developed by Pitt and Hocking (1997). It uses growth on three media at three temperatures to differentiate moulds: 5, 25 and 37°C, a high sugar content (xerophilic), a closely defined medium with nitrate as a nitrogen source and sucrose as hexose sugar, and a general medium. This technique is particularly useful for the differentiation of *Penicillium* species.

11.3.4.1 Mycotoxins

Mycotoxins are toxic secondary metabolites produced by fungi growing within or on foods. They can be a serious threat to human and animal health (Nagler *et al*., 2001). Table 11.4 details mycotoxins associated with soft drinks and fruit juice manufacture and raw materials. Patulin is the most common mycotoxin associated with fruit juice, particularly apple juice (Pitt & Hocking, 1997). It commonly occurs if juice is produced from stored apples. Mould growth in infected apples increases with time, raising levels of patulin. The use of windfall apples for juice is also a factor. Avoidance of windfall apples, filtration of juice and pressing quickly after harvest are all methods to reduce the incidence of patulin in juice. Patulin can be destroyed by fermentation to cider or by the addition of ascorbic acid (Marth, 1992). Within Europe, the European Union has set a limit of 50 μg/kg for patulin in both apple juice and cider. A recent survey of apple products in Chile found that 28% of samples of juice and concentrate exceeded this limit (Canas & Aranda, 1996).

Table 11.4 Examples of toxin-producing moulds associated fruit with fruit raw materials and soft drinks products

Fruit(s)	Mould	Toxin(s)
Apple	*Penicillium expansum*	Patulin, citrinin, roquefortine C
Citrus	*P. digitatum*	Trptoquivalins
Carbonated beverages	*P. glabrum*	Citromycetin
Bottled water	*P. roqueforti*	Roquefortine C
Diluted fruit/water beverages	*P. roqueforti*	Isofumigaclavine A and B
Treated orange juice	*Fusarium oxysporum*	Oxysporone
Fruit juices	*Aspergillus versicolor*	Geosmin sterigmatocystin

11.4 Microbiological safety problems

E. coli and enterococci have been isolated from citrus juices, and apple juice has been associated with Cryptosporidiosis. Contamination by hepatitis A and Norwalk-like viruses has been reported in fruit juices (Vasavada, 2003).

11.4.1 Escherichia coli

There have been a number of cases of food poisoning associated with pathogenic *E. coli* O157 contamination of fresh apple juice (Battey & Schaffner, 2001; Czajka & Batt, 1996; Miller & Kasper, 1994; Parish, 1997, 2000; Splittsoesser *et al*., 1996; Zhao *et al*., 1993), which was found to be able to grow in apple juice with low pH. The organism potentially has a low infective dose of <100 cells, and this, coupled with the occasional severe side-effect of haemolytic uraemic syndrome, makes *E. coli* O157 a severe hazard within unpasteurised juices.

11.4.2 Salmonella

Salmonella is generally only a problem in fresh, unpasteurised fruit juices, due to its low thermal tolerance. There have been three major outbreaks of Salmonellosis, all from unpasteurised orange juice, in the United States, Canada and Australia, involving 62, 298 and 400 reported cases in 1995 and 1999 (Bell & Kyriakides, 2001). *Salmonella* Hartford, *Salmonella* Muenchen and *Salmonella* Typhimurium were involved. The main risk factors were associated with the fertilization of agricultural crops: crops were grown in orchards where sheep grazed, manuring the soil. Fallen fruit were often used, allowing soil and faecal contamination. In addition, poor decontamination of fruit occurred in the factory; poor-quality wash water was used; pest control within the factory was poor; and the cleanup of fruit boxes and conveyors was poor. Data indicated that *Salmonella* could survive for up to 300–968 days in soil treated with animal slurry. Other studies showed that fresh juice had to be stored for 15–24 days at 4°C to achieve a 10^6 reduction from 10^7 to 10^1 (Bell & Kyriakides, 2001).

11.5 Control Measures

Control measures include maintaining the cleanliness of equipment, the control of storage temperature, hot water immersion, chemical sanitizers, surfactants, surface waxes (particularly on oranges) and UV irradiation. The chemical preservative dimethydicarbonate (Velcorin) has been used to 'cold pasteurise' fresh juice products to reduce microbial loading, minimising the use of sulphur dioxide (Threlfall & Morris, 2002; Vasavada & Heperkan, 2002). High-pressure processing, a pulsed electric field and ultraviolet light have also been investigated as control measures. Modelling of yeast and bacterial growth has been used as a technique for determining effective preservative and other control regimes (Battey & Schaffner, 2001; Shearer, *et al*., 2002).

Good hygienic practices and adherence to GMPs are the most effective control measures for microbial contamination in the soft drinks industry, particularly for yeasts. Sticky sugar and fruit residues are ideal food sources for yeasts and moulds. The Hazard Analysis and Critical Control Point (HACCP) approach has been adopted by food processors around the world. In the United States, HACCP is mandaory for fruit juice processors, with good agricultural practices (GAP) as the foundation of a successful HACCP system. In Europe, growers, distributors and packaging houses must meet the EUREGAP protocols if they wish to be certified to sell their products to certain markets or established buyers (Stier & Nagle, 2003).

11.6 Forensic grouping and applications

11.6.1 Forensic grouping

Forensic grouping involves consideration of simple behaviour patterns of micro-organisms as evidence for decision-making. Three answers are obtained from each test, namely, activity (growth/no growth), potential significance/role (related to product and/or product environment) and tentative recognition/identification (i.e. most probable genus; sometimes the name of five possible species). Emphasis is placed on simple economic methods where selected raw materials and products can be used as liquid or solid media. Some commercial bottles and containers can be used in place of expensive laboratory glassware. This latter method, combined with some conventional testing, gives the best evidence for decision-making, rather than providing data for historical records. The forensic system is also a practical approach that marries well with HACCP procedures.

11.6.2 Group formats

Each micro-organism group has a basic behaviour pattern common to all members. Members will have other characteristics that separate them as individuals. However, the group system is best operated in a series of simple steps where behaviour patterns are determined by a combination of some conventional identification methods and technology parameters (e.g. preservative resistance/sensitivity). Each step provides evidence for current use but can also indicate the best direction for further testing steps in both technology and taxonomy. Table 11.5 gives the essential patterns of the micro-organism groups that are suitable for soft drink and fruit juice products (see appendices for further details).

Table 11.5 Baseline behaviour patterns of the most common micro-organisms associated with soft drinks and fruit juices (Forensic Groups for Practical Applications and Assessments)

Group	Status	Behaviour pattern						Micro-organisms
		Fe	Fi	Pr	Ac	Os	Vi	
1	Spoilage	+	−	+	−	+	+	*Zygosaccharomyces bailii* *Saccharomyces cerevisiae* *Torulaspora delbrueckii* *Schizosaccharomyces pombe*
2	Spoilage and hygiene	−	+	−	+	±	−	*Pichia anomala* *P. membranaefaciens* *Debaryomyces hansenii* *Acetic acid bacteria*
3	Hygiene	−	±	−	+	−	−	*Geotricium candidum* *Trichosporon* spp. *Rhodotorula glutinis* (red/pink yeast) *Bacillus* spp. (bacteria with spores)
4	Special Indicators	+	+	±	+	−	+	*Kluveromyces marxianus* (dairy yeast) *Brettanomyces/Dekkera anomalus* (brewery origin)

Table contents modified from Davenport (1995a,b, 1996, 1997).
Fe = fermentation (strong, i.e. in presence of/production of $<5\%$ v/v alcohol).
Fi = surface film formation.
Pr = preservative resistance, principally common chemical agents, e.g. benzoic acid.
Ac = acitidione (antifungal compound) >10 ppm.
Os = growth at high osmotic pressure sugar $>60^\circ$Brix.
Vi = one or more B group vitamins required.
Note: All B group vitamins are essential for yeast growth. Some yeasts can synthesize all their requirements (e.g. *Pichia anomala*); the remainder can synthesize only part of the vitamin B group, hence the remainder of the set must be supplied externally.

11.7 Suggested test programme

11.7.1 Packaged products

11.7.1.1 Spoilage symptoms: dominant feature fermentation

Action points:

(i) Check formulation data and. other product history.
(ii) Assess spoilage incidence, that is, spasmodic, increasing, major, etc.
(iii) Plan an appropriate programme, that is, sample:
- direct microscopy
- record cell shape
- follow outline as in Table 11.6

Table 11.6 Scheme for soft drink forensic microbiological investigations

1 All possible information on history, analyses and other documentation should be assessed, i.e. environmental characteristics and micro-organism characteristics together determine the outcome of the organism.
2 An overall objective should be stated based on (1). In practice, this objective will have to be divided into a series of sub-objectives. The most efficient practical test regime(s) can then be designed to meet the objective(s).
3 The investigation should be designed on the following criteria:
  3.1 Both academic and technical aspects must be covered (academic = foundation of good recognised knowledge for some routine testing plus 'extra' input to allow data/responses to be assessed for future needs; technical = testing designed specifically for the objective in hand).
  3.2 Testing programmes should be significant, i.e. designed to suit the problem, not to copy an accepted publication. Too often the preoccupation with standard methods to get counts within range or correct names can be expensive and time-consuming.
  3.3 Testing should be kept as simple as possible.
  3.4 Staff should be suitably trained in bench skills and should have a clear simple under standing of beverage microbiology.

(iv) For spasmodic spoilage outbreaks, the capping system is likely to be the most frequent source of contamination.
(v) Method for (iv)
Place a new autoclave bag over the end of the closure jaws. Take a sterile swab and knock about 30–50 caps into the bag. Pour on sterile mixture of 80% product plus 20% water. Tie up the bag and leave in a warm place at 22–26°C. Observe for up to one week. If no fermentation occurs, then caps are an unlikely contamination source. If fermentation does occur, then action is required. If results indicate activity, this is evidence that technology needs attention. The most probable identity of the micro-organisms is restricted to a very few in Group 1 (see Table 11.5).
(vi) Major spoilage outbreaks mean either an inefficient cleaning programme or a possible formulation fault.
(vii) With (vi) a survey is required to determine whether this spoilage is restricted to one or two places or is more widespread.

11.7.1.2 No obvious symptoms

11.7.1.2.1 Clear products – suitable for membrane filtration. Action points:

(i) Proceed with routine membrane filtration test.
(ii) Count colonies on the membrane or place the membrane in a product/ water mixture and observe for fermentation (Section 11.7.1.1(v)) or

(iii) Pour plates: count colonies, recount colonies that look like 'stars' and calculate the percentage of stars. This percentage gives an index of strong fermentative organisms.
(iv) Pour plate contents may be put into product/water mixture and observed for fermentation (Section 11.7.1.1(v)).
(v) For further identification the test in Table 11.6 can be used.

11.7.1.2.2 Fruit juices and unfilterable drinks. Split bottle method. Action points:

(i) Proceed with routine test.
(ii) Take a very clean container (plastic or glass bottle). Carefully decant half the product contents into the container. Add about quarter sterile water to one bottle – both bottles will have heat spaces. Leave for one week at 22–26°C. Observe for fermentation.
(iii) Further identification can be carried out based on the behaviour of the organism in different media (see Table 11.7).

11.7.2 Key raw materials

Raw materials are the main means of carrying spoilage yeasts into the factory. Materials include liquid sugars, juice concentrates, comminutes and natural colors and flavours. Sample size is 250 ml (minimum).

Table 11.7 Group pattern-making: simultaneous assessment of behaviour and tentative identification of yeasts (part of a model for soft drinks)

Groups	Fermentation YM broth or product	YM	YMS	YM1	YM60	ACT	Sexual
1	+	+	+	+	+	−	+
2	±	+	±	−	±	±	+
3	−	+	−	−	−	+	±
4	+	+	+	−	−	+	+

AGARSYM
YM (oxoid difco).
YM5YM + 0.5% acetic acid (add just before pouring plate).
YM1YM + 1% acetic acid (add just before pouring plate).
YM60YM + 60% (w/w) glucose.
ACT: Actidione agar
Sexual states: ascospores for Zygosaccharomyces, Saccharomyces, Torulaspora, Schizosaccharomyces, Pichia, Debaromyces, Geotrichum (very rare), Kluveromyces and Dekkera (rare).

Action points:

(i) Collect data.
(ii) Make up a solution of the raw material at 20°Brix using sterile water or glucose solution
(iii) Add 0.5 ml if 10% yeast extract solution (10 g yeast extract in 100 ml water) and autoclave 115°C for 10 min) to ensure sufficient vitamins and nitrogen sources.
(iv) Set up a test series as shown below to register the presence or absence of Group 1 organisms

	Additions			
	None	5% Ethanol	1% acetic acid	2% acetic acid
Zygosaccharomyces bailii group	+	+	+	+
Saccharomyces cerevisiae	+	+	−	−
Atypical *Saccharomyces cerevisiae*	+	+	−	−
Schizosaccharomyces pombe (fission cells)	+	+	+	+

11.7.3 Environmental samples

The main sites to be inspected for dominant microflora include

- drains
- raw material store
- near filler
- soap lubrication from jets
- electric motor fins (all areas)
- bottle tracks
- ultraviolet insectocutors
- soak baths
- capping systems
- swabs from brushes and squeegees.

Action points:

(i) Swabs may be smeared across a series of agar plates to give evidence for activity, technology and identity simultaneously (see example given below).
(ii) Swabs must be returned to their cases and an appropriate liquid medium (e.g. YM ± modification) or product water mixture added. The samples are then observed for fermentation.
(iii) Liquid and debris samples can be streaked out on agar plates (use swab) and an enrichment culture of YM + 20% (w/w) used to select yeasts.

Example: suggested solid (agar) medium pattern for soft drink and related beverages.

YM (YM1) YM + 1% acetic acid (add acid just before pouring)
(YM20) YM + 20% (w/w) glucose
(YM40) YM + 40% (w/w) glucose
(YM60) YM + 60% (w/w) glucose
(ACT) actidione agar (Oxiod)

Characteristics of strains of spoilage yeasts

	YM	YM1	YM20	YM40	YM60	ACT
Zygosaccharomyces bailii	+	+	+	+	+	−
Saccharomyces cerevisiae	+	±	+	±	±	−
Pichia anomala	+	−	+	+	−	−
Torulaspora delbrueckii	+	−	+	±	−	−
Hygiene yeasts						
Aureobasidium pullulans	+	−	±	−	−	+
Rhodotorula clutinis	+	−	+	−	−	+
Geotrichum candidum	+	+	+	−	−	+

+ = growth; − = no growth.

11.8 Conclusions

This chapter has set out one approach to conventional microbiological testing and identification of organisms within the soft drinks industry. A choice can be made, therefore, between classical testing regimes, which are time-consuming and expensive, and a simple programme that gives evidence in the form of an assessment in the areas of activity/no activity, technology significance and tentative identity.

The time-scale for this approach ranges from a few minutes (sample data plus direct microscopy) up to a maximum of 5–7 days, without the need for obtaining pure cultures. Sample sizes can be large or small. With conventional testing, which includes most high-technology microbiological methods, a pure culture (which takes about 3 weeks) has to be used to ensure that only the characteristics of the individual organism are determined. The conventional approach presumes that once an organism is identified other conclusions are readily obtained. It has, therefore, been seen as an essential part of the problem-solving process.

In identifying dominant important organisms for the soft drinks industry, there are many pitfalls for the inexperienced yeast microbiologist. Recently, there have been three major investigations of beverage and food problems that have cost the industry millions of pounds in lost product and raw materials. These losses were due to inexperience in the identification of micro-organisms and making appropriate assessment and recommendations. Other limitations of classical or conventional methods are time (e.g. up to 3 months required for yeast identifications) and the expense of a programme involving some 80–90 individual tests and observations per organism.

For routine monitoring of raw materials, packaged product and processing and packaging environments, a simple forensic microbiological programme is, with appropriate training, straightforward to set up. The system works towards prevention and prediction at an economic cost. At any point the system is flexible enough to add or subtract any other simple method and, if necessary, to introduce more conventional programme test(s).

Appendix 11.1 Conclusions from environmental audit inspections

Table 11A.1 Examples of management indicators for conclusions of environmental audits

Geotrichum candidium *Trichosporon* spp. Acetic acid bacteria Common moulds	Unsatisfactory
Pichia anomala *Pichia membranaefaciens* Acetic acid bacteria	Cleaning programme not efficient
Aureobasidium pullulans *Rhodotorula glutinis* *Debaryomyces hansenii* *Cryptococcus* spp.	Excessive dust-like particles

The factory hygiene status in the right hand column of Table 11A.1 predicts, in the left hand column, the most likely microflora to be found. Conversely, a predication of factory status can be made if the corresponding microflora are present.

Appendix 11.2 Examples of environmental microbial indicators

During environmental audit inspections residues may be observed. The predicted dominant micro-organisms are those in the left-hand column of Table 11A.2.

Table 11A.2 Examples of environmental indicators

Zygosaccharomyces spp. *Saccharomyces cerevisiae* strains *Schizosaccharomyces pombe*	Residues Fruit concentrates Sugars syrups
Kloechera apiculata *Metchnikowia pulcherrima*	Fresh fruit and juices
Saccharomycopsis lipolytica *Kluyveromyces* spp.	Oil and/or dairy products

Appendix 11.3 Simple recognition and identification of Group 1–4 micro-organisms

This brief outline model demonstrates how simple systems can be designed to give a practical application for micro-organism recognition and identification.

Observations	**Assessment of microorganisms**
Fermenting soft drink preserved with benzoic acid	1. Predicted spoilage yeasts *Zygosaccharomyces bailii* (Zb) *Z. bisporus* (Zbi) *Z. rouxii* (Zr) Atypical *Saccharomyces cerevisiae* (ASc) S. *cerevisiae* (Sc) *Torulaspora delbrueckii* (Td) *Schizosaccharomyces pombe* (Sz)

Direct microscopy

Fission cells	= *Schizosaccharomyces pombe*
Budding cells	2. Set up split bottles test with 80% sterilised product and 20% distilled water (v/v) as follows:

Split bottle test separation of yeasts

	Zb	Zbi	Zr	ASc	Sc	Td
2.1 Product + water only	+	+	+	+	+	+
2.2 Product + water + 5% (v/v) ethanol	+	+	+	+	+	+
2.3 Product + water + 1% (v/v) acetic acid	+	+	–	+	–	–
2.4 Product + water + 2% (v/v) acetic acid	+	–	–	–	–	–

NB: Zb, Zbi and ASc are very similar in behavioural traits, and therefore collectively can be called the ‘bailii group’ since assessments and subsequent action would be the same for one or more members present.

3. Confirmation test

Growth on solid medium/microscopy

	Zb*	Zbi*	ASc**
3.1 YM agar			
3.2 YM agar + 1% (v/v) acetic acid	+	+	+
3.3 YM agar + 2% (v/v) acetic acid	+	–	–

Microscopy

*Spores with conjugation tubes
**Spores without conjugation tubes

Observations	**Assessment of micro-organisms**
Swabs from factory environment can be immersed in the product/water mixture as 2.1–2.4	Assess as for products, i.e. absence/presence of one or more Group 1 organisms

Note any fermentation

Surface growth (colonies on plates)	**Microscopy (cell-shapes)**	**Yeasts (yeast-like fungi)**
Red/pink	Oval-round budding cells	*Rhodotorula glutinis*
Very rough, almost mould-like. Wrinkled, often tough cream-brown	Threads with cross-walls. Large splitting cells. Budding cells	*Trichosporon* spp.
White/grey, dusty	Box shapes with cross-walls. Some threads with cross-walls	*Geotrichum candidum*
Rough brain/cauliflower-like, white/dirty cream sometimes colony top chocolate brown *or* smooth creamy pearl shape	Mixture of cell shapes and sizes (budding). Hat-shaped ascospores	*Pichia anomala*
	Short fat cylindrical cells. Short chains of budding cells	*P.memberanaefaciens*
Smooth white/cream/brownish conical shape	Circular, often with ring of buds on mother cell surface	*Debaryomyces hansenii*
White/cream/brownish wrinkled/striated to rough	Oval and short cylindrical	*Kluyveromyces marxianus*
Rough, small, often slow growing colonies. Distinctive smell – acidic/wine/mouse	Mixture of cell shapes and sizes. Some pointed at one end (ogive)	*Brettanomyces anomalus*

Recognition/identification separation tests for yeasts of Group 2–4 micro-organisms

	Observations/Tests					
Yeasts	Colony	Fe	Fi	Cells	44°C	Acid Pr
Pichia anomala	S/R	+	+	M	–	–
P. membranaefaciens	R	–	+	M	–	–
Debaryomyces hansenii	S	–	–	C	–	–
Kluyveromyces marxianus	S	+	–	M	+	–
Brettanomyces anomalus	S/R	+	+	M	–	++

Code S = Smooth colonies
R = Rough colonies
Fe = Fermentation in presence of 5% (v/v) ethanol:product or medium
Fi = Film formation

Cells M = Mixture of shapes
C = Circular/short oval shape
Acid Pr = Acid production: confirm with YM or WA or ME agar +0.5% calcium carbonate

Recognition of indicator organisms not included in the foregoing table

	Recognition/identification				
Yeasts	Act	Col	Fe	Cells	Fat splitting
Aureobasidium pullulans	+	F−	L−	M*	+weak
Kloechera apiculata	+	S	+	L	−
Metchnikowia pulcherrima	+	S(Pi)	+	C/O	−
Saccharycopsis lipoltica	+	R	−	M	+++ strong
Cryptococcus spp. (**not** pink forms)	+	Mu	−	C	± weak

Code
- Act = Growth on acitidione agar
- Col = Colony type
- F = Flat
- Fe = Fermentation
- S = Smooth colonies
- S(Pi) = Red/purple pigment, if enough iron and biotin are present
- L = Lemon-shaped
- C = Circular
- O = Oval
- R = Rough
- M* = Mixture of large budding cells

Moulds – various
Woolly, hair, dusty mat-like – colours and textures vary according to mould type. Common examples on

Apple	(Blue)	*Penicillium expansum*
Oranges	(Blue/green)	*P. italicum*
Onions	(Black)	*Aspergillus niger*

Common bacteria

Colonies	**Microscopy (cells)**	**Bacteria**
Transparent/watery buff/fawn/smooth	Mixture of rod shapes	*Acetomonas* spp.
Small/cream/brown Smooth to wrinkled	Predominantly small fat rods	*Acetomonas* sp.

Appendix 11.4 Media for cultivation of industrial micro-organisms[a]

Bacteria	**Media** (Oxoid or Difco or other commercial brands)
Acetic acid bacteria } Lactic acid bacteria }	Actidione agar (PM1 118); also certain yeasts can be detected (Difco–WLD–B425) Wort agar (CM247) Malt extract agar (CM59) Tomato juice agar (CM133) Rogosa agar (PM221) MRS agar (CM361)
Other bacteria[b]	Plate count agar (CM 183) Nutrient agar (CM3)

Yeasts and fungi	Actidione agar (PM118) (e.g. *Kloeckera and Brettanomyces* spp.) contains 10 ppm actidione (cycloheximide)	
	WLD (Difco–B425) contains 4 ppm Actidione (cycloheximide)	
	Wort agar (CM 247)[c]	
	Malt extract agar (CM 59)[c]	
	Corn meal agar (CM 103)	for mycelium formation
	Potato dextrose agar (CM 139)	
	WL agar (Difco–B424) (CM309)	
	Buffered yeast agar (CM 153)	

Storage medium for yeast cultures (MYGP/YM medium)

(See Wickerham, 1951)

Malt extract (powdered)	3 g
Yeast extract (powdered)	3 g
Glucose	10 g
Peptone (mycological)	5 g
Water	1000 ml

for solid medium (20 g) is added

YM agar (Difco–0712)
YM broth (Difco–0711)
Antimicrobial supplement to restrict bacteria, some moulds thus giving maximum chance for soft drink yeast – use 20% (w/w) glucose in solid and liquid YM.

[a] Based on data in Davenport (1980).
[b] For assessment of wide range of microbes (many yeasts and moulds grown on these).
[c] pH 4.5 or 3.5 (use of concentrated HCL two drops per 100 ml before pouring plates can help with selection of yeasts of particular industrial significance in some products).

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12 Functional drinks containing herbal extracts

J. Whitehead

12.1 Introduction

A functional drink can be defined as one that offers the consumer additional perceived benefits besides its primary function, which is hydration – the maintenance of body fluid at a suitable level. The benefits are usually directed towards some aspect of maintaining good health or coping with the pressures of modern lifestyles. A range of functional ingredients, including herbal extracts, is available to the formulator of functional drinks.

Herbal extracts are often used by beverage product developers as a matter of course, without necessarily fully appreciating the differences between extracts and other well-defined and easily specified ingredients such as acidulants, preservatives, stabilisers and sweeteners. This chapter will examine these differences so that formulators using herbal extracts will be aware of the particular characteristics of natural ingredients. They should then be able to make the best use of them in trying to fulfil the expectations of a marketing department providing a new product brief. Brief descriptions of the more commonly used herbs, together with their traditional benefits, are included at the end of the chapter. However, to understand today's herbal drinks, it is worth looking first at their history, how the present market has developed and briefly at the role played by extracts in the herbal drinks.

12.1.1 History

The date of the first herbal drink is open to debate. Certainly ginseng was recognised as a *qi* tonic herb about 5000 years ago (Bown, 2003) and one of the traditional ways of using ginseng was to make a tea from its dried root. It is mentioned in Shen Nong's *Cannon of Herbs*, which, although not completed until about AD 250, was founded on the work of Shen Nong, a Chinese Emperor believed to have reigned around 3000 BC. Tea (*Camellia sinensis*) has been drunk in China for about 3000 years (Bown, 2003) for the stimulating effects of its caffeine content. Later, liqueurs, although not soft drinks, were made from herbs by monks for medicinal and tonic purposes. Coffee and chocolate are nowadays simply considered popular because of their tastes, but when they were first discovered they were seen as tonic drinks with various stimulant properties including aphrodisiac effects. Even the world's most popular soft drink started life promoted as a nerve tonic (Pendergrast, 1993) and in 1899, although it was mainly promoted as a delicious and refreshing drink, it was still also being advertised as relieving physical and mental exhaustion, curing headaches and benefiting brainworkers. Other adverts talked about the use of the coca leaf by Andeans and the kola nut by Africans. In the mid-1980s a new era of herbal drinks emerged accompanied by a message of sophisticated healthiness just as the drink–drive laws began to take effect in many countries and lifestyle became a significant issue in affluent societies.

12.1.2 Market development over the past 15 years

The first generation of modern-day herbal wellness drinks arguably emerged in the latter half of the 1980s with the UK launch of Aqua Libra. The product proposition for such drinks was one of general healthiness without defining specific benefits except for the early leader, which carried the line 'restores alkaline balance' on the label. The product propositions for subsequent generations of herbal drinks became more focused as societal awareness of, and concern for, lifestyle-related health problems grew. Functional drinks, including those containing herbs, now address such issues as ageing, stress and stimulation in its various forms, whether for simple energy or for partying, or for strengthening the immune system and calming.

Initially, these drinks were flavoured with fruit juice and natural fruit flavours and were often pasteurised in the bottle to avoid including preservatives in the ingredients list. Herbal extracts have more recently been put into dilutables, juice-based drinks and, increasingly, bottled waters. A few herbal drinks are coming on to the market with organic certification.

Herbal drinks form part of the functional drinks sector. Some idea of the growing importance of this sector can be gained from the *International Functional Soft Drinks Report 2003* (Zenith, 2003), which covers the United States, Japan and 16 Western European countries. The market has become so sophisticated that the report can define four subsegments:

- Sports drinks are the largest segment in the markets covered by this report.
- Enriched beverages, which are generally juice-based drinks but increasingly also bottled waters, are the predominant segment in Europe.
- Nutraceuticals are well established in Japan and growing quickly in the United States, but are the newest segment in Europe.
- Energy drinks have a firm base in Japan and increasingly so in Europe. They have a smaller share of the market in the United States.

Consumption in 2002 grew in these markets by 11% to over 12 billion litres. Functional soft drinks now account for 6% of all soft drinks consumption by volume in these markets, up from just 4% in 1998 (see Figures 12.1 and 12.2). It follows that herbal extracts have a role to play in this increasingly important soft drinks sector, which can no longer be seen as an insignificant niche market.

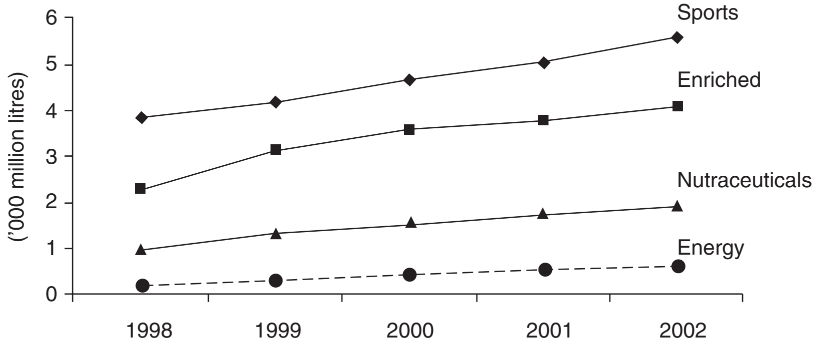


Figure 12.1 International functional soft drinks market 1998–2002: United States, Western Europe and Japan.
Source: Zenith International.

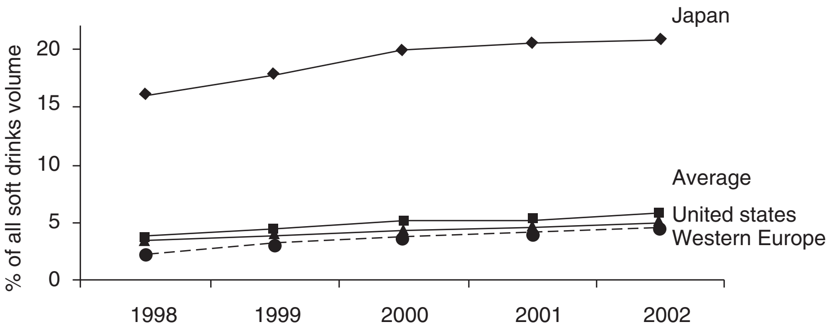


Figure 12.2 International functional soft drinks share 1998–2002.
Source: Zenith International.

12.1.3 The role of herbal extracts in beverages

The role of herbal extracts is primarily a marketing issue rather than a technical one. Part of the reason for using herbal extracts in a drink is that the herbs give the consumer a perceived benefit and therefore a reason to buy the product. To be effective in this role, it is important that the herb is recognised by the consumer. It therefore follows that, when developing a new herbal drink, the herb, besides being compatible with the product proposition, should be chosen for its recognition among the target consumers. The rule is generally that specialist herbs can be used in niche products but that for the mainstream, wider market, the choice of herbs should be limited to those more familiar.

12.2 Overview of the extraction process

This section starts by reviewing the heritage of extracts before proceeding to look at the stages of a basic extraction process, focusing on what makes extracts different from single-component ingredients. The section also considers the parameters that can affect the resultant extract.

12.2.1 Extraction heritage

Most people probably regard extracts as having their origins in medicinal usage, and it is from this tradition that some of the more established extract manufacturers have evolved. Familiarity with the descriptions of various traditional extracts may help to avoid misunderstandings when contacting extract manufacturers.

12.2.1.1 Infusion

'Infusion' is a general term for extracts produced by steeping a herbal raw material in a liquid. Traditionally, infusions were made by pouring boiling water on to the dried herb, leaving it to soak and then and then straining off the resultant liquor. Modern-day infusions for beverage applications are often carried out at ambient temperature but using some alcohol in the liquor. Because infusion is a very general term, there is no set value for the plant/extract ratio, but in practice 1 part herb yields from 4 to 10 parts infusion. The practical maximum strength is reached when the extracting liquid just covers the plant material being extracted. This maximum depends on the bulk density of the dried herb, but in general is reached with 1 part herb yielding about 2.5 parts extract. Traditionally, most extracts were made from dried herbs, as these were easier to keep in good condition from one crop to the next. If it is necessary to use fresh herbs for extraction, the increased water content, which can be up to 90% with leafy herbs, has to be allowed for. Thus, if fresh herbs with only about 10% dryweight are used in the same proportion as dried herbs, a much weaker extract is obtained.

12.2.1.2 Decoction

Decoction is a variation on the general infusion process in that the herb is steeped in boiling water to which heat is then supplied to keep it at boiling point, simmering gently rather than boiling vigorously.

12.2.1.3 Tincture

Traditionally a tincture is an ambient temperature extract that is made with a high alcohol level in the extraction liquid, typically 60–70% or more. The herb/extract ratio is usually 1 part dried herb to 10 parts tincture, although a few tinctures were specified at stronger ratios (i.e. more herb) in the British Pharmaceutical Codex and British Pharmacopoeia.

12.2.1.4 Liquid or fluid extract

The terms 'liquid extract' and 'fluid extract' are interchangeable. The extract is traditionally a 1 : 1 yield made by extracting the herb with a succession of fresh batches of extracting liquid, combining these batches and concentrating them to the original weight of the herb. This was done so that liquid extracts could replace dried herbs on a weight-for-weight basis in medicinal formulations prescribed by herbalists. A liquid extract usually contains 20% alcohol, which acts as a preservative, although some are specified at higher alcohol strengths to produce the optimum extraction from the point of view of the actives content. Owing to the heating involved in the concentration stage, a liquid extract is often dark brown in colour with a caramelised odour.

12.2.1.5 Soft extract

A soft extract is produced initially as a liquid extract but the concentration stage is continued until the resultant extract has the moisture content of 30% or less. The extract has the texture of a thick paste or viscous syrup and is usually dark brown in colour and often caramelised in flavour.

12.2.1.6 Powdered extract

A powdered extract is made by replacing the moisture in a soft extract with an equal amount of a substrate such as calcium phosphate, starch or maltodextrin. The moisture is normally removed using a vacuum oven to avoid excessive thermal degradation of the extract.

12.2.1.7 Percolation

Percolation is an alternative to infusion processes whereby the solvent is trickle-fed on to the top of the herb or spice material and collected as it emerges from the bottom of the percolation vessel, which is traditionally conical in shape. This process is sometimes used in the production of natural flavours.

12.2.1.8 Modern extracts

The flavour and modern phytopharmaceutical industries have made big changes to the traditional pharmaceutical extraction processes. Whereas ethanol was really the only significant solvent apart from water used by the traditional pharmaceutical extractors, solvents such as hexane and acetone have been used by flavour companies to make soft-extract oleoresins for natural flavour components. Sub- and supercritical carbon dioxide and also some fluorohydrocarbons are now used to produce some very high-quality extracts. Modern concentration and drying processes such as reverse osmosis, spray-drying and freeze-drying can yield extracts with less colour and caramelisation problems, but this benefit does come with a price penalty.

12.2.2 The extraction operation

Rather than an in-depth technical description of the mechanics of extraction, this section presents briefly a typical infusion process, focusing on the factors that make extracts different from single-chemical components. Extracts by their nature are complex mixtures of (often) diverse active compounds contained within a plant matrix which are brought into solution by the extraction process. The aim of the extractor is to produce, over a period of time, batches of an extract meeting a customer's individual specification with as little variation as possible. There are parameters over which the extractor has some control, and these can be used to help achieve product consistency and also to fine-tune an extract to a particular customer's needs.

12.2.2.1 Raw materials

The process starts with the herb, which itself starts as a growing plant. Here there are many variable factors, including climate, terrain and husbandry. The herbs that are most in demand usually come from cultivated sources, which bring some control to the growing environment by way of irrigation, fertilisation and possibly shelter. However, on the downside, cultivated herbs are more likely to be treated with pesticides or herbicides. Wild herbs which are gathered directly from their natural growing habitat are less likely to have been treated with chemicals but are subject to the full vagaries of the weather. They are also more likely to be subject to post-harvest adulteration and substitution. Both sources of herbs are also subject to the effects of varying soil type. A comparison can be made here with wine. The same variety of grape can produce quite different wines depending on both the weather from season to season and the different soil conditions in which it is grown.

The extract manufacturer tries to exercise as much control as possible over the raw material, but this is not easy. Sourcing from a single wholesaler who can be exhorted to source from a single merchant in a specified growing region, who in turn is urged to buy from a single locality, is not really feasible unless the project is a large one with sufficient funding for the administrative efforts and the travel necessary to set up a controlled supply line. Even if this were possible, the weather will still vary from year to year and affect levels and proportions of actives.

The maturity of the plant at harvest can also have a bearing on the actives content of the raw material. If it is possible to control the supply line, then it may be possible to control the harvesting of the crop at a specified maturity; otherwise the crop will probably be harvested by the local people to optimise the yield of bulk crop for best financial return.

Some control can be built in by specifying minimum contents of actives, but this standard is prone to downward revision for reasons of commercial expediency in times when the harvest yields levels of actives that are mainly below the specification. At such times, it is likely that parcels of the herb meeting a particular specification will carry a price premium.

If the herb selected for a given development project is one for which market demand is insufficient to warrant a commercial growing operation, it will probably come from wild sources and will be a spot market purchase subject to variability of price and availability. Probably the best that can be done here is to source from a single wholesaler.

Another option is available when the project is of sufficient size: to set up a contract for the growing of a suitable amount of the desired herb. This contract can be either directly with the grower if conditions and resources allow or through a herbal wholesaler with good contacts in a region where the required herb grows well. This will remove some of the supply variables and should enable a certain degree of control over the husbandry and harvesting of the crop. The difficulty comes at the start of a new project, when it is usually very difficult to predict the volume of sales of a new product and hence the size of the herb crop needed.

12.2.2.1.1 Availability and price. Raw material prices affect the price of an extract. The normal rule of larger volumes attracting a bulk discount does not necessarily hold true when it comes to purchasing herbs. Supply and demand are the governing factors. When a herb is selected for a new project it is advisable to ascertain from the extractor that it is readily available in the quantities needed for the forecast sales volumes. If the anticipated annual demand for the selected herb represents a significant proportion of the prevailing harvest quantity, then the price is likely to be increased by the supply chain until subsequent harvests are increased to meet the greater demand.

12.2.2.1.2 Transit and storage. It is important to consider the condition for transit and storage conditions from field to factory and storage, which can also have an effect on the raw material quality. The first step in the handling process for most herbal materials is the drying process. In the less developed nations, where a number of the herbs used in functional drinks are grown or collected from the wild, this usually consists of spreading the crop out on the ground to dry in the sun, which is a very good, cheap way of drying the herb gently. The problem arises when the drying area is just the dusty ground that is shared with domestic and farmyard animals. This is one of the reasons why some dried herb deliveries have a very high microbiological assay. In cooler growing regions sun-drying is not an option and air-drying is used. The danger with this method is that, in the interests of efficient usage of expensive drying machinery, a higher temperature than ideal may be is used to speed up the process. This can lead to excessive loss of volatiles or damage to thermally labile actives.

Poorly stored crops may be damaged by insects or other pests, such as rodents. Water damage leading to mould growth can be due either to poor or unprotected storage or insufficient drying. Other problems that can occur are cross-contamination, accidental or deliberate adulteration for commercial benefit and mislabelling. The extractor needs to be aware of these potential hazards and exert appropriate control measures to prevent damaged material from entering the extraction process.

12.2.2.1.3 Summary. It is obvious from the foregoing paragraphs that there is inherent variability in the raw material supply chain before the extraction process itself is even started. This will introduce variability into the subsequent process and influence product costs.

12.2.3 Extraction

The fine details of equipment and procedures are of interest primarily to those actually making the extracts rather than to those using them. In brief, therefore, the extraction process consists of the following stages:

- Receiving the raw material and subjecting it to acceptance examination by quality control (QC).
- A preparation stage such as milling, in order to produce a suitable particle size for extraction.
- Steeping the raw material in the chosen solvent, preferably in a closed container, which is usually made of stainless steel or a plastic capable of resisting attack by the solvent and the plant components. The quantity of solvent used needs to take into account the amount that is absorbed by the dry plant material in order to ensure the desired final yield of extract.
- Standing for a set period of time before removing the liquid from the spent herb, which may subsequently be pressed to recover as much as possible of the extract retained in it.
- Using the resulting basic crude infusion directly or further concentrating it under reduced pressure to make a soft extract, which in turn can be dried to a powdered extract. For the majority of functional drinks the infusion is the preferred form of extract as it is the easiest to dispense and mix into the end product. Figure 12.3 shows a typical infusion vessel.
- Finishing the crude infusion by filtering it, preferably through a final membrane filter of about 0.2–0.4 μm to remove as many as possible of the microbiological organisms in the extract.

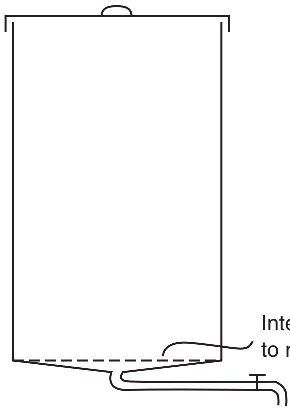


Figure 12.3 Typical infusion vessel.

- Packaging the extract, ensuring that the container used is suitable for the purpose and will not be attacked by the components of the extract, followed by storage in a cool, preferably dark place, away from excessive temperature changes and out of direct sunlight.

Within the process itself, there are several factors that can affect the quality and consistency of the final extract. These include the particle size of the raw material, the extraction time, the extraction temperature and the solvent used. The difference between these variables and those in the raw materials section is that, to a greater or lesser extent, they can be controlled by the manufacturer, so that an extract is produced as consistently as possible, using these variables to fine-tune the final extract to meet the customer's requirements. The effect that each of each of these controllable parameters has on the final extract is discussed in the following paragraphs.

12.2.3.1 Particle size

The particle size of the raw material used in an extraction process is a compromise between rapid extraction times and rapid and easy filtering of the extract when it is removed from the spent herb. The finer the raw material particles, the greater the chances are of producing the most complete extraction possible in a practicably short time, which in turn leads to consistent batch strengths (from identical batches of raw material). Conversely, coarse particles require a longer time for the soluble compounds to dissolve out into the extract, and thus increase the chances of batch-to-batch variation. Fine-particle matter blocks filter pads or membranes sooner than coarse matter, and this leads to potential wastage of extract and extended processing times. Some extract manufacturers buy their herbs in the whole or uncut form and mill them on site after they have passed the initial QC acceptance tests, which usually include tests for moisture content and soluble extractive content and microscopic examination. This helps in controlling batch-to-batch consistency of particle size. Purchase of the uncut herb by the extractor also helps to make adulterated and wrongly labelled herbs easier to detect visually. If the herb is purchased ready cut or as a powder then it is essential to specify the particle size to minimise batch-to-batch variability.

12.2.3.2 Time

Extraction is a process in which the solvent penetrates the dried herb and dissolves the soluble components of the plant material. These then diffuse out into the free solvent surrounding the plant material particles. This process eventually reaches a state of equilibrium (Theoretically, complete equilibrium is only achieved at infinite time and the approach to completion is an asymptotic curve; Figure 12.4.) In practice, about 90% of available solids come into solution within 24 hours for a typical leaf herb at ambient temperature, although harder material, such as some dried woody roots and barks, will take rather longer to achieve a satisfactory degree of extraction.

The time allotted to the extraction of a herb by a manufacturer is usually a commercial compromise between getting as close to a complete extraction as possible, which helps achieve consistency, and making effective use of costly extraction equipment and operators. To help achieve batch-to-batch

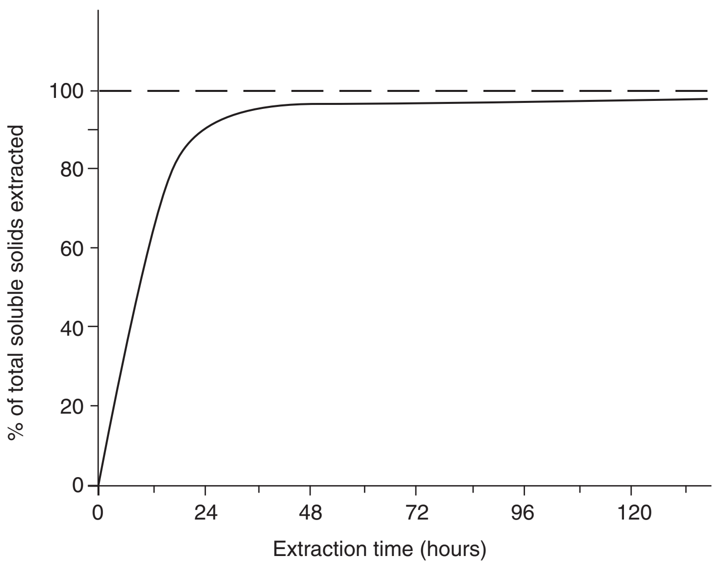


Figure 12.4 Typical extraction curve.

consistency the extraction time is usually well defined within the manufacturer's standard operating procedure. For example, an extraction should not be left standing on the herb over the weekend if a 24-h extraction period is specified. The reason for this is that different individual soluble components will diffuse out of the plant matrix at different rates, so an extract that is left to stand for longer than specified may have a higher soluble solids content, and possibly a different ratio of individual components, from those required in the standard specified extract.

It can be advantageous to specify a shorter extraction time than is required for complete extraction in cases where the active being sought from the herb is readily soluble and reaches equilibrium before undesirable components such as brown colours from chlorophyll degradates or bitter-tasting tannins are fully dissolved.

12.2.3.3 Temperature

The effect of increasing the temperature of an extraction is, first, to increase the rate at which the soluble plant materials diffuse into the solvent, consequently shortening the extraction time. This is desirable from the manufacturer's point of view. Conversely, the colder the process, the slower the diffusion rate and the longer the extraction takes, giving rise to the possibility of a weaker extract due to incomplete extraction within a specified time. It follows that the more consistency is achieved in controlling the extraction temperature, the less variation there will be between different batches of an extract (given consistent raw material, particle size and extraction time).

A second effect of a raised extraction temperature may be to increase the solubility of some of the less soluble plant components. This could lead to hazing and sedimentation since these components slowly come out of solution again on standing during storage. This may not be a problem if the extract is to be used in a cloudy drink.

12.2.3.4 Solvent

The solvent that is most commonly used in the extraction of herbs for soft drinks applications is aqueous ethanol, usually at about 20% abv (alcohol by volume). One of the reasons for this was that herbal beverage extracts were developed in the late 1980s for the (then) newly emerging 'adult soft drinks' sector. Manufactures sought to avoid any synthetic ingredients, thus enabling 'all natural ingredients' and 'preservative-free' claims to be used on product labels. A concentration of 20% ethanol in the extract liquor not only makes a very good extracting solvent but also preserves the extract effectively, if the microbiological levels have been reduced initially by filtration or other non-chemical means. Wholly aqueous extracts must be preserved, unless the extract has been

Table 12.1 Solvent comparison

	Polarity (E)	Boiling point (°C)	Latent heat evap. (cal/g)	Viscosity (cP)	
				0°C	20°C
Carbon dioxide	0.00	-56.6	42.4	0.10	0.07
Acetone	0.47	56.2	125.3	0.40	0.33
Benzene	0.32	80.1	94.3	0.91	0.65
Ethanol	0.68	78.3	204.3	1.77	1.20
Ethyl acetate	0.38	77.1	94.0	0.55	0.46
Hexane	0.00	68.7	82.0	0.40	0.33
Methanol	0.73	64.8	262.8	0.82	0.6
Dichloromethane	0.32	40.8	78.7	—	0.43
Pentane	0.00	36.2	84.0	0.29	0.24
Propan-2-ol (IPA)	0.63	82.3	167.0	—	2.43
Toluene	0.29	110.6	86.0	0.77	0.59
Water	>0.73	100.0	540.0	1.80	1.00
Propylene glycol	>0.73	187.4	170.0	—	56.00
Glycerol	>0.73	290.0	239.0	12,110.00	1,490.00

Source: Moyler, 1988.

sterilised and aseptically packed, as unpreserved extracts provide an excellent growth medium for microbiological organisms.

When choosing a herb for its specific actives, it is important that the extract of that herb is produced using a solvent that is effective in dissolving the desired actives. The properties of some solvents are listed in Table 12.1. Water is an extremely polar solvent and dissolves polar materials such as salts and sugars. Ethanol, propylene glycol and glycerine are less polar, although any water present will tend to dominate the solvent polarity unless its proportion is relatively small. Even less polar solvents such as acetone or hexane dissolve water-insoluble non-polar components such as oils, fats and resins. Given that, in the main, plants function with water as their carrier medium, most plant actives within the plant cell structure are soluble in water to some extent. Likewise, the human body has a water-based system and needs the actives to be water soluble to some degree or else rendered so by the digestion process.

Using solvents at the non-polar end of the spectrum offers a means of selectively extracting specific actives. An example from the nutraceutical sector is the selective extraction of ginkgo biloba with a high level of acetone in an acetone/water solvent mixture, which produces a 50 : 1 extract (50 kg of dried herb produces 1 kg of dried extract) standardised on 24% flavone glycosides and 6% terpenes. Sub- and supercritical forms of carbon dioxide produce very-fine quality extracts, and here again there is the possibility of using the difference in polarity between the sub- and supercritical states to assist in selective extraction.

To overcome the fact that a single solvent rarely completely extracts all the potentially available components from a herb, multi-solvent extracts have been developed. In this process, initial extraction of the herb using a non-polar solvent is followed by extraction using a more polar solvent. The two extracts are then concentrated to remove the solvents. Finally, they are combined using a suitable emulsifier system to hold the two incompatible components together without separating. This will render the extract water soluble (or at the least miscible) without settling-out in the final product.

12.2.3.5 pH

In some instances, where the desired actives to be extracted are of a basic nature (as opposed to acidic), the extraction of those actives can be selectively enhanced by adjusting the pH of the extraction solvent with a suitable organic acid such as citric acid.

12.2.3.6 Summary

These paragraphs suggest the issues that the extract manufacturer faces in production. Given the many different factors, both in nature and within the extraction process itself, that produce variability, it is perhaps a tribute to the extract manufacturers' combination of technology and art built up over a long time that extracts as sold to customers are as consistent as they are.

12.2.4 Organic extracts

The interest of consumers in the quality of what they eat and drink is increasing, and this has led to the growing use of organic products. The organic designation certifies that no chemical pesticides or herbicides, which can have an adverse impact on the environment, have been used in the growing of these foodstuffs. It is possible to produce a range of extracts that can be certified as organic. First the herb to be extracted must be certified as organic, but this is not usually a problem as most of the herbs appropriate to functional drinks are available from herb suppliers that specialise in offering organically certified herbs. In fact, all the herbs listed in Section 12.5 appear in the list of a single supplier of organic herbs. The problem arises with the extraction process. Any solvents used other than water need to be certified as organic. Currently, organic ethanol is available and this is currently the best option for beverage extracts unless alcohol is not acceptable in the product, for example Islamic markets. If the product is intended for Kosher markets then the organic alcohol will need to be from organic grain as grape alcohol could cause a problem with Kosher certification. An alternative option at this point in time is to use aqueous extracts containing preservatives, where permitted by a local organic certifying body. In the

United Kingdom, the Soil Association allows the use of the preservatives sodium benzoate and potassium sorbate.

It is theoretically possible to produce organic glycerine by steam-splitting organic vegetable oils; however, the author is unaware of any current source of this material. It should also be possible to produce a low-polarity organic solvent from natural organic raw material stock by physical means, which in turn should enable the production of organic concentrated soft extracts reasonably similar to those currently produced using acetone and hexane.

12.2.5 Extract costs

The cost of extracts is not necessarily at the forefront of considerations when developing new products, but it is useful to have a comparative understanding of the issue. There are fixed and variable costs involved in the extraction process. In general terms, the raw material handling costs, plant overheads and technical service costs such as QC are relatively independent of batch size. The main costs that vary with batch size are the raw materials, energy consumption, labour, packaging and delivery. The basic infusion process using readily available herbs, with enough volume demand to enable batch sizes of 250 kg or more finished extract, will result in an economically priced extract. Smaller one-off batches will carry a premium. The higher the raw material price (Section 12.2.2.1.1), the bigger the percentage of the overall extractions cost it respresents, and consequently the lower the influence on costs savings made by larger batch sizes.

The yield of the extract is an important factor in the price of concentrated extracts. Low-yield extracts, which have a high concentration of actives in them, will usually carry much higher prices. First, the amount of raw material required to produce a given weight of extract is much higher than for a simple infusion. Second, the process itself may be more complex, involving greater costs for solvents and concentration energy. The equipment used may also be complex and attract significantly higher production overheads.

Certified organic extracts often carry a significant raw material price premium over their non-organic counterparts. Sub- and supercritical carbon dioxide extracts are in general the most expensive because the high-pressure equipment used is very costly to purchase.

12.3 Extract characteristics and their problems

12.3.1 Specifications

An understanding of the factors that make extracts different from single-component ingredients can help to avoid frustration during use. Extracts are often complex mixtures of components, and their specifications demonstrate this. They do not, for example, have straightforward melting points or meaningful boiling points. They do not have simple spectra or other readily quantifiable properties. But they do have subjective parameters such as aroma, taste and colour. The solvent content may be specified, and dry matter also, although this may not very be high in infusions. Should glycerine be present in an infusion or soft extract, then it is not easy to determine dry solid matter content. Some concentrated extracts may carry an assay of one or more active components, for example, ginsenosides in a ginseng extract, but this still does not necessarily give a good assessment of the quality of the whole extract. For example, in the case of ginseng, the ginsenosides tend to be preferentially located in the hair roots and rootlets rather than the main roots, so that extracts with higher levels of ginsenosides can be made from these rather than the more expensive whole roots that contain other components of the herb.

12.3.2 Stability

Liquid extracts such as infusions will often, over time, produce a fine sediment. There may be a statement to this effect in the specification and on the container label. If the product for which the extract is destined is a cloudy drink or opaque like a fruit juice, then the container can be shaken each time to re-disperse the sediment before weighing out. If it is essential for the extract to be clear, then it must be carefully decanted when weighing out for a production batch. Extracts in storage will often change colour over time. Thus, specifying a colour parameter without a time factor can lead to the situation where a customer's QC accepts the extract on delivery, as it meets colour standards at the time, but rejects it on retesting later after it has spent some time in the raw materials store – particularly if the drum has been opened and is part used.

12.3.3 Hazing

Another consideration is whether the extract is stable within the finished product. If the product containing the extract is a clouded beverage, then there may not be a problem. If, however, the end product is clear and is packed in a clear, see-through container then hazing can detract from its appearance. The hazing may be due to a difference of pH between the extract and the product: typically, an infusion will have an ambient pH of about 5.5–6.5, whereas soft drinks are typically around pH 3.0. If haze development due to pH difference is an issue, then the extract can be pre-conditioned to the pH of the finished product so that any proteins, or other components that are precipitated by the stronger acid conditions, can be brought out of solution and allowed to settle before the extract is given its final filter. This way the unstable components are removed by the extractor rather than ending up in the finished product and causing a haze. Another cause of hazing can be significantly differing polarities of the extract solvent and the product matrix. Polarity mismatch can occur, for example, when a high-alcohol extract is used to achieve a specific active level from a herb and is then incorporated into a primarily aqueous soft drink. Resins and other non-polar components such as fixed oils, which can be soluble in high-strength alcohol, can come out of solution on mixing with water-based systems, again giving rise to a haze. Seeds and barks are the main materials containing non-polar components. Kola is a good example of this. Traditionally, it was extracted using 60% ethanol, which dissolved a significant amount of resinous material. This extract needed to be stabilised with glycerine to keep the resins in suspension when the ethanol was removed during concentration and during subsequent dilution in drinks and other preparations.

12.3.4 Availability

The large number of herbs that are available for customers, combined with the relatively low volumes required, means that it may not be economically viable for an extract manufacturer to carry production volumes of large numbers of extracts on the off-chance that someone will want one or two of them before their shelf-live has expired. The usual practice is for extract manufacturers to carry a fairly wide range of dried herbs in sample quantities so that when a customer asks for a sample it can be produced within a reasonably short time. Once a new herbal drink has been developed using samples and perhaps a pilot batch, a production-size batch of extract will be made for the product launch. After that, if the product sells and there is a demonstrable demand pattern, it is possible that the extract manufacturer will agree to make a batch for stock to be called off by the drink manufacturer.

12.4 Extracts and their applications

This section looks at the applications appropriate to the various types of extract available and some of the factors related to the incorporation of extracts into beverages.

12.4.1 Infusions

Infusions made by a local extract manufacturer are normally the extract of first choice for ready-to-drink beverages. As described in Section 12.2.1.1, infusions are the simplest types of herbal extracts and the first stage of most other extracts. Since they are the least processed, they are also therefore the most economical to use for beverage applications. Unless there are strong economic reasons to use a more concentrated extract, it does not make sense to take an infusion, concentrate it at extra cost, possibly damage it in the process and then return it to a dilute form when the finished product is manufactured.

Concentrated infusions are sometimes economically viable if they are off-the-shelf products. For instance, because of the high demand for it, liquid guarana extracts are available that contain standardised amounts of caffeine. The demand for these is enough to warrant large-scale production in the country of origin. At this point concentration becomes desirable because the extract is being shipped over long distances and transporting solvent is a costly business.

12.4.2 Soft Extracts

The main application for a soft extract in a soft drink product is when the product label claims a relatively high level of a plant active in its formulation. The extract needs to contain a specified amount of assayed active in it to guarantee that the product label claim is met. The technical challenge is to produce a formulation that can mask both the extract's colour and its taste. A soft extract may be used by someone blending the ingredients of a flavour system for spray- or freeze-drying that was required to contain herbal ingredients. An infusion would have too high a fluid content for this purpose.

12.4.3 Dry Extracts

Extracts dried on to a water-soluble base are useful in the preparation of powdered drinks. Soft extracts tend to contain about 70% solid matter and can be mixed into a slurry with the substrate and spray-dried or dried in a vacuum oven. The substrate is usually essential to prevent the dried extract from reabsorbing moisture and turning back into a hard or sticky mass. The dried extracts can be dry-blended with other ingredients in a powdered drink formulation.

12.4.4 Incorporation of extracts in beverages

In most applications, the extract is incorporated at the premix or syrup stage with other critical ingredients such as flavourings, colours, high-intensity sweeteners and preservatives, where these are used.

12.4.4.1 Fruit-juice-based and fruit-flavoured drinks

Herbal infusions have two features that do not facilitate their use in soft drinks. They have their own flavour, which is not usually pleasant and supportive of the desired product flavour, and they usually come with a fairly strong brownish colour. Fortunately, the levels at which infusions are normally added to drinks are low and thus their flavour contribution does not often cause a problem. Likewise, the colour contribution does not interfere with the majority of soft drinks as the light straw-brown colour generated by a diluted extract is usually covered by the oranges, yellows and reds of most products. Most herbs are appropriate to this category of drinks, and a theme that is emerging is the use of flower extracts, which convey a general image of naturalness and wholesomeness without having an overtly functional message.

12.4.4.2 Mineral-water-based drinks

Mineral water with added functional ingredients is currently one of the faster growing categories within the functional drinks sector. These products generally have a more focused functional proposition, aimed at commonly recognised lifestyle issues such as coping with stress, boosting the immune system, relaxing after a stressful day at work, calming the mind for a good night's sleep, and reviving tired minds and bodies. These products tend to contain a cocktail of not only herbs but also vitamins and minerals appropriate to the product proposition. As examples, ginseng (*Panax ginseng*) is well recognised as helping the body cope with stress, echinacea (*Echinacea purpurea*) is widely seen as an immune-system-boosting herb and chamomile (*Chamomilla recutita*), passionflower (*Passiflora incarnata*) and valerian (*Valeriana officinalis*) are all well-known herbs that calm the mind and prepare the body naturally for sleep.

12.4.4.3 Energy drinks

Nearly all energy drinks have one thing in common other than their calorific value: a high caffeine content. But not all products in this category contain herbal extracts; indeed, brand leaders in both Europe and Japan do not. This section focuses on those that contain herbal extracts. Herbal extracts used in these drinks are generally from stimulant herbs of one kind or another. Most commonly used is guarana (*Paullinia cupana*), which is a natural source of caffeine and is native to Brazil. Other herbs that contain similar amounts of caffeine are kola nut (*Cola acuminata*, *C. itida* and *C. vera*), coffee (*Coffea arabica* and *C. cannephora* – robusta coffee), tea (*Camellia sinensis*) and maté (*Ilex paraguariensis*). The cocoa bean, which is the seed of *Theobroma cacao* and which is in the same family of plants as the kola tree, contains the stimulants theophylline and theobromine, which are similar in structure to caffeine.

Extracts of coffee bean and cocoa bean have been produced experimentally that contained about 3% of caffeine and 3% theobromine respectively without the disadvantage of flavour concentration typically associated with those raw materials. These are potentially alternative natural sources of effective stimulants to guarana and kola. Other supplementary herbs that have a place in energy drinks are ones that support the concept of exercise or vitality, such as ginseng or Siberian ginseng (*Eleutherococcus senticosus*), or aphrodisiacs such as damiana (*Turnera diffusa*, *syn*. *Damiana aphrodisiaca*) and muira puama (*Liriosma ovata*) for drinks designed for social occasions, primarily for sale in clubs and bars.

12.4.4.4 Regulatory issues

Regulations differ from country to country and vary over time as new legislation is enacted, so it is difficult to give any universal guidance other than to suggest that product development technologists use the food legislation experts at one of their local independent food research organisations, whose role it is to keep abreast of the latest developments in their national regulations.

In Europe and the United Kingdom, there are two issues to address. First, herbal drinks should avoid using levels of herbal extracts that are high enough to render them liable to be considered as a herbal remedy. In simple terms, this means using levels of extracts that avoid delivering cumulative therapeutic levels of herbal actives from a typical daily consumption of the herbal drinks. Information and guidance on these matters should be available from herbal extract suppliers or consultants.

The second issue is that of claims. This subject is difficult to write upon definitively as new or changed regulations are often put into effect. However, the trend seems to be that claims will need some form of substantiation based upon scientific reports. In the future, folklore and anecdotal information about herbs are less likely to be permitted on product labels, advertisements and point-of-sale literature.

For Europe and the United Kingdom, a useful reference work (*Natural Sources of Flavourings*) is published by the Council of Europe. It is commonly known as the ‘Blue Book’ and is gradually being superseded by a series of update volumes with greater detail on safety of use. To date only the first report, which deals with the most commonly used plant materials, has been published. This contains, as the name implies, a list of plants that have been used to generate flavourings for foods and drinks. Many of the plants listed are traditional herbs used before modern medicines for their curative or tonic benefits. The plants in the positive list (there are a few plants listed as being hazardous for use in food and drink) are divided into six categories, which are defined as follows:

- Category 1 – plants, animals and other organisms or parts thereof consumed as food in Europe: no restriction is made on the parts used under the usual conditions of consumption.
- Category 2 – plants etc. and parts thereof, including herbs, spices and seasonings, not commonly used as foodstuffs in Europe and considered not to constitute a risk to health in the quantities used.
- Category 3 – plants etc. and parts of these or products thereof normally consumed as food items, herbs or spices in Europe that contain defined ‘active principles’ or ‘other chemical components’ requiring limits on usage levels.

- Category 4 – plants etc. and parts of these or products thereof and preparations derived therefrom not normally consumed as food items, herbs and spices in Europe that contain defined 'active principles' or 'other chemical components' requiring limits on usage levels.
- Category 5 – plants etc. and parts of these or products thereof and preparations derived therefrom for which additional toxicological and/or chemical information is required.
- Category 6 – plants etc. and parts of these or products thereof and preparations derived therefrom that are considered to be unfit for human consumption in any amount.

12.5 Some commonly used herbs

This section gives information about some of the more commonly known herbs that have been used in drinks. This list generally excludes herbs and spices that are associated primarily with culinary or flavour use, although many of these will also have some health benefits. Some of the herbs listed below are of European or American origin and are listed in the 'Blue Book'. The names of listed herbs are followed by their category. Several herbs that have been used in the past, such as St John's Wort, Ma huang (Ephedra) and Kava-kava, have been omitted as they are currently withdrawn from the European market due to health concerns by the regulators.

Artichoke

Botanical name: *Cynara scoelymus*.

Region of origin: Mediterranean region.

Part of herb used: The fresh or dried basal leaves.

Main actives: Caffeic acid derivatives (*c*.1%) including chlorgenic acid, flavonoids (*c*.0.5%) and sesquiterpene lactones (up to 4%) – the major component is cyanaropicrin.

Benefits: The herb is claimed to stimulate the gall bladder and detoxify and regenerate the liver tissues. It has been used to treat dyspeptic problems. It has also been shown to reduce blood lipids, serum cholesterol and blood sugar levels. The high inulin content makes it a valuable vegetable for diabetics.

Folklore: Artichokes were grown as vegetables by both the Greeks and the Romans. It is only relatively recently that the artichoke has become medicinally interesting with the discovery of its beneficial action on the liver (Bown, 2003; Gruenwald *et al.*, 2002; Tierra, 1998).

Burdock

Botanical name: *Arctium lappa*.

Region of origin: Europe.

Parts of herb used: Dried aerial parts and roots.

Main actives: The aerial parts contain flavonoids and the root contains bitter components and inulin.

Benefits: In traditional terms, burdock is used as a blood purifier: it is claimed that it helps the kidneys to remove toxins from the blood.

Folklore: Burdock was traditionally used to treat skin complaints and inflammations.

The roots can be eaten raw in salads or cooked like carrots and the young leaf stalks can be scraped and cooked like celery (Bown, 2003; British Herbal Medicine Association, 1983; Hutchens, 1973; Shealy, 1998; Tierra, 1998).

Clover (red)

Botanical name: *Trifolium pratense*.

Regions of origin: The herb is indigenous to Europe, Central Asia, India and North Africa.

Part of herb used: Flowering tops.

Main actives: Volatile oil including benzyl alcohol, isoflavonoids, coumarin derivatives and cyanogenic glycosides. It has also been shown to contain genistein, a mildly oestrogen-like compound.

Benefits: Red clover is traditionally believed to have relaxant, expectorant and wound-healing properties.

Folklore: Clover was used to treat coughs and chest problems, especially whooping cough. It was also used to help with skin complaints. Clover was a very important fodder crop, to the extent that even in the medieval period varieties had been cultivated to improve the persistence and flowering time of the herb. The solid extract of clover has been used as a flavouring agent in a range of food products (Bown, 2003; British Herbal Medicine Association, 1983; Gruenwald *et al.*, 2002; Hutchens, 1973; Tierra, 1998).

Damiana

Botanical name: *Turnera diffusa*.

Region of origin: Mexico, Central America and northern South America.

Part of herb used: Dried leaves.

Main actives: Volatile oil, tannins, resins and glycosides.

Benefits: A bitter aromatic herb with a fig/date-like flavour that is claimed to be a nerve stimulant and was used to treat nervous exhaustion and anxiety of a sexual nature. It is claimed to be a mild irritant of the genito-urinary tract.

Folklore: The Mayans knew this plant as an aphrodisiac (Bown, 2003; British Herbal Medicine Association, 1983; Hutchens, 1973; Tierra, 1998).

Dandelion

Botanical name: *Taraxacum officinale*.

Region of origin: Europe extending north to the Arctic region, east to the Orient and south to North Africa.

Parts of herb used: The fresh and dried root and leaves.

Main actives: Sesquiterpene lactones, which are bitter flavoured, triterpenes, steroids, flavonoids, mucilages and an inulin content that varies from 2 to 40% in the autumn.

Benefits: The bitter components were used to promote the flow of digestive juices in the upper intestinal tract.

Folklore: The French name for this herb is 'Pissenlit', which is self-explanatory. The herb is regarded as a good diuretic to help purify the system by removing toxins. For some time now the roots have been roasted and then extracted to make a caffeine-free dandelion coffee. Dandelion has also been used in root beers and soft drinks such as Dandelion and Burdock (Bown, 2003; British Herbal Medicine Association, 1983; Gruenwald *et al.*, 2002; Hutchens, 1973; Shealy, 1998; Tierra, 1998).

Echinacea

Botanical name: *Echinacea purpurea*.

Region of origin: Indigenous to North America.

Parts of herb used: Fresh or dried rhizomes and roots.

Main actives: Water-soluble polysaccharides and glycoproteins, volatile oil (up to 2%), caffeic and ferulic acid derivatives including cichoric acid (0.6–2.1%), alkamides (0.01–0.04%), polyynes and pyrrolizidine alkaloids.

Benefits: Echinacea is held to be one of the most effective detoxifying herbs in Western medicine for a range of ailments, and is now used in ayurvedic medicine. The polyynes and cichoric acid components are reported to have antibacterial and virostatic effects. Echinacea also demonstrates an anti-inflammatory effect due to the alkamides component. As an immune stimulant, it significantly raises immunoglobulin M levels. Antiviral activities against both the Herpes simplex virus Type I and the influenza-A virus have been observed.

Folklore: *Echinacea purpurea* was used by native North Americans to treat wounds. Its use was greatly promoted by the Eclectic movement from the 1850s until the movement declined in the 1930s (Bown, 2003; Gruenwald *et al.*, 2002; Hutchens, 1973; Shealy, 1998; Tierra, 1998).

Elderflower

Botanical name: *Sambuccus nigra*.

[fruit: Category 3 (with limits on hydrocyanic acid); flowers and tips: Category 1; leaves and extracts: Category 5 (with limits on hydrocyanic acid)].

Region of origin: Europe.

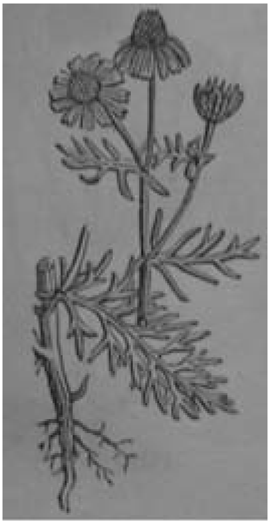
Part of herb used: The dried or fresh flowers.

Main actives: Flavonoids such as rutin, isoquercitrin and quercitrin, chlorogenic acids and volatile and fixed oils.

Benefits: Elderflowers have traditionally been used for colds and fevers as their main action is claimed to be to induce sweating and reduce temperature.

Folklore: Many old superstitions surround the elder. It was considered most unwise to cut down an elder tree without first seeking permission of the 'elder mother' spirit in the tree (Bown, 2003; British Herbal Medicine Association, 1983; Gruenwald *et al.*, 2002; Hutchens, 1973; Shealy, 1998; Tierra, 1998).

German chamomile



Botanical name: *Chamomilla recutita*.

Regions of origin: Indigenous to Europe and northern Africa.

Parts of herb used: Flowers and flowering tops.

Main actives: Volatile oil containing bisabolol compounds, flavonoids, coumarin compounds and mucilages.

Benefits: A bitter aromatic herb traditionally used for its gentle sedative, calming properties. It is also used to calm the digestive system. It is a mild herb that has been used for children's complaints.

Folklore: German chamomile is used in toiletry and cosmetic preparations as a hair conditioner and lightener (Bown, 2003; British Herbal Medicine Association, 1983; Gruenwald *et al.*, 2002; Hutchens, 1973; Shealy, 1998; Tierra, 1998).

Ginkgo

Botanical name: *Ginkgo biloba*.

Region of origin: China.

Part of herb used: Dried leaves.

Main actives: Proanthocyanidins (8–12%), flavonoids (0.5–1.8%), biflavonoides (0.4–1.9%), diterpenes and sesquiterpenes.

Benefits: Ginkgo is highly regarded as a tonic for the circulatory system. Studies have shown that it improves blood flow to the extremities of the body by reducing blood viscosity and dilating the blood vessels. Ginkgo has been shown to help retard the degenerative effects of Alzheimer's disease on cognitive functions, due presumably to improved blood flow in the cerebral capillaries. Other studies have shown that ginkgo improves memory and learning capabilities. One report has also claimed that ginkgo helps to counter loss of libido caused by antidepressants. It has been used by mountaineers to help counter altitude effects and by mountaineers to keep extremities warm.

Folklore: The ginkgo tree comes from a very ancient order of plants; the modern single species is almost identical to fossilised plants that were growing 65 million years ago – before mammals evolved. Chinese medicine used the seeds of the ginkgo tree, but it was only in the latter half of the twentieth century that Western medicine started to research the properties of the leaves (Bown, 2003; Gruenwald *et al.*, 2002; Shealy, 1998; Tierra, 1998).

Ginseng

Botanical name: *Panax ginseng*.

Region of origin: Indigenous from Nepal to Manchuria.

Parts of herb used: The main root, side roots and rootlets.

Main actives: Ginseng contains a complex mixture of triterpene saponins (0.8–6.0%) and also many ginsenosides, of which the predominant ones are Rb1 (0.15–1.2%), Rb2 (0.06–0.8%), Rc (0.1–1.2%), Rd (0.04–0.7%), Re (0.15–1.5%) and Rg1 (0.2–0.6%). Ginseng also contains water-soluble polysaccharides and some polyynes.

Benefits: Ginseng's main action is to help the body fight off physical, chemical and biological attacks by raising the body's own defence mechanisms. In human studies, there was a visible benefit in terms of physical and mental performance. It was also shown to reduce blood sugar levels in Type II diabetics.

Folklore: Ginseng is one of the oldest known tonic herbs, having been in use for about 5000 years in China. It was introduced into Europe several times from the end of the ninth century onwards, but did not become established until the middle of the twentieth century, following studies by Russian scientists that established its adaptogenic properties. Most ginseng is now cultivated since very few plants are found in the wild. South Korea grows a large amount of Ginseng under government control to ensure quality standards are observed (Bown, 2003; British Herbal Medicine Association, 1983; Gruenwald *et al.*, 2002; Hutchens, 1973; Shealy, 1998; Yeung, 1983).

Guarana

Botanical name: *Paullinia cupana*.

Region of origin: Amazon Basin.

Part of herb used: The seeds, which are purple–brown to black with a characteristic white 'eye', contained within a red–orange fruit about the size of a hazelnut.

Main actives: Guarana has the highest known caffeine content of any herb at 3.6–5.8%. It also contains small amounts of theophylline and theobromine, the other stimulant purine alkaloids similar to caffeine. Besides these, guarana contains about 12% tannins and some saponins.

Benefits: The caffeine content makes guarana a strong central nervous system stimulant. It is traditionally used as a tonic for fatigue and to allay hunger and thirst. It also has short-term diuretic effects. The tannin content gives guarana an astringent effect and it has been used to treat diarrhoea.

Folklore: Guarana is traditionally prepared by roasting the seeds to enable the shells to be removed, after which the seeds are crushed and ground into a paste that is fashioned into stick form and dried over a smoking aromatic charcoal fire. The Guaramis and other Amazon Indian groups grated a little of this dried guarana paste into water to make a stimulating drink that enabled them to overcome fatigue and hunger on long hunting trips (Bown, 2003; Gruenwald *et al.*, 2002; Tierra, 1998).

Hops

Botanical name: *Humulus lupulus*.

Region of origin: Europe, but now grown in temperate regions around the world.

Part of herb used: The dried strobile (female inflorescence).

Main actives: About 10% of α and β bitter acids including humulone and lupulone, volatile oil (0.3–1.0%)

Benefits: Hops are a traditional sedative and soporific (sleep promoter). The bitter acids have been shown to be antibacterial and antifungal and also to stimulate the secretion of gastric juices. Hormonal anaphrodisiac effects have also been reported.

Folklore: Hops are primarily associated with the brewing of beer, but many other herbs were used in brewing long before hops came to prominence. Hops were traditionally used to stuff pillows as a way of promoting sleep (Bown, 2003; British Herbal Medicine Association, 1983; Gruenwald *et al.*, 2002; Hutchens, 1973).

Horehound

Botanical name: *Marrubium vulgare*.

Region of origin: Indigenous from the Mediterranean region to Central Asia.

Part of herb used: The fresh or dried aerial parts of the plant.

Main actives: Diterpene bitter components including marrubiin (c.1%), caffeic acid derivatives including chlorogenic acid, flavonoids and a trace of volatile oil.

Benefits: The herb is an aromatic bitter that has been used to stimulate digestive juices. The herb is also a traditional expectorant.

Folklore: This herb was used as far back as ancient Egyptian times as a cough remedy. More recently it has been made into candy cough sweets. At one time horehound ale was brewed particularly in the East Anglia region of the United Kingdom. The leaves have also been used in liqueurs (Bown, 2003; British Herbal Medicine Association, 1983; Gruenwald *et al.*, 2002; Hutchens, 1973).

Kola

Botanical name: *Cola nitida* and *C. acuminata*.

[kola and kola nut extract: Category 4 (with limits on caffeine)].

Region of origin: West Africa.

Part of herb used: The seed after removal of the testa.

Main actives: Purine alkaloids – mainly caffeine (0.6–3.7%) with some theophylline and theobromine, tannins, proanthocyanidins and 45% starch.

Benefits: Besides the nervous stimulant effect due to the caffeine, kola is used in its indigenous region to stimulate the digestive system since it is claimed that it helps to break down fat. It has also been shown to have a mild diuretic effect, which is consistent with its caffeine content.

Folklore: Kola is traditionally used in tonics for exhaustion and poor appetite. The tannins have an astringent effect in cases of diarrhoea. In the countries of origin the seed is ground as a condiment for food and chewed before meals to promote good digestion (Bown, 2003; British Herbal Medicine Association, 1983; Gruenwald *et al.*, 2002; Tierra, 1998).

Lemon balm

Botanical name: *Melissa officinalis*.

Regions of origin: Eastern Mediterranean and Western Asia.

Part of herb used: Aerial parts of carefully dried herb.

Main actives: Complex mixture of volatile oils, glycosides of the alcoholic and phenolic volatile components, caffeic acid derivatives, flavonoids and triterpene acids.

Benefits: *In vitro* the herb has been shown to have antibacterial and antiviral effects. It has also been used as a calming sedative for nervous indigestion and is one of the herbs that are given to children for stomach upsets.

Folklore: The oil has insect repellent properties. The herb is used in cooking to impart a lemon flavour to the food. It is an ingredient of a melissa cordial made by Carmelite nuns as well as being included in other liqueurs such as benedictine and chartreuse. Traditionally the herb was seen as an antidepressive. (Bown, 2003; British Herbal Medicine Association, 1983; Gruenwald *et al.*, 2002; Tierra, 1998).

Limeflower

Botanical name: *Tilia cordata* and *T. platyphyllos*.

Region of origin: The tree is common throughout northern temperate zones.

Part of herb used: The dried flowers, extracts of which have a honey-like flavour with astringency.

Main actives: Mucilages (about 10%), flavonoids, tannins, chlorogenic acid and volatile oils.

Benefits: An aromatic mucilaginous herb that has traditionally been used for its diuretic and expectorant properties. It is claimed to calm the nerves, lower blood pressure and improve digestion.

Folklore: Limeflowers were thought to cure epilepsy if the sufferer sat under the tree. The wood of the lime tree is valued for its pale colour and its suitability for turning and carving. It is used in the manufacture of musical instruments (Bown, 2003; British Herbal Medicine Association, 1983; Gruenwald *et al.*, 2002; Shealy, 1998; Tierra, 1998).

Maté

Botanical name: *Ilex paraguariensis*.

Region of origin: South America between 20° and 30° latitude.

Parts of herb used: The dried leaf and leaf stems.

Main actives: Caffeine (0.4–2.4%) and theobromine (0.3–0.5%), caffeic acid derivatives including chlorogenic acid and neochlorogenic acid, flavonoids including rutin and isoquercitrin, saponins and volatile oil.

Benefits: The herb has stimulant effects due to the caffeine and chlorogenic acids. It is also diuretic and reportedly has lipolytic (fat-burning) effects.

Folklore: In South America, a tea brewed from the herb (also called 'maté') is served on social occasions as a communal recreational beverage that is very stimulating. The maté is prepared in a bowl that is passed around the assembled people; it is drunk from the bowl by means of a silver straw with a strainer on the lower end to prevent the leaves being ingested (Bown, 2003; British Herbal Medicine Association, 1983; Gruenwald *et al.*, 2002).

Meadowsweet

Botanical name: *Filipendula ulmaria*.

Region of origin: Europe.

Parts of herb used: The flowering tops and leaves.

Main actives: Salicin. It was from this plant that in 1838 salicylic acid was first isolated.

Benefits: Meadowsweet has traditionally been used for its astringent and antacid properties. It has long been held that it soothes and relieves pain, especially in joints and the digestive tract.

Folklore: Along with vervain and watermint, meadowsweet was one of the most important herbs for the Druids. It was also a popular strewing herb in medieval times (Bown, 2003; British Herbal Medicine Association, 1983; Shealy, 1998).

Nettle

Botanical name: *Urtica dioica*.

Regions of origin: Common throughout the temperate zones of the world.

Part of herb used: Fresh or dried aerial parts of the plant.

Main actives: The fresh leaves and stems are rich in vitamins A and C and iron. They also contain histamine, serotonin, acetylcholine and formic acid in the stinging hairs. The dried herb contains Flavonoids (0.7–1.8%) including rutin and isoquercitrin, silicic acid (1–5%), a trace of volatile oil and potassium and nitrate ions.

Benefits: Significant antirheumatic and antiarthritic actions have been demonstrated in several studies, some with large groups of participants. Diuretic properties have been reported in connection with prostate problems and in cases of lower urinary tract infections.

Folklore: The name urtica is believed to be derived from the Latin verb ‘urere’, to burn, most probably referring to the stinging action of the plant. Nettle is a fibrous plant and was used in cloth manufacture from the Bronze Age until the early twentieth century. The fresh young plant tops have been cooked as a spinach-like vegetable dish, and used to be brewed into a nettle beer in certain parts of the United Kingdom. The herb was known as a blood purifier which, in current terms, is a detox herb. The herb has a high chlorophyll content and has been used as a source for extraction of this natural colour (Bown, 2003; British Herbal Medicine Association, 1983; Gruenwald *et al.*, 2002; Hutchens, 1973; Shealy, 1998; Tierra, 1998).

Passionflower

Botanical name: *Passiflora incarnata*.

Region of origin: South-eastern United States to Argentina and Brazil.

Part of herb used: The aerial tops of the stems, comprising leaves flowers and fruit.

Main actives: Flavonoids.

Benefits: The flavonoids have led to this herb's longstanding use as an effective, non-addictive sedative that does not cause drowsiness. Passionflower is an ingredient in many herbal sedative remedies.

Folklore: Spanish missionaries in South America regarded the flower of this herb as a symbol of Christ's passion, the three stigmas representing the nails, the five anthers the wounds and the ten sepals the apostles present. The herb was used in native North American medicine, especially by the Houma tribe, who put it into drinking water as a tonic. It became popular as a treatment for insomnia in the nineteenth century and was included in the US National Formulary from 1916 to 1936 (Bown, 2003; British Herbal Medicine Association, 1983; Gruenwald *et al.*, 2002; Hutchens, 1973; Tierra, 1998).

Rooibos

Botanical name: *Aspalathus linearis*.

Region of origin: Western Cape region of South Africa.

Parts of herb used: Leaves and stems of new growth shoots.

Main actives: A range of polyphenols.

Benefits: Japanese studies in the 1980s showed that the polyphenol components had antioxidant properties similar to superoxide dismutase (SOD), an enzyme that is a free radical scavenger and is thought to slow down the ageing process. The herb has less tannin than oriental tea, so tastes less bitter, and it is caffeine free.

Folklore: Locally, the herb is used in cases of allergy, such as eczema, hay fever and asthma. It is also used in schnapps and liqueurs (Bown, 2003).

Rosehip

Botanical name: *Rosa canina*.

Region of origin: Europe.

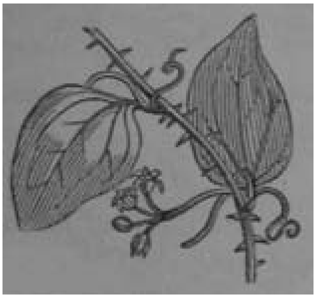
Part of herb used: Fruit.

Main actives: Vitamin C (0.2–2.4%), fruit acids (3%), pectins (15%), sugars (12–15%), carotenoids and flavonoids.

Benefits: A source of natural vitamin C that has been used in cold and influenza preparations.

Folklore: Rosehips are also used to make syrups for babies and young children. Traditionally rosehips were made into preserves to retain their health benefits into the winter months (Bown, 2003; British Herbal Medicine Association, 1983).

Sarsaparilla



Botanical name: *Smilax regelii*, *S. aristolochiaefolia*, *S. febrifuga*.

Region of origin: Tropical and subtropical Central America.

Parts of herb used: The dried rhizomes and roots.

Main actives: Steroidal saponins (0.5–3%).

Benefits: Sarsaparilla has long been used for skin complaints such as psoriasis; it is also believed to be a good diuretic and diaphoretic so it has been used as a blood purifier and for kidney complaints.

Folklore: Although there are steroidal compounds present in sarsaparilla, the rumoured presence of testosterone, which made it of interest to body-builders, has not been substantiated. The root has been used in soft drinks and root beers. Sarsaparilla was introduced into Europe following the Spanish colonization of South America. It was regarded as a cure-all and was established in pharmacopoeias until the early twentieth century (Bown, 2003; British Herbal Medicine Association, 1983; Gruenwald *et al.*, 2002; Tierra, 1998).

Schisandra

Botanical name: *Schisandra chinensis*.

Region of origin: China.

Part of herb used: Fruit.

Main actives: Essential oil, fruit acids, sugars and resin; the seeds contain schizandrins, sitosterol, vitamins C and E, resin, tannins and sugars.

Benefits: A sweet and sour herb that is claimed by Chinese herbalists to control the secretion of body fluids, thus moistening dry and irritated tissues, and to act as a tonic for the nervous system and the circulatory system.

Folklore: The herb was mentioned in Chinese texts during the Han dynasty (AD 25–220). Its Chinese name is Wu Wei Zi, which means five-flavours fruit, because it has both sweet and sour flavours in the fruit skin and flesh, and acrid, bitter and salty flavours in the seeds. It was used by both men and women as a sexual tonic, and by women to improve the complexion (Bown, 2003; Shealy, 1998; Yeung, 1983).

Siberian ginseng

Botanical name: *Eleutherococcus senticosus*.

Regions of origin: Siberia and northern parts of China, Korea and Japan.

Parts of herb used: The dried root and root bark.

Main actives: The main constituents are triterpene saponins, steroid glycosides, hydroxycoumarins, caffeic acid derivatives, lignans, steroids and polysaccharides.

Benefits: The actions of Siberian ginseng are similar to those of *Panax ginseng* but stronger. Indigenously it is used as a tonic for strength and revitalisation. The polysaccharides in the herb have been shown to have a good immunostimulatory action.

Folklore: Siberian ginseng was brought to prominence when Russian researchers were investigating *Panax ginseng* and looked at other plants in the same family to see whether they had similar properties. Several Eleutherococcus species have been used in Chinese medicine for 2000 years (Bown, 2003; Gruenwald *et al.*, 2002; Shealy, 1998; Tierra, 1998; Yeung, 1983).

Valerian

Botanical name: *Valeriana officinalis* [roots: Category 5].

Regions of origin: Europe and temperate regions of Asia.

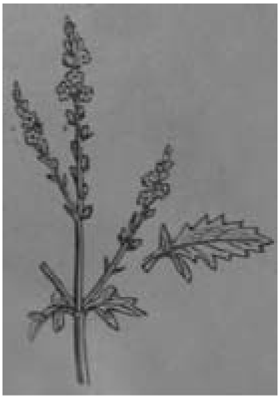
Parts of herb used: Fresh or carefully dried rhizomes and roots.

Main actives: Valepotriates (0.5–2.0%), volatile oil (0.2–1.0%) and valeric acid (0.1–0.9%).

Benefits: Valerian has been used as a daytime sedative to reduce anxiety and stress and it has been demonstrated to reduce the time it takes to fall asleep. Valerian root extracts and volatile oils are used as components in the flavour industry, especially in alcoholic beverages such as beers and liqueurs and in soft drinks such as root beers. They have also been used in tobacco flavours.

Folklore: The aroma of valerian is very attractive to cats and rodents, and it has been used as bait in traps. It is thought that valerian was the basis for the story of the Pied Piper of Hamlin ridding the city of rats (Bown, 2003; British Herbal Medicine Association, 1983; Gruenwald *et al.*, 2002; Hutchens, 1973; Shealy, 1998; Tierra, 1998).

Vervain



Botanical name: *Verbena officinalis*.

Region of origin: Mediterranean region.

Part of herb used: Aerial parts of the herb.

Main actives: Iridoids, flavonoids and caffeic acid derivatives.

Benefits: The herb has been used for its astringent, cough suppressant and lactation promoting properties. Traditionally it has been used as a diuretic and to calm the nerves and improve the liver and gall bladder functions.

Folklore: In Western medicine, vervain has been used mainly for nervous complaints. Vervain is one of the herbs most commonly used to make herbal teas and is also an ingredient in liqueurs (Bown, 2003; British Herbal Medicine Association, 1983; Gruenwald *et al.*, 2002; Shealy, 1998; Tierra, 1998).

Wolfberry

Botanical name: *Lycium barbarum*.

Region of origin: China.

Part of herb used: Fruits, which are bright red when first dried but darken with age.

Main actives: Carotenes, vitamins B1, B2, B3 and C, β-sitosterol and linoleic acid.

Benefits: In traditional Chinese medicine, wolfberry is used as a nourishing herb for convalescence; it is also used in cases of impotence. It has been reported to lower blood pressure and blood cholesterol levels. It is also reported to be a liver and kidney tonic, inhibiting the deposition of fat in liver cells and promoting the regeneration of liver cells.

Folklore: Although of Chinese origin (referred to as Gou Qi Zi as early as 200 BC) the plant has been established in Europe for some centuries and, in Britain, was known as the Duke of Argyll's tea-tree. It was also known as matrimony vine because if planted near the home it was said to create discord between husband and wife. In traditional Chinese medicine it is combined with Schisandra and if taken for one hundred days is believed to develop sexual stamina (Bown, 2003; Shealy, 1998; Tierra, 1998).

Wormwood

Botanical name: *Artemesia absinthium*. [Herb: category 4 (with limits on eucalyptol, methyleugenol and thujone); essential oil: category 4 (with limits on eucalyptol, methyleugenol and thujone)].

Region of origin: Mediterranean zones.

Parts of herb used: The upper shoots and leaves.

Main actives: Volatile oil (0.2–1.5%) containing thujone and bitter sesquiterpene compounds, including absinthin (0.20–0.28%) and artabsin (0.04–0.16%).

Benefits: The herb is described as an aromatic bitter, and as such is used to stimulate the appetite. It has been shown that the bitterness on the palate automatically stimulates an increase in secretion of digestive juices in the stomach. Also, it has been shown to increase liver function in patients with liver disorders.

Folklore: Wormwood has traditionally been used for digestive problems, including expelling intestinal worms. The essential oil of wormwood was used in the distillation and production of the aperitif absinthe, starting around the end of the eighteenth century. The thujone in absinthe created some problems in Europe and the United States, as it emerged that it was addictive and could cause hallucinations in cases of overindulgence. This led to the drink being outlawed in certain countries (Bown, 2003; British Herbal Medicine Association, 1983; Gruenwald *et al.*, 2002; Hutchens, 1973; Tierra, 1998).

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13 Special topics

Q. Palmer

13.1 Complaints and enquiries

Tactful and sympathetic handling of communications with consumers will pay dividends. Complaints can often be defused with minimum loss of goodwill and promptly answered enquiries will generate it.

13.1.1 Complaints

No matter how much care a manufacturer takes, it is inevitable that occasionally consumers will believe they have cause for complaint about a product. It is often said that fewer than one in 10 people who are dissatisfied with a soft drink bother to make a complaint but that most of them will tell their friends and family.

Complaints reach the manufacturer either directly from the consumer or via a retailer or from a government agency (in the UK either a Trading Standards Officer (TSO) or an Environmental Health Officer (EHO)). Most complaints fall into one of the following categories:

1. substandard composition
   - ingredients omitted, added twice or the wrong ingredient added
   - syrup over or under dosed
2. substandard formulation
   - label claim not met (for example a vitamin claim or ‘no added preservatives’)
3. deteriorated product
   - improper storage (for example too hot or in direct sunlight)
   - aged product
   - loss of carbonation (PET bottles or faulty closures)
   - microbiological spoilage (fermentation, mould growth or bacterial spoilage)
   - metal pick-up (old or damaged cans)
4. contaminated product
   - accidental (glass in product, inadequate washing of returnable bottles)
   - deliberate (extortion or grudge)
5. damage, illness or injury caused by product
   - staining of carpets or textiles after spillage or leakage
   - glass injuries from exploding or dropped bottles

  - internal injuries from glass in product
  - illness due to consumption (contamination, revulsion due to off-flavours, high metal content; pathogens unlikely since pH generally too low)
6. personal feelings
   - dislike of the product
   - objection to an ingredient used

The nature of the complaint dictates the appropriate response, which could range from a conciliatory letter ('I am sorry you do not like our product...') to a full public recall of product and cessation of production until a fault is resolved (for example a major incident of fermentation in a glass bottled product). In all cases the cycle of events must be to understand the problem, to identify the cause, to decide the response and then to take appropriate action.

Written complaints and those raised by or via government agencies do not require an *instantaneous* response to the complainant, but increasingly nowadays a consumer will telephone, often very angry, and always expecting an immediate resolution of the issue. A calm, polite and measured response is called for, without waffle or technical jargon.

It is an excellent maxim always to consider the situation from the complainant's point of view and if the product is clearly at fault then an apology should be offered; this often serves to disarm a complainant expecting to do battle. Note, however, that admitting liability should be avoided as it restricts negotiations if legal proceedings ensue and may invalidate liability insurance.

All complaints should be dealt with as rapidly as possible, and a telephoned response to a letter not only saves time but gives an opportunity to impress the complainant that the matter is being taken seriously.

Threats to 'take it to the newspapers/police/public health' can be made when a complainant is very upset, or feels that compensation should be offered and wants to talk-up the amount. In such cases attempts at dissuasion are likely to be counterproductive. It is generally best to agree that the complainant is at liberty to take this action and to suggest the most appropriate body (EHO or TSO) but also to point out that it will inevitably delay investigations.

A typical complaint should be processed through some or all of the following stages:

1. Receipt of complaint
   - note date of arrival
   - send acknowledgement if investigation is likely to be lengthy
2. Follow up if full information not provided
   - nature of complaint
   - product and pack
   - production code and time
   - where and when purchased

3. Initial assessment and categorisation
   - nature of fault
   - degree of seriousness
   - extent of fault (specific to one package, to a quantity of badly stored product or to part or all of a production batch)
4. Is there a need to obtain the complaint sample?
   - is there a parallel EHO or TSO investigation?
5. Check with production site
   - were there any abnormalities noted at the time?
   - was it the start or end of a batch?
6. Assess product if available
   - 'tastes nasty' could be caused by microbial spoilage, adverse storage, compositional faults or contamination
7. More detailed investigation if appropriate
   - chemical and/or microbiological analysis of drink
   - physical and/or chemical examination of foreign bodies
8. Brief legal advisor or insurers if appropriate
9. Formulate internal action to be taken, if any
   - modification of product formula
   - changes to factory procedures
   - product recall
10. Prepare and issue response to complainant

The final response to the complainant should include a simple explanation of the cause of the fault, what is being done to rectify it, what steps (if any) are being taken to prevent a recurrence and an apology.

A typical example would be:

Dear Mr...

Reference your letter dated...

I am very sorry indeed that one of the bottles of our orange juice that you purchased had a layer of mould on the surface.

Examination of the product has shown that there is a small chip on the neck of the bottle. This prevented the crown cap from making a perfect seal and allowed a mould spore to enter the bottle and grow on the surface of the juice.

We have re-evaluated our washed-bottle inspection procedures at the factory where the juice was bottled and have made modifications that should prevent similar incidents in future. I am sending you separately some replacement bottles of orange juice, which I hope will make up for the distress and inconvenience you have been caused.

Yours sincerely,

13.1.2 Enquiries

Consumers are increasingly more curious about the composition of the soft drinks they buy, and are also more concerned about health and dietary considerations. Many soft drink companies now operate telephone consumer response services and some also have pages on the Internet. These make enquiries much easier for the consumer and undoubtedly encourage communication with the manufacturers. This has benefits for both parties. The consumer has an easy route to obtain product information and to enhance his relationship with a favourite brand of drink, and the manufacturer can build on the consumer's loyalty and establish a database of known consumers for subsequent consumer research or marketing activity.

Responding to enquiries can be speeded up and handled by non-technical staff if an information database is established. Typical product enquiries for which answers should be available are:

How many calories are in . . .?
Which of your products are suitable for
- vegetarians?
- diabetics?
- Muslims?

Why do you use
- preservatives?
- sweeteners?
- 'additives'?

More general enquiries tend to originate from the education system and it is interesting to speculate how many GCSEs the average consumer response operative has earned on behalf of enquiring young consumers.

The predictable 'How do you make soft drinks?' is readily answered with some publicity material. In the UK there is some excellent published material available from the British Soft Drinks Association but responding to more advanced queries can be time-consuming. Examples of more complex questions are:

- 'My A-level project is about vitamin C in foods: how much vitamin C is in your soft drinks and what is the best analytical method for checking?'
- 'For my final year degree project I am researching the contribution of soft drinks to the diet. Please advise nutrition information and consumption patterns for all your drinks, covering per capita consumption for the following age ranges . . .'

13.2 Nutrition

Soft drinks and fruit juices are usually consumed to quench thirst or as a sociable activity, rather than for their nutritional content. However, they always provide dietary water and, depending on formulation, can also contribute significant quantities of carbohydrate, vitamins, minerals and protein. The calculation of nutrition information is straightforward.

13.2.1 Nutritional components

13.2.1.1 Water

Sedentary adults in a temperate climate need to consume about 2 l of water per day, a figure that can substantially increase when high temperature and/or physical activity causes significant sweating. Soft drinks and fruit juices provide a valuable water source, as shown in Table 13.1.

Table 13.1 Water content of soft drinks and juices

Drink	Typical water content (% v/v)
Soda water	99.8
Lemonade	94–97
Cola	93.5
Orange juice	88

13.2.1.2 Carbohydrates

With the exception of soda waters and most low-calorie drinks, all soft drinks rely at least partially on sugars to provide sweetness and body.

Although different sugars have different sweetness factors relative to sucrose they are all identical in energy content, on a dry basis. Thus to achieve a high energy content without cloying sweetness, sugars or carbohydrates of low sweetness must be used. This is illustrated in Table 13.2.

Table 13.2 Sugars, sweetness and energy

Carbohydrate	Sweetness, relative to sucrose	kcal required to provide the same sweetness as 1 g sucrose
Fructose	1.3 ×	3
Sucrose	1.0 ×	4
Dextrose	0.8 ×	5
Glucose syrup (50 DE)	0.6 ×	6.7
Maltodextrin	0.05×	80

Most soft drinks contain less carbohydrate than fruit juices, although this is not the perception of the consumer (Table 13.3).

Table 13.3 Carbohydrate content of soft drinks & fruit juices

Beverage	Typical carbohydrate content (% w/v)
Low-calorie lemonade	0
Lemonade	5
Grapefruit juice	8.5
Orange juice	9.1
Cola	10.6
Pineapple juice	10.8

13.2.1.3 Protein

Unless deliberately added, soft drinks contain negligible amounts of protein, and fruit juices typically contain between 0.2 and 0.6% (Table 13.4)

Table 13.4 Fibre and protein contents of fruits and fruit juices

	Fibre (% w/w)		Protein (% w/w)	
	Fruit	Juice	Fruit	Juice
Apple	1.8	trace	0.4	0.1
Grape	0.7	trace	0.4	0.1
Grapefruit	1.3	trace	0.8	0.4
Orange	1.7	0.1	1.1	0.5
Pineapple	1.2	trace	0.4	0.3

Source: Food Labelling Data for Manufacturers (1992), published by the Royal Society of Chemistry and the Ministry of Agriculture, Fisheries and Food.

Protein-fortified soft drinks have been developed and marketed but there are no mainstream brands currently successful in the world. Generally the protein source used is dairy based; usually whey or skimmed milk. Great care must be taken to prevent precipitation as the protein is acidified through its isoelectric point to a microbiologically safe pH and it is necessary to use stabilisers for long-term cloud stability.

The high cost of ingredients and processing mitigates against commercial success and against such drinks providing a practical nutritional supplement in Third World and famine situations.

13.2.1.4 Fat

The fat content of soft drinks is negligible and any present will derive from essential oils added as constituents of flavourings, citrus comminutes or clouding emulsions.

Processed fruit juices are usually manufactured to a maximum oil content of 0.03% v/v at natural strength.

Drinks fortified with fat are usually dairy based, which puts them outside the scope of this book.

13.2.1.5 Fibre

This term is used to refer to food constituents not metabolised by humans and analysis has shown the majority of such constituents to be polysaccharides.

In the UK fibre is defined as 'non-starch polysaccharides' (NSP) and for consistency the Englyst analytical method has been adopted (Englyst and Cummings, 1988).

The only sources of NSP in soft drinks are fruit materials, gums and stabilisers such as sodium carboxymethylcellulose (CMC) and pectins. Of these items, only fruit juices are used in significant quantities. The NSP content of most fruits falls within the range 0.9–3.6% w/w but, as Table 13.4 shows, very little remains in processed juices. Citrus comminutes are conventionally considered to have the same NSP content as the corresponding fruit but if they are known to contain substantial amounts of peel extracts this should be accounted for in calculations.

The recommended daily intake of NSP is 18 g/day so conventional soft drinks provide an insignificant contribution. High levels of fortification can be achieved but a high-fibre drink recently launched in the UK did not succeed, despite good taste and texture and skilful marketing.

13.2.1.6 Vitamins

The only vitamins likely to be found in unfortified soft drinks are vitamin C (either added as an antioxidant or deriving from fruit materials) and vitamin A precursor (beta-carotene, added as a colour). However, soft drinks provide a good medium for vitamin fortification, the limitations being solubility (for fat-soluble vitamins), flavour impairment (for example the 'meaty' notes of thiamine) and stability.

It is very important to minimise the air content of vitamin-containing drinks to reduce oxidative damage and to ensure that sufficient 'overage' has been added to enable any label claims to be substantiated throughout the life of the product.

13.2.1.7 Minerals

Soft drinks will contain sodium (from water and added sodium salts, e.g. benzoate, saccharin, citrate, CMC), calcium and magnesium (from water and fruit materials) and potassium and phosphate (from fruit materials).

Adding minerals is easily achieved but excesses can give rise to salty or astringent notes, metallic taints and laxative effects.

13.2.1.8 Energy

Palatable high-energy drinks are readily formulated by selecting the appropriate carbohydrate blend (Table 13.2).

13.2.1.9 Low-calorie drinks

Paradoxically, the nutritional component of greatest commercial significance is the absence of nutrition, achieved by substituting carbohydrate sweeteners with non-nutritive sweeteners such as saccharin and acesulfame-K, or the amino acid sweetener aspartame.

13.2.2 Calculation and declaration of nutrition information

Within the European Union the provision of nutrition information is only obligatory if nutrition claims are being made or implied for the product and, whether obligatory or not, if information is provided it must be derived in the prescribed manner. In the UK the requirements are set out in the Food Labelling Regulations 1996 (SI 1996 1499).

The descriptor 'low calorie' is specifically exempted from necessitating nutrition information but reference to the following terms would act as a trigger:

diet
slim (used alone or as part of a brand name)
high energy
no added sugar
protein rich

The minimum requirement is to declare, per 100 ml (or 100 g), the amount of the following in the product:

energy (both as kJ and kcal)
protein (g)
carbohydrate (g)
fat (g)

If claims relating to specific nutrients are made (for example 'no added sugar') then the following additional data must be given:

carbohydrate: to be subdivided into sugars, polyols and starch, as appropriate
fat: to be subdivided according to saturation, if appropriate.
fibre (g)
sodium (g)
vitamins
minerals

If vitamins and/or minerals are claimed they must be declared both quantitatively and as a percentage of the recommended daily allowance specified in the regulations.

Values must be quoted in the units specified and are to be average values. They may be based on an analysis of the drink or may be calculated from generally established and accepted data, such as *Food Labelling Data for Manufacturers* (Royal Society of Chemistry/Ministry of Agriculture, Fisheries and Food).

For energy calculations the factors shown in Table 13.5 must be used.

Table 13.5 Energy factors per gram

	kJ	kcal
Carbohydrate	17	4
Polyols	10	2.4
Protein	17	4
Fat	37	9
Ethanol	29	7
Organic acids	13	3

Example

Calculate the full nutrition information for an orange drink with the following composition:

Ingredient	Quantity per 1000 l	Nutrition contribution
Sugar	60 kg	carbohydrate
Sodium benzoate	0.16 kg	sodium
Orange juice	50 l	carbohydrate, protein, fibre
Acesulfame-K	0.1 kg	—
Aspartame	0.1 kg	protein
Citric acid anhydrous	2.2 kg	organic acid
Sodium citrate dihydrate	0.8 kg	organic acid, sodium
Flavouring (50% v/v ethanol)	1.0 l	ethanol

Total acidity: 0.27% w/v, as citric acid anhydrous

Orange juice composition:

	% w/w
Carbohydrate	8.7
Protein	0.5
Fat	0.02
Fibre	0.15
Apparent density	1.042 g/ml (so 50 l = 52.1 kg)

Calculations

		Per 1000 l	g/100 ml
(i)	Carbohydrate content		
	added sugar	60.0 kg	
	ex juice, 52.1 × 8.7/100	3.53 kg	
		63.53 kg	6.35
(ii)	Protein content		
	aspartame (100% protein)	0.1 kg	
	ex juice, 52.1 × 0.5/100	0.26 kg	
		0.36 kg	0.036
(iii)	Fat content		
	ex juice, 52.1 × 0.02/100	0.01 kg	
	ex flavour	Negligible	
		0.01 kg	Trace
(iv)	Fibre content		
	ex juice, 52.1 × 0.1/100	0.05 kg	0.005
(v)	Organic acids		
	total acid content, 1000 × 0.27/100	2.7 kg	
	sodium citrate, 0.8 × 192.13/294.1	0.52 kg	
		3.22 kg	0.322
(vi)	Ethanol		
	1 l flavour @ 50% v/v = 0.5 l		
	ethanol, apparent density = 0.789 g/ml		
	mass of ethanol	0.39 kg	0.039
(vii)	Sodium		
	sodium benzoate, 0.16 × 23/144	0.026 kg	
	sodium citrate, 0.8 × (23 × 3)/294.1	0.188 kg	
		0.214 kg	0.021

(viii) Energy content per 100 ml (Table 13.5)

	kJ	kcal
due to 6.35 g carbohydrate	108.0	25.40
due to 0.036 g protein	0.6	0.14
due to 0.322 g organic acids	4.2	1.00
due to 0.039 g ethanol	1.1	0.27
total	113.9	26.81

The full declaration will thus be as follows, rounded off appropriately:

Energy	114 kJ (26.8 kcal)
Protein	0.04 g
Carbohydrate	6.4 g
(of which sugars	6.4 g)
Fat	0.0 g
Fibre	Trace
Sodium	0.02 g

13.3 Soft drinks and dental damage

Soft drinks have long been blamed for causing damage to teeth, especially among children. In this section the validity of this is discussed in the context of the widespread use of fluoridated toothpaste, mechanisms of damage are reviewed and ways of minimising damage are considered.

13.3.1 Causes of dental damage

Figure 13.1 shows the structure of a tooth, which relies on the integrity of the hard enamel external layer to protect the vulnerable interior.

There are three routes to tooth damage, all of which involve the breaching of the enamel and all of which have existed since mankind evolved:

Trauma. Accidental or deliberate damage to the tooth structure.

Toothwear. This occurs in three separate ways:

(i) by abrasion – gnawing on bones or chewing food contaminated by grit (for example crudely milled grain). In developed countries a more frequent cause is aggressive tooth brushing, especially when the enamel has recently been softened by acidic foodstuffs.
(ii) by attrition – tooth to tooth contact, caused by poor alignment of the upper and lower rows of teeth, and by habitual grinding of the teeth.
(iii) by erosion – chemical damage caused by acids. Chewing, sucking or drinking acidic foods, liquids or medications brings the teeth into direct contact with acids that will dissolve calcium and phosphorus from the enamel layer. Additionally, gastric reflux (the regurgitation of acidic stomach contents) can cause erosion on the back surface of the front teeth. This is specially frequent in infants and in those with eating disorders such as bulimia nervosa.

Caries. Oral bacteria, in association with glycoproteins from saliva, form a sticky coating on the surface of the teeth known as plaque. When carbohydrates

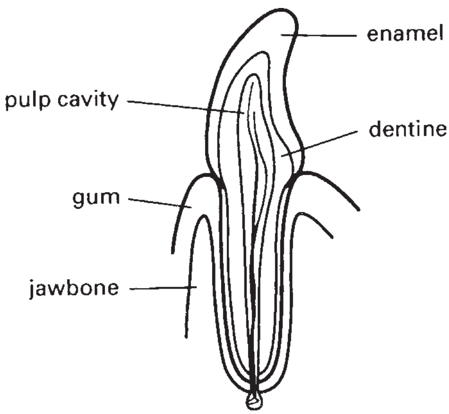

Figure 13.1 Tooth structure.

are eaten they provide substrate for the plaque bacteria to ferment into organic acids (mainly lactic acid). Sugars are directly metabolised: starch must first be digested to sugars by salivary amylase. The acids are retained in contact with the tooth surfaces by the plaque and, as in erosion, can attack the enamel by demineralisation. Once the mouth has cleared fermentation ceases and saliva acts to raise the pH at the tooth surface, allowing remineralisation to take place.

Unlike erosion, caries damage is localised to areas where plaque most readily accumulates, i.e. between the teeth, at the gum margins and in fissures and irregularities on the tooth surfaces.

13.3.2 Can soft drinks cause dental damage?

The answer is clearly yes: except for low-calorie drinks they provide a nutrient source of mono- and disaccharides for acid formation and, like all acidic foodstuffs, they have the potential to cause erosion. However, there are two mitigating factors serving to reduce greatly the damage that soft drinks might at first be thought to cause. These are:

(i) their very brief residence time in the mouth: unlike sucked sweets or chewed starchy or sugary foods there is no debris left behind after swallowing, reducing available substrate for the oral bacteria
(ii) the saliva flow stimulated by their acidity neutralises acidity in the mouth and plaque, and contains calcium and phosphate for remineralisation.

The effect on plaque pH as a consequence of consuming various foods demonstrates these effects clearly (Schachtele and Jensen, 1981). Demineralisation can occur at pH 5.7 or below so measurements of the time plaque pH remains below 5.7 after consumption of different foodstuffs allows ranking in order of potential tooth damage:

Least damaging	cheddar cheese
↓	skimmed milk
	soft drinks
	potato crisps
Most damaging	cake

13.3.3 Fluorides

The introduction of fluoridated toothpaste during the 1970s caused a much greater than expected improvement in the dental health of the nation, which far outweighs dietary initiated tooth decay in those that use it. There are now very few brands of toothpaste on sale in the UK that do not contain fluoride.

Fluoridated drinking water is currently available to only 12% of the UK population, a figure unlikely to grow due to strong opposition from pressure groups opposed to mass medication. Fluoride protects teeth in four ways (Shellis and Duckworth, 1994):

(i) it reduces mineral solubility
(ii) it inhibits mineral dissolution
(iii) it promotes remineralisation of the enamel
(iv) it inhibits acid production by plaque bacteria.

Figure 13.2 shows how the incidence of tooth decay amongst 12-year-old children has declined over recent years, despite a significant increase in their consumption of soft drinks.

13.3.4 Making soft drinks more tooth friendly

The beneficial protective effects of fluoride are obviously only available to those who use it and recent studies have shown that there are substantial variations, both geographically and by social class, in rates of toothpaste use (Davies and Hawley, 1995).

Dentists believe there is a strong case to formulate soft drinks to minimise the damage they could potentially cause to teeth. They have recently put forward proposals to reduce their erosivity and cariogenic properties and these are:

(i) a reduction in sugar content, to combat caries
(ii) an increase in pH to 5.4 or above, to reduce erosion.

Practical steps against caries. Sugar-free drinks are widely available and are targeted at all age ranges rather than just at slimmers (a reduction in sugar content would have little effect: it is its total absence that is necessary).

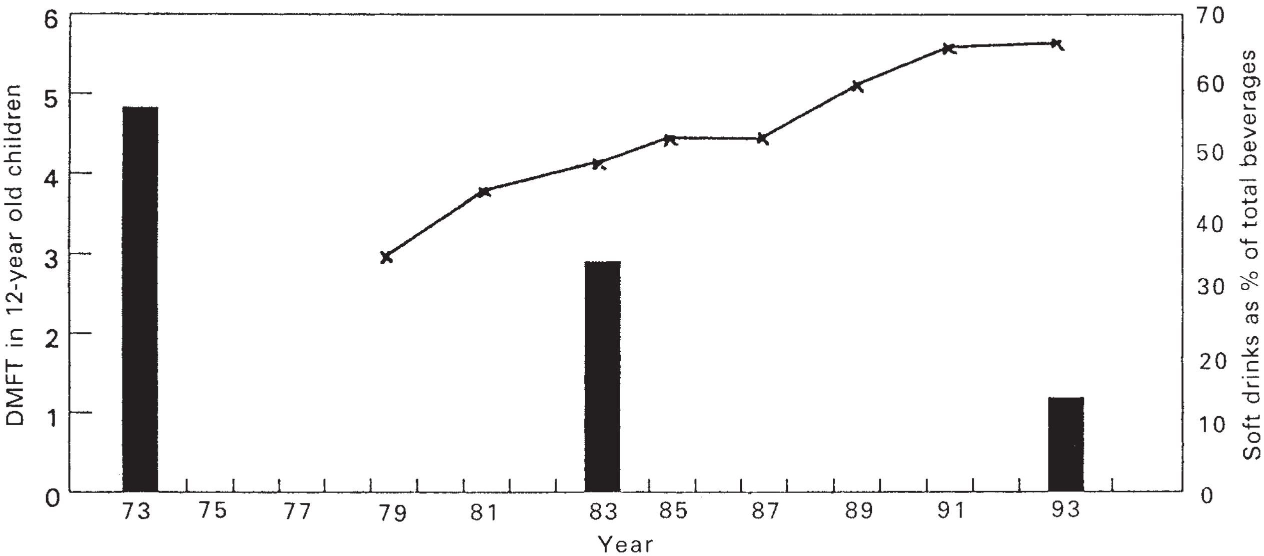

Figure 13.2 Tooth decay and soft drink consumption 1973–93. Bar chart: decayed, missing and filled teeth (DMFT) in 12-year-old children (O’Brien, 1994). Line graph: soft drinks as a percentage of the total beverages consumed by 10- to 12-year-old children (data from Nestlé National Drinks Surveys).

Practical steps against erosion. The proposed pH target is not sensibly achievable. pH 5.4 is generally recognised as the cut-off point for erosion but unfortunately this is far higher than the pH barrier of 4.5 above which pathogenic organisms can survive and grow. Any soft drink with such a high pH would need to be retorted: patently not a practicable proposition, especially for carbonated drinks. Fruit juices and fresh fruit all have pHs below 5.4 and cannot be modified. Typically soft drinks have a pH in the range 2.4 to 3.5 and it would be possible to move most of them towards the higher end of this range, although this would still be well below the target value.

It has been shown that the titratable acidity of a drink is a better indicator of its erosivity than its pH (Grenby *et al*., 1989). However, to significantly reduce acidity would have a much greater effect on the character of a drink than would buffering it to a modestly higher pH.

Given that their normal consumption is unlikely to damage teeth, the best approach to reducing the possibility of erosion by soft drinks and juices would be to promote guidelines for consuming them in the best way, i.e.:

– swallow drinks rather than sipping them over prolonged periods
– do not swill them around the mouth
– do not drink them last thing at night.

13.4 Ingredient specifications

In this section the purpose and preparation of ingredient specifications are discussed.

13.4.1 Why have specifications?

A specification serves to define the ingredient being purchased and should form part of the contract between buyer and seller. It is not sufficient to order 1 tonne of citric acid: the order should be for '1 tonne of citric acid, anhydrous, to our specification reference...'. In the UK a properly drawn up and agreed specification can also form part of a due diligence defence against a prosecution under the Food Safety Act 1990.

13.4.2 What a specification should include

There are three key aspects that should be included in all specifications:

(i) it should describe the item, including, if appropriate, a quantified definition of its functionality

(ii) it should specify the required purity or composition of the item and the maximum acceptable levels of contaminants (organic, inorganic and microbiological)
(iii) it should identify how the item will be packaged and labelled, how it should be stored and its minimum shelf life.

13.4.3 Preparation of a specification

A good starting point is always the supplier's own specification, which can be expanded to include requirements specific to the required end use (for example particle size for powdered drink ingredients).

Additional information sources include:

(i) The various European Union Directives specifying criteria of purity:
   - 95/31/EC Sweeteners for use in Foodstuffs
   - 95/45/EC Colours for use in Foodstuffs
   - 96/77/EC Food Additives other than Colours and Sweeteners for use in Foodstuffs

(ii) The various Pharmacopoeias:
   - British
   - European
   - US

(iii) The Food Chemicals Codex, 4th edition
(iv) The Merck Index

The specification must be discussed and agreed individually with all potential suppliers, who must be provided with a copy of the final document.

Although the preparation of the specification appears to be a straightforward process there are pitfalls that must be considered.

(i) When the supplier is not the manufacturer, but is an agent buying on the open market, is the supplier's expertise sufficient to ensure that all sources are capable of meeting the specification?
(ii) When the supplier manufactures to a standard grade and will not deviate from it, is the required deviation essential and realistic? If so, an alternative supplier must be found.
(iii) Seasonal variations influence fruit material availability. It is generally possible to source fruit materials to quite tight specifications but this can incur a heavy cost penalty for blending or selection. Setting as wide a tolerance as possible saves money and adds flexibility in poor seasons.

(iv) Specified parameters must be unambiguous and test methods must be defined, for example, is ‘Brix’ to be measured by density or by refractometer? Is it a corrected value or not? Subjective parameters such as aroma and flavour are particularly difficult and the goodwill and co-operation of the supplier is important. ‘Matches previously accepted delivery’ can lead to a gradual drift in quality and ‘Matches original sample’ relates to an ageing and deteriorating specimen.

(v) It must be stipulated that no changes to the material or its manufacturing process may be made without prior notification.

13.4.4 Supplier performance

A specification agreed with a supplier is not a guarantee that all deliveries will fully comply with it and arrive in full and on time. A Certificate of Analysis adds confidence and if the supplier is accredited to the ISO 9000 series or a national equivalent it shows that the quality systems have been independently audited and found to be acceptable. However, there is no substitute for regular performance reviews, backed up as appropriate by audits of the manufacturing facilities and quality systems (including incoming raw materials). This is a very onerous and time-consuming exercise, well beyond the resources of most soft drink manufacturers.

A Food Safety Act infringement caused by a substandard ingredient can be countered with a defence of ‘due diligence’. This requires the defendant to establish that ‘all reasonable checks’ were made to ensure the ingredient was satisfactory and capable of performing as required. Case history is limited but the operative word for small companies must be ‘reasonable’.

13.5 Sports drinks

In this section sports drinks are defined, the bodily need for them is discussed and aspects of their formulation are considered.

13.5.1 Definition and purpose

Sports drinks serve to provide water, energy and electrolytes in a palatable and readily assimilable form, suitable for consumption before, during and after sporting and other strenuous physical activities. Some brands also contain vitamins. Although designed and marketed for those engaging in strenuous activity, sports drinks are often consumed by those merely aspiring to such activities or who just enjoy watching them.

13.5.2 Physiological needs

To perform physical work involves the expenditure of energy and leads to loss of water and electrolytes in the form of sweat. The progressive depletion of energy reserves and the loss of water both accelerate the inevitable onset of fatigue.

13.5.2.1 Energy

To provide energy, the body metabolises predominantly fat and carbohydrate. As the rate of work increases, carbohydrate metabolism plays an increasingly important role and by the time the body is operating at 85% of its maximum volumetric oxygen uptake (85% $VO_{2\ max}$) virtually all energy used derives from carbohydrates; either muscle or liver glycogen, or directly from blood sugars. Thus for sustained sporting activities energy must be provided in the form of carbohydrates. Many papers have been published on the relative merits of different carbohydrates but apart from considerations of sweetness and molecular weight (hence osmotic contribution) there appears to be little to choose between any of the commercially available sugars. The exception is fructose, which, being metabolised in the liver, is not rapidly available for utilisation.

13.5.2.2 Water

In a sedentary state body temperature can be regulated by varying the amount of clothing worn or by adjusting the surrounding temperature. However, these mechanisms are inadequate when strenuous work is undertaken and temperature regulation must then be achieved by the evaporation of sweat from the surface of the skin.

Water loss by sweating is influenced by:

- (i) the rate of work: an adult generates 70 W when sedentary, but 1.1 KW when running a marathon in 2 h 30 min.
- (ii) the duration of the work
- (iii) the external temperature and humidity.

Sweat loss of up to 2 l/h is common, rising to 3 l/h in hot and humid conditions. Loss of significant amounts of water reduces plasma volume. This impairs the delivery of blood to muscles and skin, eventually leading to loss of temperature control and heat exhaustion. When dehydration reaches 2% of body weight performance is impaired; at 5% work capacity falls by about 30% (Saltin and Costill, 1988). Water is also lost in respiration but this is of less significance than sweating during strenuous activity.

13.5.2.3 Electrolytes

Sweat is not just water but a dilute solution of electrolytes, mainly sodium and chloride. The actual composition varies from individual to individual and within individuals, according to circumstances. Typical values are shown in Table 13.6, from which it can be seen that sweat is considerably less concentrated than plasma. Hence sweating causes an increase in plasma electrolyte concentrations. Only in events of long duration (more than 3 h) is it considered essential to replace lost sodium during the event to guard against hyponatraemia (low plasma sodium concentration) (Gisolfi and Duchman, 1992). However, sports drinks conventionally contain added sodium, chloride and other electrolytes at levels similar to those found in sweat.

Table 13.6 Electrolytes in sweat, plasma and muscle (mmol/l)

	Sweat	Plasma	Muscle
Sodium	40–60	140	9
Chloride	30–50	101	9
Potassium	4–5	4	162
Magnesium	1.5–5	1.5	31

(Costill and Miller, 1980)

The key function of sodium is its role in assisting the absorption of glucose and water from the small intestine, both by complex formation (Schultz and Curran, 1970) and by a phenomenon known as ‘solution drag’ (Fordtran, 1975). Sodium also aids post-activity recovery by:

(i) raising plasma osmolality: this reduces urine output so enabling the body to retain more ingested water
(ii) reducing the thirst-quenching capability of water, thus enabling more to be ingested.

13.5.2.4 Vitamins

Thiamin is proportionately linked with carbohydrate metabolism and the ‘antioxidant’ vitamins A, C and E can aid the elimination of free radicals formed within muscle at an increased rate during strenuous activity (Davies *et al.*, 1982). Vitamins can thus be of value during post-activity recovery.

13.5.3 The absorption of drinks

Once swallowed, a drink passes via the oesophagus to the stomach. From there it is released to the small intestine and only then can its components be absorbed and utilised.

13.5.3.1 Gastric emptying

This is the transfer of the stomach contents to the intestine, which is a controlled process influenced by several factors:

(i) the volume of fluid in the stomach – the greater its contents, the faster it will be emptied. Carbon dioxide liberated from carbonated drinks gives additional fluid and hence faster emptying.
(ii) the concentration of carbohydrate in the fluid – any carbohydrate content will slow emptying but the effect does not become noticeable until a concentration of about 40 g/l (Vist and Maughan, 1995). It should be noted that the slower release of a more concentrated solution can still deliver carbohydrate to the intestine at a faster rate than is achieved by the more rapid emptying of a weaker solution.
(iii) the osmolality of the fluid – although conflicting evidence has been published it appears on balance that increasing osmolality delays emptying, though the effect may only be slight at the concentration of typical sports drinks.
(iv) strenuous activity – emptying is slowed when exercise levels exceed a rate of about 70% $VO_{2\,max}$ (Fordtran and Saltin, 1967).

Optimised gastric emptying would thus seem to be achieved by the regular drinking of large volumes of highly carbonated water or dilute carbohydrate solution – not a comfortable regime during strenuous activity.

13.5.3.2 Absorption from the intestine

Plain water crosses the walls of the intestine by osmotic action alone. However, if sodium and sugars are also present then the active transport mechanisms described earlier operate and water absorption can be enhanced. The effect is dependant on concentrations: the maximum rate of water uptake occurs when the concentrations give a slightly hypotonic solution (200–250 mOsm/kg, cf. 287 mOsm/kg for isotonicity) (Wapnir and Lifshitz, 1985). Conversely, when the lumen contents are significantly hypertonic, water is secreted from plasma into the intestine by osmotic action; this is a dehydrating effect.

The type of sugar seems to have relatively little effect on absorption rate, with the exception of fructose. This is not actively absorbed and nor does it benefit from the presence of sodium.

13.5.3.3 Consumption pattern

Many regimes for ingesting water, electrolytes and carbohydrates in association with strenuous activity have been published (see for example, Gisolfi and Duchman, 1992 and Olsson and Saltin, 1971). In general, carbohydrate and electrolyte supplementation are not considered necessary during the activity until the duration exceeds about an hour but in longer events and to aid post-activity recovery such supplementation plays a key role.

13.5.4 Formulation

13.5.4.1 Preliminary stage

Before detailed formulation commences it is necessary to define the target end product broadly, particularly with respect to:

(i) carbohydrate system
(ii) electrolyte contents
(iii) osmolality.

(i) Carbohydrate system. First, a quantitative decision must be made, balancing energy content against the inhibition of gastric emptying at higher concentrations. Typically, sports drinks contain in total 60–80 g of carbohydrate per litre.

Next it must be decided which carbohydrate or carbohydrates will be used. Key factors are molecular weight (and hence contribution to osmolality) and electrolyte content.

Commercially available carbohydrates, all contributing 4 kcal/g dry basis, include:

Glucose: Low molecular weight (MW) of 180, hence a major impact on osmolality. Low electrolyte content.

Fructose: MW 180 as for glucose, not rapidly absorbed from intestine nor quickly available for muscle utilisation.

Sucrose: MW 342, so lower osmolality contribution per gram than the monosaccharides. Negligible electrolytes. Inverts to glucose and fructose in acidic solution, effectively halving MW and giving a rise in osmolality.

Glucose syrup: a complex mixture of sugars produced by the hydrolysis of starch. Available in various grades of dextrose equivalence (DE), typically varying from 42 to 95. As DE increases, the average MW reduces and sweetness increases (average MW at 42 DE is 412, and at 95 DE is 186). Usually a moderate electrolyte content.

High fructose corn syrup: made by the partial enzyme conversion of glucose to fructose in a high DE glucose syrup. The grade widely available in Europe contains, on a dry basis, 42% fructose and 52% glucose, with remaining sugars present as higher saccharides. MW similar to 95 DE glucose syrup. Sweeter than glucose syrup and generally a low electrolyte content. The presence of fructose is not advantageous but low levels of this syrup give no more fructose than partly inverted sucrose.

Maltodextrins: produced by the partial hydrolysis of starch. They vary from 15 to 30 DE and have a very high average MW (typically 1100 for 15 DE). High electrolyte content, virtually no sweetness.

By blending from the above a very wide range of osmolality contributions can be achieved for any given carbohydrate content.

(ii) Electrolyte content. Although only sodium plays an active role in the absorption of water and carbohydrate, sports drinks are typically also fortified with potassium, magnesium, calcium and chloride. Levels usually approximate to those found in sweat (Table 13.6).

The electrolyte contributions of the production water and carbohydrate must first be deducted, together with those from any known additives (such as sodium benzoate or citrate, acesulfame-K or potassium sorbate). Salt additions can then be calculated to achieve the target values. Suitable additives include:

- sodium: chloride, citrate
- potassium: chloride, citrate, phosphates
- calcium: chloride, lactate
- magnesium: chloride, sulphate

(iii) Osmolality. The majority of sports drinks are formulated to be isotonic, i.e. to have an osmotic pressure matching that of blood serum. This is considered to optimise absorption from the intestine although, as stated earlier, water uptake is optimal with slightly hypotonic solutions.

Blood serum osmolality varies slightly within the individual, and from person to person, but is generally taken to be 287 mOsm/kg.

The osmotic pressure of a solution is a colligative property, i.e. it is proportional to the number of solute particles present in the solution, rather than to their weight. It is usually determined by measuring another colligative property, the depression of freezing point relative to water.

For all non-ionic substances a molar solution will depress freezing point by the same amount (1.86 °C), but for ionic substances the depression of a molar solution will be 1.86 multiplied by the number of ionic components generated per molecule.

Sports drinks are complex solutions of non-ionic and ionic substances; the latter will be dissociated to varying extents according to their nature and the other solutes present. Their osmolality cannot therefore be precisely calculated; theoretical estimates must be checked by measurement and their compositions must be fine-tuned to achieve the target value.

13.5.4.2 Palatability

It is crucial that the consumer finds the product pleasant and comfortable to drink, both during and after heavy physical activity. Sipping small amounts in a laboratory during a day of sedentary work cannot give a valid appraisal and field testing with athletes is essential. Particular aspects needing attention are carbonation, sweetness, saltiness, acidity and flavour intensity.

13.5.4.3 Prototype development

Having outlined the product composition it is useful to calculate the approximate osmolality from published tables, for example in the *CRC Handbook of Chemistry and Physics* (CRC Press Inc.), *The Merck Index* (Merck & Co. Inc.) and *The British Pharmacopoeia* (HMSO). Ingredients for which osmolality values are not available can be allowed for by using values for substances of similar molecular weight and ionic character.

The approximation will show if major adjustments are necessary prior to bench work; the value at this stage should be about 80% of the target level. A reduction in osmolality can be achieved by substituting low MW sugars with a maltodextrin or by reducing the total carbohydrate content. Similarly, an increase can be achieved by the opposite moves.

A first prototype can then be put together with the decided carbohydrate system and with salts added to approximate to the target electrolyte levels. Sweetness must be adjusted, probably by adding non-nutritive sweetener(s), an acidity level must be selected and flavour, colour and preservative must be added at appropriate levels. If the drink is to be carbonated a level must be chosen (a low level is preferable).

All the adjustments will contribute to the osmolality, which should be measured to assess what further fine-tuning will be required. It is important to measure the osmolality of the drink as presented to the small intestine, by which time carbonation will have been virtually eliminated.

13.5.4.4 Final formulation

It is necessary to undertake a series of adjustments to achieve the target composition. If the osmolality has to be increased by replacing maltodextrin with monosaccharides then additional salts will be required to maintain electrolyte levels; these in turn will increase the osmolality and require a further reduction in maltodextrin. Table 13.7 indicates how the components of a typical isotonic sports drink contribute to its osmolality.

Table 13.7 Osmolality of a typical isotonic sports drink

	% of total osmolality
Carbohydrate system	70
Salts	10
Juice/flavour/acid/sweeteners	15
Carbonation	5
	100

13.5.4.5 Powdered sports drinks

It is straightforward to prepare sports drinks in powder form for dissolving at point of use. Isotonicity will depend on the accuracy with which the instructions for use are followed and electrolyte content will vary with the composition of the water used.

13.6 Niche drinks

This section examines various beverage types outside the mainstream of soft drinks.

13.6.1 Alcoholic type drinks

These are drinks with a low alcohol content, designed to mimic stronger versions. (Note: the currently popular and highly successful 'alcopops' are, because of their high alcohol content, excluded from consideration as soft drinks.) The first drink of this type in the UK was probably ginger beer. This was introduced in the early nineteenth century, with a strength of about 5–8% ABV (alcohol by volume), and was made by the fermentation of sugar solution containing ginger root. In 1885 it was regulated by the Customs and Inland Revenue Act to an alcohol content of less than 2% proof spirit (1.14% ABV). To meet the limit, fermentation had to be arrested when, hopefully, the alcohol content was still below 2% proof spirit.

Though still a popular soft drink, ginger beer is now produced from compounds manufactured by the flavour industry, most of which are made by direct extraction rather than fermentation. As such it is no longer really within this category.

The only alcoholic-type soft drink currently on the UK market is lemonade shandy, which simulates the traditional pub blend of 50/50 lemonade and beer (usually bitter). This was introduced as a soft drink in the early 1960s and, in order to be sold in unlicensed premises, had to be below 2% proof spirit. The Food Labelling Regulations 1984 added a minimum alcohol content of 1.5% proof spirit, but both limits were replaced by a new maximum of 0.5% ABV imposed by the Licensing (Low Alcohol Drinks) Act 1990.

Lemonade shandy is made with 'shandy ale', a bitter beer brewed to 6.5% ABV to minimise transport costs. For colouring the product it is important to use an ammonia caramel as the sulphite ammonia caramels used for conventional soft drinks will react with tannins in the beer and precipitate out.

Even at this low alcohol level microbiological spoilage is a hazard. Suggested manufacturing processes are benzoic acid preservation with either sterile filtration or flash-pasteurisation of the syrup, or in-pack pasteurisation.

Ginger beer, shandy, lager and lime, cider shandy, rum and cola and also apple drinks with a cider content of 5–10% have also been produced in low-alcohol versions but are now no longer marketed.

13.6.2 Energy drinks

These products aim primarily to provide a boost to mental energy or 'buzz'. They are marketed as 'pick-me-ups', and command a high profit margin. An impressive list of ingredients is essential for credibility and typical components include caffeine, taurine, glucuronolactone, inositol, maltodextrin, vitamins and herbal extracts such as guarana, ginseng and schizandra.

Claims made for these products must be carefully worded. If they are specific to the product they must be capable of substantiation and if they refer to benefits conferred by the ingredients then there must be sound scientific evidence available in support. Exactly what evidence is required is not laid down in law so it is left to the manufacturer to decide if a claim can be justified if challenged.

In UK regulatory terms 'energy' is always taken to mean nutritional energy. Arguably, therefore, to justify the description 'energy drink' there should be a higher calorie content in the drink than in a standard one. Low energy drinks are defined and must contain at least 25% fewer calories than the corresponding standard drink. A logical extension of this would be that energy drinks, not presently defined, should contain at least 25% more calories than a standard drink.

The additional calories to achieve this level are often added as maltodextrins; their low sweetness does not make the drink cloying.

13.6.3 Functional drinks or nutraceuticals

All food and drink has a function, providing nutrition, and should also give pleasure. Increased awareness of the additional health benefits conferred by some ingredients has allowed these properties to be exploited in the form of functional drinks, also known as nutraceuticals. These are drinks that claim to have health-giving properties and they originated in Japan in 1988. In the UK they must avoid any medicinal claims in order to be classified as foodstuffs. Such claims are specifically excluded by the UK Food Labelling Regulations 1996, which prohibit 'a claim that a food has the property of preventing, treating or curing a human disease or any reference to such a property'. As for energy drinks, any claim that is made must be capable of substantiation.

Functional drinks continue to be very successful in Japan and the major ingredients exploited there are fibre, calcium, iron and oligosaccharides. Label text refers to beneficial effects on the digestive system, bones, teeth and other parts of the body as appropriate. In Europe these drinks are only beginning to be developed. Examples that are available in the UK include:

– *ACE drinks*, containing beta-carotene (vitamin A precursor) and vitamins C and E. These materials are antioxidants and there is evidence that eliminating free radicals in the body will protect against cancer and cardiovascular diseases, particularly in older people. Suggested levels to achieve beneficial effects are, per day, 6–20 mg beta-carotene, 100–150 mg vitamin C and 60–100 mg vitamin E.

– *Fibre drinks*, containing both soluble and insoluble fibres (non-starch polysaccharides). These drinks add bulk to the diet and increase stool weight (a low stool weight is associated with an increased risk of bowel cancer and gall stones). Fibre also binds cholesterol (a risk factor for heart disease), thus reducing its adsorption from the intestine. To support a claim, the quantity of drink consumed in a day must provide at least 3 g of fibre, i.e. one-sixth of the recommended daily allowance of 18 g.

13.6.4 Powder drinks

Powder drinks form an insignificant part of the UK market but have substantial sales in the USA. Their formulation closely mirrors that of liquid soft drinks, with the exception of water content. The components that do differ are:

– flavouring and clouding emulsions, which, after conventional preparation, are spray dried
– fruit materials, which are spray or freeze dried.

13.6.4.1 Manufacture

Production must be carried out in an area of controlled (low) humidity. To prevent separation after blending all components should be as similar as possible in particle size and bulk density. The ingredients must be sieved before addition and it is often preferable to prepare a pre-blend of some ingredients to facilitate the addition of small mass items and mixing in. Mixing is carried out in ribbon blenders and can be on either a batch or a continuous basis. Packaging is in sachets or jars.

Long-term stability is better than for liquid drinks and powder drinks are ideal for vitamin fortification because of their slow decay rate in the absence of water and without ingress of air.

Effervescence can be achieved by the incorporation of sodium bicarbonate, but care needs to be taken to avoid saltiness.

Normal dose rates are about 100–120 g of powder to make a litre of sugar sweetened drink and as little as 10–20 g for low-calorie drinks and unsweetened drinks where the consumer adds sugar to taste.

13.7 Dispensed soft drinks and juices

13.7.1 Introduction

Dispensing systems are used in licensed and catering outlets as a convenient, rapid and economical way of serving ready-chilled drinks without the need to handle bottles or cans.

There are two basic systems, pre-mix and post-mix:

- pre-mix: the outlet receives bulk stainless steel containers (tanks) of carbonated soft drink (usually 18.75 l/5 US gallons). The drink is dispensed by CO_2 top pressure via a chiller.
- post-mix: the outlet receives a concentrated syrup in non-returnable packaging, usually a 10 l 'bag-in-box' (BiB). The syrup is diluted with chilled carbonated water at the point of dispense.

13.7.2 Pre-mix and post-mix compared

The two systems offer different benefits.

Pre-mix: control of product quality remains with the manufacturer and all the water used is treated
- requires cheap and simple equipment at the outlet
- gives typically 94 × 20 cl servings per tank
- distribution economies are relatively small
- containers are returnable and washing for re-use needs care to ensure valves are cleaned and do not leak.

Post-mix: relies on the quality of water at the outlet
- product composition depends on the settings of the dispensing equipment
- requires a carbonator as well as a chiller at the outlet
- gives typically 320 × 20 cl servings per 10 l syrup bag
- distribution economies are substantial
- uses non-returnable packaging and for 'bag-in-box' systems packaging stocks take very little storage space
- requires a simplified manufacturing process at the factory
- aseptic packaging of syrups is an option.

Pre-mix systems are best suited for manufacturers operating in small distribution areas and for outlets with relatively low throughputs.

In the UK, where currently two manufacturers dominate the industry, virtually all dispensing is by the post-mix system.

13.7.3 Equipment

A typical post-mix unit is shown schematically in Figure 13.3. Water is supplied via a pressure reducer and non-return valves to a carbonator. If the water quality requires it, particulates can be removed by fitting a cartridge filter and chlorine can be removed with a carbon filter. The carbonator is pressurised to a regulated level with carbon dioxide from a gas cylinder.

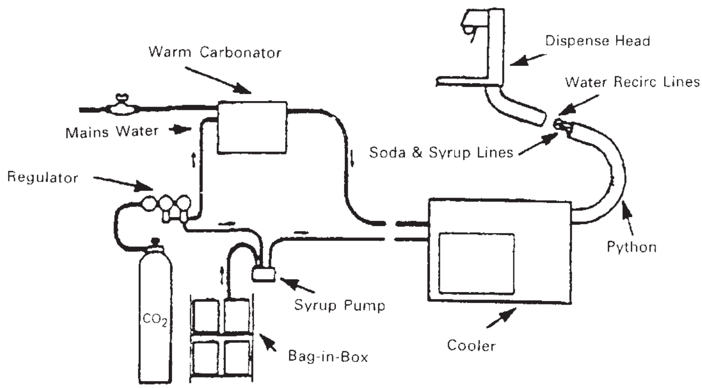

Figure 13.3 Post-mix unit.

After carbonation at ambient temperature the carbonated water passes through a cooling coil in a cooler and on to the dispense head, where the dispensing valves are housed. The cooler is a tank of water with refrigerated walls on which a layer of ice (the 'ice bank') accumulates. This ice allows a reserve of cooling capacity to be built up during less busy periods.

Syrup is pumped from a BiB container or syrup reservoir by a pressure activated pump, through another cooling coil in the cooler and on to one of the dispense valves. When the valve is opened to dispense product, pressure in the syrup line drops and the pump is activated until the valve shuts. The ratio of syrup to carbonated water is adjusted by flow restrictors in the feed pipes to the valve.

The cooler is connected to the dispense head by a 'python'. This is an insulated bundle of tubes, one feed tube for each syrup and a loop line of chilled carbonated water, continuously circulating from the cooler, round the dispense head and back to the cooler via the carbonator. The circulation is powered by a lightweight plastic pump when water is not being drawn off and it is carried out to ensure that cold drink can be delivered instantly, even when the dispense head is at a considerable distance from the cooler.

Carbonated water is teed-off to each dispense valve and when the valve is opened it feeds annularly round a central syrup flow (Figure 13.4). Mixing occurs as the liquids leave the valve.

An alternative system is to operate with cold carbonation, in which case one or sometimes two carbonators are located within the cooler water bath. This system is able to operate at a much lower carbonation pressure (typically 2.7 bar compared with 5.4 bar for an ambient carbonator).

Cold carbonation gives a more compact installation and is generally the preferred choice for large installations.

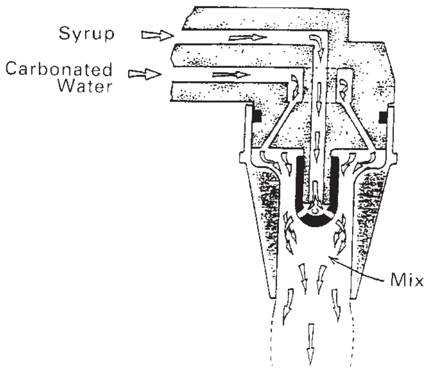

Figure 13.4 Post-mix dispensing valve.

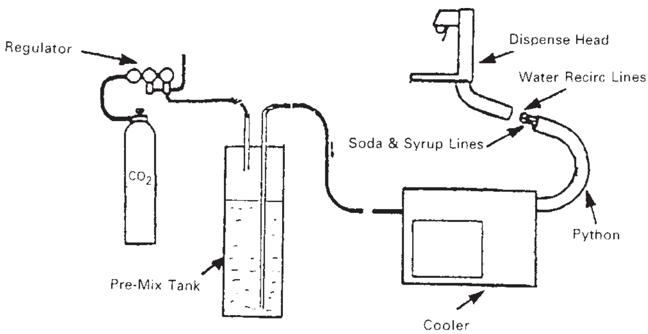

Figure 13.5 Pre-mix unit.

Figure 13.5 shows the much simpler equipment needed for pre-mix dispensing. Top pressure is applied from a carbon dioxide cylinder to a pre-mix tank, factory filled with carbonated drink. When the dispense valve is opened the pressure delivers product from the base of the tank via the dip tube through the cooler to the valve. Product in the python feed to the valve is kept cold by the pumped circulation of chilled water from the cooler bath.

13.7.4 Outlets

There are several important considerations for outlets:

(i) water quality (relevant for post-mix only): the dispenser must be plumbed in to the mains water supply, rather than be fed from a storage tank. It is essential to guard against carbonated water being forced back into the water main should water pressure drop. This is usually achieved by fitting two non-return valves in series.

(ii) sales volume: the rate of sale of drinks influences the sizing of the chiller and must exceed a critical level to justify making an installation.

(iii) distance between chiller and dispense head: the rate of heat gain in the python is obviously proportional to its length so a larger chilling capacity is required when distances are long.

(iv) large new outlets: dispensing arrangements should be planned from an early stage to allow concealed pipe runs and proper planning of locations.

13.7.5 Hygiene

A rigorous hygiene programme must be operated at the outlet with daily cleaning of the dispensing valves and drip trays, and of the disconnects when syrup containers are changed. Periodic sanitation of syrup lines is required. This must be done more often when fruit drinks or juices are dispensed.

Hygiene training must be given by the soft drinks company, backed up with simple instruction charts fixed near the equipment.

13.7.6 Post-mix syrup formulation

With certain exceptions the same formulation can be used for a dispensed drink as is used when it is bottled or canned. The key differences are:

(i) *Pulp*. No matter how fine it is, any pulp in a carbonated post-mix syrup will inevitably lead to a blockage in the system, for it will rise to the top in the BiB and then enter the valve in a concentrated form. Accordingly fruit drinks must be reformulated for dispensing by replacing conventional concentrated juices and comminutes with clarified concentrated juice. Probably a fortification of the flavour system will be necessary to compensate for the loss of character associated with clarification.

(ii) *Preservation*. The dispensed product will have a shelf life of perhaps 20 min at most so the syrup need only contain sufficient preservative to maintain its own microbiological integrity.

(iii) *Dispensing accuracy*. Typically a dispenser will be capable of producing drinks with an accuracy of ±5% for both water and syrup supplies. When calculating ingredient additions the possibility that these tolerances will operate in opposing directions should be taken into consideration as well as the syrup tolerance.

Example
Formulate a post-mix syrup with a ratio of 1 to 5.4 for a product containing the maximum amount of saccharin to meet a legal limit of 80 mg/l.

Nominal syrup ratio is 1 volume syrup plus 5.4 volumes water (15.625% syrup). Maximum concentration occurs when syrup quantity is +5% of target and water quantity is −5% of target, i.e. maximum drink composition is 1.05 volumes syrup plus 5.13 volumes water (16.99% syrup). Thus to ensure a maximum saccharin content of 80 mg/l in the drink, the syrup must contain a maximum of (80 × 100)/16.99 mg/l of saccharin, i.e. = 470.9 mg/l.

Assuming the syrup is also manufactured to an accuracy of ±5%, then the target saccharin concentration must be (470.9 × 100)/105 mg/l of saccharin, i.e. 448.4 mg/l.

At a target ratio of 1 to 5.4, 448 mg saccharin per litre in the syrup would give 70 mg/l in the drink.

Conclusion:

	mg saccharin/litre
post-mix syrup	448 ± 22 (±5%)
dispensed drink	70 ± 10 (±14%)

(iv) *Carbonation*. This will be effectively identical for all heads on any dispenser and is conventionally set at 3.5 volumes. This must be kept in mind when formulating dispensed drinks, which are usually carbonated to a lower level (e.g. orange drink, normally about 2.5 volumes).

(v) *Viscosity*. Syrup viscosity influences its flow rate through a dispensing valve so if a formulation change significantly alters viscosity then all dispensers will have to be reset.

13.7.7 Post-mix syrup packaging

Modern systems use BiB packaging for syrups. A double bag is used: the inner bag is blended polymers (for example LDPE and EVA) and the outer protective bag is a laminate with an inner layer of metallised polyester sandwiched between LDPE. The bags are sealed with a coupling valve designed to connect only to the drink manufacturer's dispensing equipment and after filling are dropped into cardboard outer boxes.

Earlier systems used syrup packed into 5 l HDPE jars, which served to fill reservoirs coupled to the dispenser, giving ample opportunity for contamination, dilution or for use of syrups from other manufacturers ('pirating').

13.7.7.1 Post-mix syrup production

Syrup manufacture follows the same procedure as bottling or canning syrups, after which it is packed off.

13.7.7.2 Product quality at the outlet
There are four key criteria:

(i) concentration: measured either by a ‘Brixing cup’, which checks volumetrically the volumes of syrup and water delivered by a dispensing valve, or by hand refractometer. In the latter case the refractometric solids of the actual syrup being used should first be measured so that allowance can be made for the syrup inversion that will have occurred since manufacture.
(ii) taste and flavour: to check the cleanliness of the system and filters.
(iii) carbonation: measured by collecting dispensed product in a container with a sealable lid incorporating a pressure gauge.
(iv) temperature: normally targeted at 4.5°C, measured in a glass pre-chilled by filling and emptying before taking the test drink.

13.7.7.3 Uncarbonated systems

(i) bowl dispensers: these are very simple devices for dispensing diluted squashes. A perspex bowl fitted with a chilled base and a paddle stirrer is filled with squash and water, and product is dispensed from a tap. Usually the squash is purpose-made for catering outlets and is double normal strength (i.e. for dilution at 1 + 9 rather than 1 + 4).
(ii) juice dispensers: these operate on a post-mix basis, using as a ‘syrup’ aseptically filled concentrated juice (diluted to a suitable viscosity). Sometimes a low level of sulphur dioxide is added to protect juice in the dispense valve, which for citrus juices must be modified to cope with high pulp content.

One or more valves on a carbonate dispenser can be modified to handle juice, replacing carbonated water with chilled water from a separate cooling coil in the chiller.

Alternatively, a purpose-built dispenser can be used. One counter-top system stores a BiB container of concentrate in a refrigerated compartment above a water chiller. Concentrate is transferred directly to the dispense valve by a peristaltic pump acting on a tube that is replaced with every BiB. This ensures that the only permanent part of the equipment contacting the juice is the valve.

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