Whisky

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Contributors

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His post-graduate life started in packaging research and development, and contract research. He has over 20 years experience with Diageo and its predecessors, working on process, quality and analytical aspects of distilled spirits. Currently active in risk management and brand protection, particularly the brand and generic authenticity of Scotch whisky, gin and vodka. Experienced extensive overseas travel to support spirits producers and anti-counterfeit programmes.

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Had an early career as a research and development scientist with Tuborg Brewery in Copenhagen, Guinness Brewery in Dublin and the Brewing Research Foundation at Nutfield. He became Technical Manager of Moray Firth Maltings and Director of Production for Malting and Malt Distilling in United Distillers plc and recently retired after 35 years in the industry.

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His first degree was in Pharmacy and after obtaining a PhD he carried out postdoctoral research at the Department of Bioscience and Biotechnology, University of Strathclyde. This was on projects jointly funded by the Government (BBSRC) and industry (Chivas Brothers Ltd) on flavour changes in whisky during maturation which resulted in the development of a clearer understanding of ways in which the whisky matrix can enhance or suppress the release of aroma compounds. He is currently a research scientist with the Scotch Whisky Research Institute and is working on Maturation and Product Integrity related projects.

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Has over 20 years commercial experience spent working in a variety of senior management roles at William Grant & Sons, specializing in the international marketing of Scotch whisky brands. He was closely involved in the early development of single malt Scotch whisky, largely pioneered by Glenfiddich (the world's leading brand), owned by the fifth generation family firm William Grant & Sons. He is Director General, Institute for Family Business (UK); Deputy Chairman, William Grant & Sons Ltd and Non-Executive Member of the Board, Laurent-Perrier S.A. His education includes Harvard Business School (OPM30), Kellogg Graduate School of Management (MBA), Northwestern University and HEC Lausanne University, Switzerland

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Has been Environmental Manager with Diageo (Scotland) Ltd since 1990. His early career was in the heavy chemicals industry, followed by 21 years in several roles in the field of water pollution control; both as regulator and regulated.

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Has extensive experience in the alcohol industry including: Analytical Chemist at Hiram Walker & Sons (Scotland) Ltd; Manager at the Tormore Distillery, Speyside; General Manager at the Laphroaig Distillery, Islay; Project Scientist at Long John International, Laboratory Manager at James Burrough Distillers Ltd; Laboratory Services Manager at Allied Distillers Ltd and Technical Manager at Demerara Distillers Ltd, Guyana, South America. In addition he is a teacher of Chemistry, Physics and Science.

Bob Pass BSc
Has been involved in both the technical and marketing aspects of Distillers Feeds for 25 years. He is currently the Marketing and Technical Manager, Animal Feeds for Diageo, the world's largest producer of alcoholic drinks. Before joining Diageo he was with Pentlands Scotch Whisky Research, a research organization funded by the whisky industry where he was head of the by-products section. He had also previously worked with BP Proteins on the technical marketing of yeast biomass protein (cultured aerobically on normal paraffins) and with Dow Chemical.

Ken Reid PhD
Has 16 years experience in Scotch whisky research, with his work focused mainly on whisky maturation chemistry. He graduated from Heriot-Watt University, Edinburgh with a degree in chemistry and in 1989 he gained his PhD (on the subject of whisky maturation chemistry), whilst based at Reading Scientific Services Limited, Reading, England. He then joined the Scotch Whisky Research Institute where he is now the Research Manager in charge of the areas of Maturation and Product Protection.

Foreword

During the past three decades I have been fortunate enough to have filled several technical roles in the Scotch whisky industry, at company level (in a very small and later in a very large company) and at national industry level, within both the Malt Distillers' and Scotch Whisky Associations. In all of these roles, and more recently as Visiting Professor at the International Centre for Brewing and Distilling (ICBD), I have been asked by students, new entrants and experienced colleagues, whether I could recommend a good textbook on the technology of whiskies and the associated distilling processes.

In most technical and scientific disciplines, including the closely related subjects of brewing and winemaking, this would not have been a difficult question to answer and indeed a choice of admirable titles could be mentioned in these fields, depending upon the needs and background of the enquirer.

On the subject of whiskies – especially Scotch whisky – many volumes have been published on the heritage and geography of distilleries and on the variety and tastes of their products. Indeed, the enthusiastic consumer can choose from several shelves of such books, and every year sees at least one or two worthy additions to this bibliography. However, for the serious scientific student seeking to extend or update their knowledge across the breadth of whisky technologies, there is a dearth of suitable textbooks – indeed only one or two have appeared in the past century and none at all, to my knowledge, in the past decade. Yet the technical knowledge of distilling (and its related disciplines) continues to be advanced steadily by a number of eminent researchers, and a regular stream of scientific papers continues to be published in our specialist journals.

Therefore, I am delighted that early in the new millennium we can welcome this much needed volume which updates our scientific knowledge across the whole field of whisky distillation and fills an acknowledged gap on the shelves of our technical libraries.

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Preface

There are surprisingly few technical books available that discuss distilled beverages. This book focuses specifically on whisk(e)y and allows the reader to delve beyond the art of the beverage, into the science and technology behind one of the world's most loved drinks.

In this first book in the Handbook of Alcoholic Beverages series, highly respected experts from academia and industry provide a unique perspective into a production process that has traditionally been cloaked in secrecy. Most publications have treated the production process as more art than science, despite the high quality of research and development, and quality assurance/quality control[**AQ] that the industry invests in and prides itself on.

The history of the development of whisky distillation, starting with its monastic roots in Ireland and Scotland, is reviewed in the first chapter by George Bathgate, retired Director of Production for Malting and Malt Distilling (United Distillers Plc). In the next chapter, Tim Dolan who has extensive experience in malting and distilling (ABM malting and The Highland Distilleries) examines malt whiskies in terms of raw materials and processing.

In the third chapter, three authors from the Scotch Whisky Institute: Tom Bringhurst, Anne Fotheringham (Broadhead) and James Brosnan discuss grain whisky in terms of raw materials and processing. Iain Campbell, who has lectured and carried out research for many years at the International Centre for Brewing and Distilling, Heriot Watt University, comprehensively covers the topics of yeast and fermentation as well as grain whisky distillation. Denis Nicol, who has extensive expertise working with a number of distilleries over the years, explains batch distillation. Maturation and blending are covered by three experts from the Scotch Whisky Research Institute: John Conner, Ken Reid and Frances Jack. The Marketing and Technical Manager – Animal Feeds, (United Distillers and Vintners) Robert Pass and I. Lambert (also from UDV) discuss co-products in terms of volumes produced, markets, and nutritional characteristics. This is an area where today there is great interest both from an environmental and economic point of view . The intricacies of whisky analysis are discussed by Ross Aylott (Diageo) who is active in risk management and brand protection, particularly the brand and generic authenticity of Scotch whisky. The final chapter in the book details the marketing of Scotch whisky and is written by Grant Gordon – it covers managing brand images through to

routes to market. Grant Gordon has over 20 years commercial experience with William Grant & Sons, specializing in the international marketing of Scotch whisky brands and was closely involved in the early development of single malt Scotch whisky, largely pioneered by Glenfiddich.

One area that the book does not discuss is the variety and specific tastes of the product. There are numerous books available that discuss this area in great depth, but it would be remiss not to address the one technical question that consumers always ask . . . does one mix the whisky with water, with soda or is it best to consume it straight ?

The opinions on this are as varied as the number of products on the market and there is no doubt that the experts will never agree to one best presentation. It will vary with the individual product, the country and indeed current trends. However, from a scientific point of view, the general wisdom is that the addition of a small amount of water is key to release the aromatics. How much water and what type of water? What about soda water? That is another debate. Ice . . . even more debate!

In the end, each consumer's decision is the correct one when they serve it in the way that gives them the flavour that they most enjoy . . . and for that particular consumer that is the correct way to drink the product.

Regardless of how the drink is best enjoyed, this book will give the reader a better appreciation of the science, technology and marketing of whisky.

Inge Russell PhD DSc FIBrew
Canada

Chapter *1*

History of the development of whiskey distillation

George N. Bathgate

Introduction

Nearly all distilled alcoholic beverages have similar generic roots depending on the basic raw material, whether grape, grain or sugar. Such distillates were in the past regarded as having great medicinal, almost mystical, properties and their recipes were entrusted to the religious clerics of their day and tradition. The generic Latin name given to these distilled products was *aqua vitae* (the water of life). This is certainly true in the case of whiskey, which is a corruption of the Gaelic *uisge beatha* (again, water of life). This appellation, spelled without an 'e' in Scotland but with an 'e' in Ireland and America, is the generic name for a distilled product made from saccharified and fermented cereal extracts. In some cases this definition is even more specific, in that the saccharification process is implemented only by the enzymes of malted barley. In other traditions, similar distillates from alternative sources of fermentable sugar were translated from Latin as *l'eau de vie* in France or *aquavit* in Scandinavia and so on. All these spirits were regarded as having remarkable restorative properties.

From its monastic roots in Ireland and Scotland, the distillation of whiskey developed over several centuries into a commercial enterprise in various continents. It is now well known that the first record of a commercial transaction involving the supply of whiskey (*sic. aqua vitae)* was between the Benedictine monastery at Lindores Abbey in Fife and the Court of King James IV at Holyrood, Edinburgh in the year 1494 (Anon., 1494). The distiller in charge at Lindores must have been a Father John Cor. because the Exchequer Rolls of Scotland records the supply of 'eight bolls of malt' to the said Friar 'to make

aqua vitae'. As well as telling us that whiskey was even then consumed in the highest social circles, it also records that the whiskey was malt whiskey and that the malting of barley was probably a separate commercial enterprise.

The consumption of whiskey may have been partly for pleasure at the Royal Court of Scotland, but it was still regarded very much as a tonic. It would appear that the legitimate manufacture of whiskey for 'medicinal purposes' passed from the ecclesiastical world to the secular via the professional medical practitioners of the time, the Guild of Surgeon Barbers. For example, the City of Edinburgh granted a whiskey distilling monopoly to the Guild in 1505, and this was given Royal Approval by a Seal of Cause in 1506 (Scott-Moncrieff, 1916). From then on, not only in Scotland but also in every other country that has adopted the distillation of whiskey, a constant 'war' has been waged between state and private distillers for control of a very valuable commodity. There is no doubt that state control and, in particular, attempts to extract excise duty have played (and still play) a major role in the worldwide development of whiskey distilling.

This is not the only common impetus for the development of whiskey, and there is remarkable similarity in the development of international markets and brands. Drivers for development can be summarized as:

1. Existing technology to produce the distilled product locally
2. A national demand to consume the distilled product
3. The drive and initiative of a few strong entrepreneurs to realize emerging commercial and export marketing opportunities
4. A commitment to supply and maintain a product of consistent high quality.

How these events evolved in the five largest international whiskey distilling countries – Scotland, Ireland, the USA, Canada and Japan – is described in the following sections.

Scotch whiskey

Commercial development

Although it is commonly accepted that the art of distilling was brought to Scotland by Irish monks at the time of St Columbus and thereafter, domestic distilling of whiskey developed in parallel and was common practice by the middle of the sixteenth century. Indeed, brewing and distilling were by then regarded as staple requirements because the Scottish Parliament decreed, following a particularly bad harvest in the West of Scotland in 1555, that grain should only be used in the Burghs of Ayr, Irvine, Glasgow and Dumbarton for 'baking bread and the brewing of ale and *aqua vitae*' (Scott-Moncrieff, 1916). The said *aqua vitae* was therefore being distilled not only in farming communities but also in the burgeoning towns of Scotland.

The granting of monopolies to surgeons and apothecaries was an attempt to control whiskey distilling for use only for 'medicinal purposes', but this was

not very successful because of ever-increasing commercial pressures. It is not surprising that historical records show that some of the good burghers of Edinburgh, such as one Besse Campbell, regularly broke these cartels. The Edinburgh Town Council Records of 1556 state that Besse should 'desist and ceis fra ony forthir making of *aqua vitae* within this burgh in tyme cumyng or selling of ony therein **except on the market day**' (author's emphasis), and also 'to conform to the privelge granted to the (surgeon) barbers under the Seill of Caus' (Scott-Moncrieff, 1916). As well as showing us that the Town Clerk had some spelling difficulties, the record clearly demonstrates that while Besse was not allowed to distil whiskey, she was allowed to sell it in the town's market as a sort of licensed grocer of the period. The other important conclusion to be drawn from this record is that there was a growing market, albeit a local one, since Besse felt obliged to distil her own whiskey to augment the 'legal' supply. Besse was therefore the first recorded entrepreneur of whiskey, and the first (but not the last!) female distiller to boot.

Government intervention continued into the next century, and following the outbreak of civil war in England in 1642 the Scottish Parliament imposed a series of excise levies. So began the age-old conflict between Customs & Excise (the Gaugers) and distillers, who were increasingly driven to hide in the hills and glens and the isles of the Inner Hebrides to carry on what they regarded as their legitimate trade, free from punitive government taxes. However, not all distilling was illicit. Following the restoration of the monarchy in 1660, the tax on ale, beer and whiskey (which was still referred to as *aqua vitae* in all statutes of the period) was essentially doubled, and it was estimated that this provision would yield £384 000 in revenue (Statute 1661, Car II, c.128). To raise this huge sum there must have been several large legitimate stills in existence, such as those of John Haig & Co., who claim that a Robert Haig established their business in 1627 (Anon., 1914). What is interesting, from a technical viewpoint, is the fact that these taxes were imposed not only on malted barley but also on spirit 'not made of malt'. Other chronicles of this period similarly allude to spirit being made from a mixture of grains, such as oats, barley and wheat (Smith, 1776) as well as malt. So even from the earliest times some whiskey was being distilled from unmalted grain, and not all malt was made from barley. The malt tax introduced in 1701, for example, states that duty shall be paid: 'upon all Malt, ground or unground, whether the same shall be made of Barley, or any other Corn or Grain whatsoever' (Statute 1701, 12 &13 William III, c.5).

After the Treaty of Union with England in 1707, the first London-based Parliament of the now United Kingdom of Great Britain and Ireland decreed that Scotland should pay the same high rates of duty as England. This was in direct contradiction to the Treaty of Union, and led to the infamous Malt Tax riots of 1725 (Devine, 1999). The government backed off from such universal revolt, and it took nearly another 100 years before excise duties were harmonized throughout the United Kingdom (Smith, 1980). Nevertheless, legitimate distilling continued to develop, particularly in the Lowlands of Scotland. By 1756 duty was paid on 433 811 gallons of spirit, nearly eight times the volume taxed the year after the Treaty of Union (Craig, 1994). Despite the public

resistance to the Union and its effects on taxation, trade with England had been opened up and there was an increasing market for good quality whiskey. Following the Malt Tax riots, duty in Scotland was still only half the rate applied to English (particularly gin) distillers (Craig, 1994), and so Scottish traders could afford the additional transport costs and still make good margins on their product to the disadvantage of their English counterparts.

The story was somewhat different in the Highlands, where *uisge beatha* was historically used for family or local consumption and illicit stills numbered in the thousands (Craig, 1994). The tradition of distilling Highland whiskey in small stills, widely dispersed throughout the region, can be traced to these illegitimate roots. With ever-increasing demand from the south, smuggling of illicit Highland whiskey became highly organized and effective to the extent that, by the early nineteenth century, almost half the whiskey consumed in Scotland was duty free and, despite the hated Excise Gaugers confiscating up to 14 000 illicit stills every year, the illegal trade continued to prosper (Scotch Whiskey Association, 2002). The government had to compromise, and in 1784–1785 licensing acts were passed to allow illicit distillers to become legitimate in return for a fee and a reasonable rate of duty to be paid on fermenting wash.

Over the next twenty years the number of licensed distillers increased, but illegal distilling and smuggling still continued, despite several further compromises made to the excise laws. It was not until the Excise Act of 1823 was introduced to harmonize taxation in Scotland and Ireland that illicit distilling and smuggling were finally brought under control. Duty on distilled spirit was reduced to two shillings per proof gallon, and the separate tax on fermenting wash was abolished. A distiller could now, at long last, buy a license for £10 and develop his business without crippling taxation (Statute 1823, Geo.IV, c.94).

The rest of the nineteenth century witnessed the remarkable growth of Scotch whiskey to become the only truly international, generic brand of potable spirit. This was driven by a combination of opportunist entrepreneurs and fortuitous developments. The first technical development that assisted this phenomenal rise in exports was the differentiation of grain and malt whiskey distilling, and the subsequent blending of these two types of whiskey to produce consistent high quality products. Greater volumes of grain whiskey could also be produced following the introduction of continuous distilling in patented Coffey stills (Coffey, 1830). Although mixed grists, i.e. malted barley and unmalted grain, had been used in both Highland and Lowland traditions, the latter half of the nineteenth century saw a gradual change until Highland whiskey was exclusively single malt and Lowland grain distilleries used only a small amount of malt to saccharify cooked unmalted grain.

The other technical innovation that helped to characterize Scotch whiskey and establish its commercial growth happened more by chance than by design. During the years of secretive distilling, whiskey had to be hidden and then moved surreptitiously from its place of concealment to the market. Virtually the only storage and transportation vessels available to both illicit and legitimate distillers alike were wine, sherry and port casks that had been imported from France, Spain and Portugal. Many of these casks were pur-

loined for use in the ever-growing distilling trade after their original contents had been consumed. Even by the end of the eighteenth century, Highland whiskey had a reputation for being smoother and mellower than the fiery Lowland spirit. It was not long before astute distillers realized that the longer new-make spirit was kept in oak casks, the more it was appreciated by the consumer.

These innovations on their own helped to make Scotch whiskey unique, but they were not, *per se*, the reason for the huge expansion in whiskey distilling in the eighteenth century. Throughout the century several entrepreneurial families developed the legitimate marketing and selling of Scotch whiskey internationally. Some of these businessmen, such as John Walker, John Dewar, James Chivas, William Teacher and George Ballantine, started as small family grocers and wine merchants. They all selected good quality malts to blend with Lowland grain whiskey to sell into their local markets before venturing into exports, first to England and then increasingly to the USA and eventually worldwide. Others, such as the Haig family, William Grant and James Buchanan, had always been involved with either distilling or whiskey merchanting, the latter almost entirely in London. The drive and initiative of these great 'whiskey barons' was essential for the continued development of the Scotch whiskey industry. They were adept at seeing marketing and trading opportunities, but were also aided in some cases by a certain amount of serendipity. From around 1863 the vineyards of France had been devastated by plagues of phylloxera aphids, and by 1879 most of Europe had been affected. There were therefore great shortages of claret and cognac, the principal alcoholic beverages of the middle and upper classes in Great Britain, and an opportunity was presented for blended Scotch whiskey to capture the entire brandy market. It could be said that Scotch never looked back, and from a secure base on the home market Scotch traders were then able to venture out to capture international markets as well.

With so much trade for blended whiskey being generated by these merchanting companies, distillers, particularly the Lowland Patent-still grain distillers, were finding it difficult to regulate supply. In 1856 a one-year agreement on distilling volumes was set up and in 1865 a more formal trade association was agreed, which eventually led to the formation of the Grain Distillers Association in 1878. A similar Association for Malt Distillers was formed under the banner of the North of Scotland Malt Distillers Association, now the Malt Distillers Association of Scotland, a little earlier in 1874 (Craig, 1994). The formation of the Grain Distillers Association was contemporaneous with consolidation of several of the largest grain distilleries into the Distillers Company Ltd (DCL) in 1877 (Ross, 1923). This event brought most of the large grain distillers under one umbrella company, and from the start until its demise in 1986 DCL operated more as a consortium of individual companies than as a single production and marketing operation. Nevertheless, the formation of the DCL and the trade associations brought some much needed organization and control into the fledgling industry. Similar consolidations were to take place within the malt-distilling sector of the industry. Scottish Malt Distillers Ltd was established initially as a joint venture com-

pany, with the DCL as the major shareholder. This then acquired the majority of shares in 1925, when the company became a wholly owned subsidiary of DCL.

During the first quarter of the twentieth century, the DCL was already the largest supplier of grain and malt whiskey to the major brand companies such as Walker, Dewar and Buchanan. By this time the DCL was not only a production company but had also itself built up some thriving brands. When some of the independent brand companies realized that there were better opportunities in the marketplace by merging their operations rather than fighting each other, rationalizations rapidly followed. First Buchanan and Dewar merged in 1915, and, following the DCL's purchase of Haig, the new Buchanan–Dewar and Walker were, in 1925, both absorbed into an ever-expanding DCL. Mergers and takeovers have continued ever since, until there are now only a few major players in the global whiskey market. Following the Guinness (then owners of the Arthur Bell's Brand) takeover of the DCL in 1986, the new United Distillers finally merged with Grand-Metropolitan (owners of J&B) to form, in 1997, the world's largest distilled beverage company, Diageo plc. With Ballantine now being part of the Allied-Domeq Group, Chivas joining with the French group Pernod–Ricard, and Dewar being in the Bacardi stable, Scotch whiskey has become only part (although a very important part) of a portfolio of spirit brands being sold on a global scale by a handful of international companies. This is, of course, the trend with a whole range of consumer goods that are now marketed as international brands, and may mark the end of the consolidations that commenced in the early part of the last century. Since Scotch whiskey is still the most popular generic brand in the international spirits market, and one of the most valuable to any company focused on the alcoholic beverage sector, the industry will no doubt enjoy a more settled future in the twenty-first century.

Technical development

The earliest distillations, carried out by alchemists and apothecaries, were performed in retorts, the basic (pelican) shape of which evolved into the pot-stills that are typical of the modern Scotch malt whiskey distillery. These early types of small retorts are in fact still used in the French cognac industry.

They were invariably connected to a copper 'worm' (coil) condenser, which was immersed in a tub of cold running water (or in the case of illicit stills, in a convenient burn). These stills were also small so that they could be easily dismantled and hidden from the eyes of patrolling excise men. It was fortunate that the only malleable metal from which such apparatus could be made in these early years was copper, the partial dissolution of which is a vital ingredient of Scotch whiskey – in particular malt whiskey (Bathgate, 1998a).

However, the development of Scotch whiskey is as much dependent on the raw materials – cereals, water and yeast – as it is on the distilling technology and copper. As mentioned earlier, the earliest whiskies were predominantly

made from malted barley, but many distillers also ground unmalted wheat, barley or oats into their grists. The amount of unmalted grain that could be converted by the enzymes from the malt was no doubt estimated by empirical means, and such additions helped to reduce the overall cost of the process in much the same way as, today, most brewers use unmalted cereals in their grist. Of course this procedure ultimately led to the development of grain whiskey, made predominantly from ungerminated grain, and this will be discussed later.

From the earliest times, malting was regarded as an integral part of the process of making Scotch whiskey. Nearly everyone recognizes the structure of a Scottish malt distillery by the characteristic 'pagoda'-style roof of the malt kilns, and not the still-house. Most distilleries have retained these 'pagodas' (which were really the housings for the kiln chimneys), even when the associated maltings have been closed down.

It is therefore surprising that the development of malting is not as well recorded as that of distilling. Nevertheless, it was recognized that the quality of the barley and the way it was processed had an important effect on both the yield and quality of spirit. For example, Sir Robert Moray (1678), in relating 'the manner in which malt was made in Scotland', highlighted how the harvested barley should be kept warm and dry to break natural dormancy and to preserve germination. He also recognized the importance of barley variety, and was adamant that two-rowed barley was preferred over the coarser four-rowed cultivar 'Bere', which was grown throughout Scotland at that time. Moray went on to relate that the malting of barley was very much a farm-based process involving only very small amounts of barley. Improvements in developing land races of barley throughout the eighteenth and nineteenth centuries followed by, in the twentieth century, the development of crossbred hybrids, have continuously improved the alcohol yield. This development has been most marked in the last 50 years, when we have seen yields rise by about 20 per cent over the period (Table 1.1).

Table 1.1
Development of Scottish malted barley spirit yields

Period	Predominant varieties	Approximate spirit yield (l abs. alc./tonne)
Pre-1950	Spratt and Plumage Archer	360–370
1950–1968	Zephyr	370–380
1968–1980	Golden Promise	385–395
1980–1985	Triumph	395–405
1985–1990	Camargue	405–410
1990–2000	Chariot	410–420

Normally only surplus grain was used for distilling purposes, until, during the nineteenth century, commercial development of distilling created a higher added value for the malting of barley over its use for human and animal feed. As has already been seen, this caused great concern in times of harvest failures and local famines, when government regulations were introduced to prohibit malting. Nettleton (1893), in his famous treatise *The Manufacture of* Spirit, recorded the size of a typical Highland malting/distillery in the 1820s. A reasonable-sized operation would have produced about 6500 litres (2500 proof gallons) of absolute alcohol per annum, and it has been estimated that this would only have required about 20 tonnes of malt arising from approximately 25 acres of arable land (Duncan *et al.*, 1994).

The technology of malting did not change for several centuries, in that barley that had recovered from dormancy was steeped in cold water for several days and then spread out to germinate on a barn floor. The grain was turned manually on the floor to prevent overheating, and after a period of seven to ten days was kilned gently over a peat fire. The impartation of peat smoke onto the malt is the most important contribution of malt to the final flavour of malt whiskey, and was characteristic of all Highland and Island malts up to the second half of the last century. It is interesting to speculate that in the Lowlands, and in particular in East Lothian, where coal has been mined for over 400 years and peat has never been available, the use of coal-fired kilns imparted a lighter, unsmoked flavour to these malts. Although floor malting continued as the predominant on-site method of malt manufacture until the late 1960s, many distillers experimented with novel types of mechanical 'pneumatic' maltings and these have always been at the forefront of malting technology. For example, Speyburn–Glenlivet Distillery Ltd installed one of the first 'Galland' drum maltings in 1905. This very compact malting unit had a central belt-drive mechanism to turn the drums and the ventilation fans, a double-decked kiln for fuel efficiency, and was successfully operated for over 60 years by the DCL. Similarly, Mackenzie Bros. developed one of the first Saladin Box maltings, with novel mechanical turners, at Dalmore Distillery in 1956, and the DCL built the first fully electronically automated maltings in the UK at Roseisle, near Elgin, in 1979–1980.

The phenomenal growth of the Scotch whiskey industry after the Second World War created such a demand for malted barley that on-site floor maltings using only locally grown barley could no longer cope. Many distilleries doubled their capacity and therefore had to be supplied from large off-site maltings in the Lowlands, or even had to import malt from England. From 1965 to 1980 several on-site Saladin-box type maltings were built, as well as six large off-site maltings along the Moray Firth coast. Over 95 per cent of all malted barley is now processed off-site, with only United Distillers & Vintners Ltd (previously the DCL) having significant in-house malting capacity, the rest being supplied by independent maltsters. The other in-house capacity of any importance is that of the small floor maltings specializing in peated malt on the islands of Islay and Orkney.

These new maltings created an annual demand for over a quarter of a million tonnes of barley. Good malting varieties such as Proctor and Maris Otter had

been developed for the kinder climate of South-East England and were agronomically unsuitable for Scottish farmers, but could be transported to the Highlands of Scotland relatively cheaply following the development of a comprehensive rail network in the latter part of the nineteenth century. Wagons that brought sacked barley up-line could be conveniently back-filled with casks of mature whiskey, so making this a reasonable economic option. However, increasing transport costs and ever-increasing demand made locally grown barley an essential requirement to sustain malting operations. Fortunately, the introduction of the variety Golden Promise in the late 1960s fulfilled this demand. Golden Promise was a semi-dwarf, early maturing variety that could withstand the colder, windier conditions in the North of Scotland, and was the preferred variety for distilling malt for nearly twenty years. Since then new cultivars with higher agronomic and distilling yields (see Table 1.1) have been developed, with no one variety having dominance for more than three to five years. This is a trend that shows no sign of diminishing, with the effect that average malt distillery yields (which had averaged around 390 l alc./tonne for those twenty years between 1969 and 1989) increased in the next five years to over 405 l alc./tonne and are still rising (Bathgate, 1998b).

Distillers have been more conservative in the development of the next stage of the process of malt distilling. Historically, malt would have been ground manually and mixed with hot water using a wooden paddle and a wooden mash vessel. In all probability the dissolved malt wort would not have been separated but would have been fermented *in situ* with yeast retained from previous batches. Malt mills and mechanical mashing vessels were developed in the nineteenth century, and remained unchanged for nearly another hundred years. Since a malt grist of generally well-modified material is used, sophisticated milling and mashing techniques are not necessary and, unlike in the brewing industry, development of malt conditioning, multi-roll mills and separate mashing and wort separation technology has been deemed unnecessary. A typical set-up was to have a simple four-row mill with a deep-bed mash tun fitted with rotating mash paddles and a Steeles mash mixer.

These traditional mash tuns were originally made of cast iron or, more recently, mild steel. Since the 1970s mash tuns have been gradually replaced with stainless steel lauter tuns fitted with lauter knives that can be raised and lowered, continuous sparge systems and fully automated control systems. The traditional system required four separate additions of water to the mash tun, with each batch being filtered almost to dryness before the next addition. Lautering (or more correctly semi-lautering, since the mash is not converted in a separate vessel as in brewing) requires a continuous addition of temperature-profiled sparge water and is therefore more efficient in terms of water usage, producing more concentrated worts. The general trend to mash filtering as opposed to lautering has not been followed in malt distilleries as yet, since early experiments with less well-developed mash filters showed no advantage over traditional mash tuns or semi-lauters (Wilkin, 1983). On the other hand, the recent development of the Meura pneumatic mash filter has revolutionized this particular technology and it is expected that at least one malt distillery will have its traditional mash tun replaced with this equipment in the near future.

The history of the fermentation stage of the malt distilling process is not well documented. It can only be surmised that the earliest distillers carried forward yeast inocula from batch to batch, with all the inherent risks of bacterial and wild yeast infections. As distilling progressed into an industrial scale operation, it is well documented that distillers made use of surplus yeast from the Brewing Industry (Sharp and Watson, 1979). The development of large grain distilleries created a massive demand for yeast, and it became common practice for the larger distilleries to propagate their own strains. Specialist yeast factories then evolved from these grain distilleries (Jones, 1998), producing cultured yeast specifically for the distilling industry. The predominant strain has been the DCL's 'M' Yeast, first produced as a hybrid over 30 years ago, but other cultured yeasts are now available and are used either on their own or as a mix with surplus brewer's yeast (Sharp and Watson, 1979) Once the worts had been run off and collected at gravities between 1050 and 1060 they were traditionally fermented in open wooden wash backs, but over the last 30 years there has been a move to fermenting in closed stainless steel vessels, which are more hygienic and facilitate the control of carbon dioxide emissions.

As mentioned above, malt distillers retained traditional copper pot stills whereas grain distillers developed continuous distillation. Nevertheless, pot stills have evolved from simple, small retorts to quite sophisticated pieces of technology in their own right (Nicol, 1989). Development took place principally in the nineteenth century, when each individual malt distiller sought to differentiate his product from neighbouring competitors by varying the geometry of the swan neck and lyne arm or introducing a swan-neck purifier. With the density of distilleries in, for example Speyside, this neighbour might literally be next door. It is therefore a tribute to these developers that they were able to produce and retain such diversity of spirit characters. Once the still design and functionality had been established, however, it remained unchanged for the next hundred years, and even when stills have to be replaced it is meticulously copied, whether a wash still or a spirit still. In the first industrial operations this did not matter so much because there was only one still in which, after the first distillate was accumulated, it was re-distilled either once or twice.

It is often quoted that the only difference between Irish and Scotch whiskies is that the former is triple distilled. Some Scottish malt distilleries do carry out a triple distillation or have a variation on feints recycling that gives a similar effect. Nevertheless, these distilleries are very much in the minority. It is now the most common practice to double distil, with the fermented wash being distilled to low wines in a dedicated wash still, which is usually larger than the spirit still used to re-distil the combined low wines and recycled feints. The design of these stills is such that a balanced distillation pattern can be maintained – or in other words, there is a 1:1 or 2:1 ratio of spirit to wash distillations.

The only major developments in distilling technology over the last 40 years has been in the way stills have been heated and the way in which the spirit is condensed. Traditionally both stills were directly heated with either a peat or coal furnace underneath the still. This could lead to problems in the wash still,

with wash solids 'burning on' to the copper base. To prevent this, most stills were fitted with copper chain stirrers or 'rummagers' to keep solids in suspension and so prevent the finished spirit becoming tainted with the 'burned' flavour notes associated with furfural and related pyrolytic compounds. From the 1960s, nearly all malt stills were refitted with internal steam heating coils supplied from oil-fired boilers. More recently, centralized boilers have been automated and are increasingly being changed to natural gas firing. This development, along with automation of mashing and fermentation, means that distilling is less labour intensive and much more energy efficient than of old.

The other major development has seen the partial demise of the condensing worm and associated worm tub in favour of the more energy-efficient shell and tube condenser. Recent research has demonstrated that spirit copper levels and hence spirit character are heavily influenced by the way in which the spirit interacts with copper (Reaich, 1998). In particular, spirit condensed in worms contains less copper and is associated with 'heavier' or more 'sulphury' flavour notes than that condensed in shell and tube condensers (Bathgate, 1998a). It is therefore important to retain a number of traditional worm stills to maintain product diversity, especially for those blends that require a certain quantity of 'heavier' characters.

The heart of a good blend of Scotch whiskey is always a consistent, lightly flavoured grain spirit. The development of such grain distilling can be traced to the early nineteenth century. As we have seen, the demand for 'light' spirit steadily increased, particularly from London gin distillers, who required a clean, rectified spirit for redistillation with the essential botanicals that are the characteristic of the beverage. Empirical research with mixed grain and malt mashes must have demonstrated that the higher the content of unmalted grain the lighter the spirit, even if the distillation was carried out in pot stills. Batch distillation inevitably restricts output, and demand was outstripping capacity. Following several trials in 1828–1829 the first continuous still, designed by Robert Stein, was installed at Kirkliston distillery outside Edinburgh. Following the success of these trials a similar still was installed at Cameronbridge distillery in Fife, so increasing its capacity enormously (Robson, 2001). The Stein still was a somewhat complex apparatus, and when Aeneas Coffey created his simpler and more efficient Patent still at his Dublin distillery in 1830 he introduced the standard design that is now almost universal throughout the distilling industry. The Coffey still is particularly suited to grain distilling when the spent grains (draff) are not separated from the fermented worts. Although stainless steel is used in some stills, a copper purifier is essential to maintain a clean spirit, and most distillers prefer all-copper designs.

Over the last century the actual grain recipe has changed considerably. The legal definition of Scotch whiskey allows any cereal to be used, as long as it is saccharified by the enzymes of malted barley. Therefore the mash bill has been driven more by economics than by any technical or quality issues. For many years imported American maize was the cheapest cereal available, but since the UK entry into the European Union locally grown wheat has been the better

economic option even though it does not have as high a spirit yield as maize (Walker, 1986). The process whereby the unmalted cereal is mashed has seen little change except that separation of worts is no longer the norm, and there is still some debate as to whether milling is a necessary prerequisite to cooking. Nearly all the remaining large grain distilleries favour 'all-grains in' fermentation as opposed to separating spent grains from the worts, and some prefer to pressure-cook whole grains and allow explosive decompression to disintegrate the gelatinized cereal rather than carrying out energy intensive dry milling beforehand.

The two disciplines of distilling combine at the maturation stage of the overall process. As mentioned above, the maturation of Scotch whiskey in oak barrels happened partly by chance, and initially there was no stipulation on age. Age limitation was introduced by legislation rather than a drive to achieve better quality, and the three-year minimum was only introduced during the First World War as a government initiative to restrict the volume of whiskey coming onto the market in the hope that this would prevent drunkenness in munitions factory workers. Nevertheless, it is now well established that whiskies do differ in their optimum age of maturation, with grain whiskey generally maturing faster than malt. Traditional single-story (or at most two-story) warehouses were deliberately cold damp places so that spirit losses were minimized and maturation was slow and even. Indeed, one of the oldest bonded warehouses in Scotland, at Port Ellen Distillery in Islay, is built literally on the seashore, with no damp proofing. The relative humidity is therefore constantly high, and the temperature remains between 10°C and 15°C all the year round. Casks were stowed two or three high in at least a three-man operation and, since casks had also to be filled on site, filling and warehousing remain the most labour-intensive tasks in a traditional distillery. New-make whiskey is now generally road-tankered to a central filling point adjacent to large warehousing complexes situated in the central belt of Scotland. While there have been arguments about the rate and evenness of maturation in racked or palletized warehouses compared with traditional bonds, the huge volumes that are now distilled make the former an economic necessity. The vast bulk of maturing whiskey is therefore bonded in racked warehouses where casks are easily accessed by mechanical hoists. This has massively reduced the labour required for cask movements, both in for maturation and out for disgorgement and blending.

The development of maturation in oak casks is defined in law, but this has only been the case since the beginning of the last century; prior to that the definition only stipulated that maturation had to take place in 'wooden' casks. When distilling was a relatively small operation, there were sufficient recycled wine and fortified wine casks to suffice. Inevitably residual wine flavours absorbed into the wood imparted a specific character to the whiskey, to the extent that some brands of malt whiskey still specify this finish as an essential requirement. The growth of grain distilling inevitably swamped the available supply of European casks, and alternatives had to be found. The development of bourbon whiskey distilling in North America provided the solution. Since bourbon casks are generally only used for one filling, have been charred

internally and are made from high quality oak, they are ideal for maturing light flavoured Scotch – especially the faster maturing grain spirit. To facilitate transport of empty casks, North American barrels were broken down into bundles of staves (shooks) and then reconstituted, sometimes with the addition of an extra stave to create a 'dump hogshead'. Availability of suitable oak is now creating problems on both sides of the Atlantic, and since sherry wine is not so popular as it once was, new solutions have proved necessary. Once a cask is deemed to be 'exhausted', after a number of refills and prolonged periods of maturation (up to 25 years), it can be mechanically scraped to remove old 'char' and then re-charred and treated with imported bulk sherry if necessary (Philp, 1989). With average maturations of between five and twelve years for standard and deluxe blends respectively, cask re-use can now be extended almost indefinitely. However, shortages of suitable re-use casks and wood for the coopering of new casks remain a concern within the industry.

Blending of Scotch whiskey is a relatively new technology, given a history spanning hundreds of years. Originally new-make whiskey was consumed straight from the still as a clear spirit somewhat similar to schnapps, and was very erratic in quality. Early distilling must have been a hit-and-miss affair, since there were no adequate measuring devices to aid process and quality control, and so the variation in new-make spirit character must have been equally hit or miss. The introduction of hydrometry, and in particular the Sikes hygrometer in 1802, to measure alcohol strength did make distilling more consistent. However, the whiskey merchant must have had great difficulty in maintaining quality, and the only effective means he had was to mix batches to create a consistent spirit character. The first blending operation was not, however, a blend of grain and malt, but rather a mix of batches of a single malt, and had to have approval by Customs & Excise. Andrew Usher was, in 1853, the Edinburgh agent for Glenlivet malt whiskey, and he is accredited with producing this first vatted malt. Blending of grain whiskey with malt was not given approval until 1860. It was from that date that the great brands of blended whiskey flourished. The proportion of grain to malt, the specific distilleries used as the source of the spirit, the age of each malt and grain whiskey, and the type of wood used for maturation gave the blender an almost infinite number of combinations to create a unique blend. The recipes for the most successful blends are closely guarded secrets, so it comes as a surprise to outsiders to find that individual brand companies swap or 'reciprocate' malt and grain whiskies with one another. However, internal trading is regarded as one of the strengths of the industry, giving the blender a much wider scope in selecting appropriate malt and grain characters for a wider range of products.

Packaging of Scotch whiskey has always been an important aspect of marketing and of presenting the consumer with an image of the highest quality. For more than a century the industry has promoted 'bottled in Scotland' as a means of maintaining the exclusivity of the product, so preventing counterfeiting and bulk blending of Scotch with locally distilled spirit. In contradiction to this, Scotch was originally sold to British publicans in bulk, and the spirit had then to be decanted into large glass decanters that usually sat in a

prominent position on the bar. These attractively designed decanters held a gallon or more of whiskey, and were usually decorated with brand advertising. They have become something of a collector's item since Scotch is now exclusively sold in glass bottles – except for some PET 'miniatures', which are bottled for consumption on aircraft.

Development of new glass designs is a continuous process as the Scotch whiskey industry fights to maintain its market position as a deluxe product. Nevertheless, some brand companies have maintained a traditional bottle shape that is synonymous with its content. Walker's square bottle and the triangular bottle associated with Grant's products are perhaps just as well known as the distillers. In this respect the ultimate accolade must be for the whiskey to be named after the package, as in Haig's Dimple or Walker's 'Swing'.

Scotch whiskey is therefore still developing both technically and commercially. The Victorian whiskey barons were probably better known for their marketing acumen than they were for their distilling skills, and their modern-day counterparts are still the market leaders in many aspects of promotion and advertising. Such development will no doubt continue to maintain the worldwide popularity of Scotch whiskey.

Irish whiskey

Commercial development

It is widely acknowledged that whiskey distilling originated in Ireland, and that the necessary skills were brought to Scotland by missionary monks who first settled in Whithorn and Iona in the West of Scotland. It is therefore surprising that Irish whiskey has not been commercially exploited to the same extent as Scotch. One reason for this may lie in the different excise regulations exercised in both countries. If anything, the duty levied on Irish whiskey in the eighteenth and nineteenth centuries was even harsher than that imposed in Scotland. The inevitable rise in illicit distilling was similar in both countries, with something like 2000 illicit poteen stills in operation throughout Ireland by the end of the nineteenth century. The incentive to become legitimate may not have been so great, or perhaps there was a lack of necessary capital to build larger distilleries, but whatever the reason Irish distilling remained 'underground' for much longer – indeed, it is alleged that the distilling of poteen is still an active past-time in the Irish Republic.

Having said that, one of the oldest licensed distilleries in the British Isles (if not the oldest) is Bushmills Distillery in Coleraine. Charles Craig (1994) records that a license was granted to Sir Thomas Phillipps to make *aqua vitae* at Bushmills in 1608. Interestingly, this was a pot still malt distillery, and is the only Irish malt still in existence. The development of the few large commercial enterprises in Ireland is, however, very similar to that in Scotland in that they were dependent on a small number of entrepreneurial

families such as the Powers, Jamiesons and Roes. For example, George Roe & Co. commenced business when Peter Roe bought a small distillery in Dublin in 1757. This enterprise was continued by Richard Roe from 1766 to 1782, by which time he was working a still of some 234 gallons capacity (McGuire, 1973). Dublin soon became the centre of distilling operations, with the above families creating four large distilleries by the early nineteenth century.

These family concerns continued to dominate the Irish distilling industry for the rest of the century. There were strong links with the Scottish distilling industry, however, and the DCL in particular had interests in Ireland almost from its inauguration in 1877. For example, it operated a malt distillery, Phoenix Park, in Dublin until the 1920s, purchased Dundalk Distillery in 1912, and at one time had a 50 per cent interest in the United Distillers of Ireland's grain distilleries. The history of the family dynasties followed a similar pattern to that of their Scottish counterparts, with mergers and consolidations. The last two great family concerns, Power's and Jamieson's, combined forces with the Cork Distillers Company in 1966 to form the Irish Distillers Group. In 1970 this Group also acquired the Bushmills Distillery, to bring the whole of Irish distilling virtually into one entity. Irish distilling also fell victim to globalization, when in 1989 the Irish Distillers Group was purchased by the French drinks giant, Pernod–Ricard.

This abbreviated commercial history is somewhat similar to that of Scotch whiskey, but does not explain the relatively modest international recognition of Irish whiskey. All the opportunities were there, but it may be that the great Victorian entrepreneurs of Scotch whiskey were quicker off the mark, especially in exploiting the North American markets. Here, the huge Irish diaspora should have been the launching pad for establishing Irish whiskey across the continent. Nevertheless, Irish whiskey is internationally recognized as a distinctive generic brand in its own right and continues to flourish from its one large distilling, maturation and packaging centre at Middleton, near Cork.

Technical development

Premium Irish whiskey, such as Jamiesons, is pot distilled from a mixed grist of malt and unmalted barley. In this respect Irish distilling is immediately differentiated from Scotch single malt pot stills. As stated earlier, Scottish pot distillers historically used a mixture of malted and unmalted grains in their grist and only became exclusively malt distillers in the nineteenth century. Irish distillers have retained this tradition, and until quite recently also included about 5 per cent of unmalted oats in the grist as well as unmalted barley. Just as traditional English brewers used to use oat hulls as a mash filtration medium, Irish distillers maintained that a small percentage of oats in the grist improved mash-tun run-off (Lyons, 1995).

The scale of Irish pot distilling operations is also markedly different from that in Scotland, with the average batch size being roughly double in the former. For example, Lyons (1995) quotes wash-back volumes for Irish pot distilling in the range of 50 000 to 150 000 litres. However, the most obvious

technical differences between the two traditions are to be found in the still house. The size and design of Irish pot stills are quite different, as are the distillation profiles. With very large volumes, the aspect ratio of exposure of liquid to copper vessel surfaces is much smaller in large pot stills as opposed to small Scottish pots, where the volume may be as low as 5000 litres. It is not surprising then that Irish stills are fitted with 'purifiers', which return condensate to the still, so increasing the amount of reflux over copper and hence reducing the concentration of certain undesirable flavour congeners in the distillate. The distillation patterns differ in that in Irish distilling the low wines derived from the wash still are split into two fractions of different alcoholic strength. The weak low wines are distilled separately in a low wines still to provide two further fractions, strong and weak feints. The weak feints are recycled for redistillation with the next batch of weak low wines, while the strong feints are combined with strong low wines for the final spirit distillation. The spirit collected is therefore about 30 per cent stronger in spirit strength than the equivalent Scottish malt. The overall effect of what is essentially triple distillation is a smoother and, some would say, sweeter product that is probably closer to Scottish grain whiskey than it is to a single malt.

North American whiskies

Commercial development

The art of whiskey distilling migrated to the eastern seaboard of North America in the ownership of the many Scottish and Irish settlers in the seventeenth and eighteenth centuries. As in their homelands, early distilling was a cottage- or farm-based enterprise and was principally carried out for domestic consumption. Initially settlers used barley, oats and wheat grown from their own imported cereal seed, but increasingly used rye, which was cultivated in abundance in Maryland and Pennsylvania. Later they created 'corn' whiskey, using native 'Indian corn' (i.e. maize) as their principal raw material. Commercial distilling grew from these roots, particularly in western Pennsylvania.

Predictably, government intervention in the form of an excise revenue was inevitable. Sure enough, the fledgling Continental Congress attempted to levy a tax on whiskey production to pay off debts incurred during the War of Independence. Equally predictable was the ensuing western Pennsylvanian 'Whiskey Rebellion' of 1791–1794. The resistance of the notoriously feisty Scottish–Irish colonialists proved an embarrassing political situation for George Washington. A settlement was reached whereby distiller/farmers were offered incentives to move across the Ohio River into Kentucky, in return for a levy on their distilled liquor. At that time Kentucky was part of the state of Virginia, governed by no less a personage than Thomas Jefferson. Jefferson, presumably in collaboration with Washington, offered each of these pioneer

distiller/farmers some 60 acres of prime agricultural land in what was soon to become the new state of Kentucky. They were encouraged to grow Indian corn in the fertile Bluegrass region of Kentucky and to use the surplus grain to distil whiskey. In recognition of the assistance received by the emerging United States of America from France (which still had colonial interests in Louisiana), the first counties in Kentucky were given names associated with their European allies. In particular, one county was named after the royal family of France, the Bourbons, and it was here that many of the early pioneers from Pennsylvania settled and started to produce 'bourbon' corn whiskey. Not all Kentucky whiskey is distilled in Bourbon County, but most of the early produce was exported down the Ohio/Mississippi rivers to New Orleans via Bourbon. All transhipped casks were stamped with the name 'Bourbon' as their port of origin, and so it came to pass that nearly all Kentucky corn whiskey was termed bourbon. This mode of transport also had an important part to play in the development of the character of bourbon whiskey. One of the distinguishing characteristics of bourbon is that it is matured in oak casks that have been charred prior to filling with new-make whiskey. How this technology came into being is somewhat obscure. One version is that some early coopers charred their oak staves to make them more pliable and some astute distiller noticed that such casks produced a smoother, less fiery spirit. The more interesting (but perhaps apocryphal) story is that the Reverend Elijah Craig of Bourbon was short of casks to transport his whiskey. The only casks available had previously contained pickled fish, and, to remove the malodour, he burned off the inside of the cask, leaving a layer of charcoal. With the continuous movement of the spirit as the casks were floated downstream on flatboats, the whiskey was subjected to an instant maturation – much to the delight of the receiving merchant. Now why a man of the cloth should be dealing in whiskey is not related, but the fact that the said Reverend. Craig was a Scot does lend some authenticity to a story that has become part of the folklore of Bourbon.

So the name 'bourbon' became almost generic for any corn-based whiskey, and a legal definition was only agreed by a Congressional resolution in 1964. The basic elements of this definition are that the spirit must be made from a mash that contains at least 51 per cent maize and is matured for at least two years in charred casks made from new oak. Although the state of origin is not stipulated, as it is for Scotch, nearly all bourbon is distilled in Kentucky. As the Kentucky Distillers Association (2002) is quick to point out, Kentucky is the only state allowed to put its name on a bourbon label, and it jealously guards the traditions of its products. Thus the commercial and technical developments of American whiskey are inextricably linked.

As with the other whiskey traditions, the further commercial development of American whiskey was dependent on a number of notable families who can still trace their roots back to those early settlers. They all brought something to their brands, by variations in their cereal recipes, developing propriety yeast strains or adapting their technology in some way that enhanced and differentiated their particular spirit. Jack Daniel's Tennessee whiskey perhaps exemplifies this better than any other. As well as utilizing a sour mash the new-

make spirit is slowly filtered through a bed of charcoal prior to maturation, so producing a particularly smooth 'sipping' whiskey. The strength of these family ties is such that many survived the difficult times of prohibition in the USA and carried the industry forward to the point where a number American whiskey brands now enjoy an international reputation.

Following the end of prohibition, federal controls were introduced that banned the sale of whiskey in bulk casks and introduced quality standards, which are now in the remit of the US Bureau of Alcohol, Tobacco and Firearms (BATF). These standards define bourbon, corn, rye, and wheat whiskies according to their respective mash bills. These must contain at least 51 per cent of corn, rye or wheat. Corn whiskey differs from bourbon in that it can be matured in used or uncharred casks, and may include a mixture of other whiskies. The general definition is that 'whiskey is a spirit aged in wood and obtained from the distillation of a fermented mash of the aforementioned mash of grains'.

Canadian whiskey developed industrially on the back of demand from the USA. This is best exemplified by Canada's most famous brand, Canadian Club. In 1858, Hiram Walker built and developed his whiskey business, not in his native Detroit but just across the border in Windsor, Ontario, where he felt he was not subject to the same legislative restrictions. Not only did he build the necessary distillery, warehouses and packaging facilities, he also built a village, Walkerville, for his employees. Similarly, in 1857 Joseph E. Seagram started up a small distillery out in the prairies, so availing himself of an abundant source of corn and rye. In 1928 Seagram's became part of the Distillers Corporation, which had been founded a few years previously in 1924. After prohibition Distillers Corporation–Seagram's expanded rapidly into the USA, and so, like Hiram Walker, the commercial development of both organizations was inextricably linked to the American market. Regulation of beverage spirit is now perhaps more strictly controlled in Canada, where the principal whiskey is derived from rye grown in the prairies east of the foothills of the Rocky Mountains (Morrison, 1995). The industrial growth of this particular whiskey dates from the late 1940s when distillers took the opportunity to utilize the abundance of rye, which can be grown as an autumn sown crop in some of the fertile sandy soils of Alberta.

It is ironic that these two great bastions of North American whiskey have recently been acquired by European/Global Spirit Businesses, with Hiram Walker now part of Allied Domeq, and Seagram's split up by a joint venture between Diageo and Pernod–Ricard. Previously the acquisitions had been in the other direction, when Hiram Walker was a major player in the Scotch whiskey industry and Seagram's had purchased Chivas Brothers–Glenlivet Distillers.

Technical development

As elsewhere the early North American farmer/distillers used portable pot stills, but from the start of industrial operations in the nineteenth century

American distillers were quick to develop continuous distillation using Coffey-type column stills or a combination of both column and pot still. This also allows very large batches of cooked grain to be processed. Although some continuous cooking has been developed, the major Bourbon and Tennessee Distillers are committed to batch cooking.

Commercial development and technical development have again gone hand in hand, since the main differentiation of American whiskies is based on six technical factors (Ralph,1995):

1. Proportion of different cereal species in the mash bill
2. Mashing technique
3. Strain of yeast and yeasting technique
4. Type of fermentation (controlled or uncontrolled)
5. Type of distillation (single continuous or doubler)
6. Cask type and maturation processing.

Each of these key parameters has developed from traditional processes. Obviously, the cereal content was and is dependent on availability. While the mash bill recipes are now regulated, there is considerable scope for variation; however, generally the proportions used are as shown in Table 1.2.

Table 1.2
Grist composition of North American whiskies

Type	Corn (%)	Rye (%)	Malted barley (%)
Bourbon	70	15	15
Tennessee	80	10	10
Rye	39	51	10

The cooking and mashing process is very similar to that of Scotch grain whiskey, with one or two interesting developments. Nearly all North American distillers recycle stillage (pot-ale) after the removal of spent grains. Since all fermentations take place without the removal of unconverted cereal, spent grains and yeast are removed from the bottom of the column still as a suspension in the residual liquor (stillage). Solids are removed by screening or centrifuging, and a portion of the quite acidic residue is added to the next mash as 'backset'. This process is probably related to the historical practice of recycling a portion of the fermenting mash to inoculate the next batch of cooked cereal. Similarly, the process of 'sour mashing' has a historical precedent in that recycled yeast will have built up a commensal population of lactic bacteria. This develops into a controlled 'yeast mash' of rye and malted barley, which is inoculated with lactobacillus and allowed to acidify until the pH is 3.8 The bacteria are then killed off by increasing the temperature to 100°C, and the now sterile 'sour mash' is inoculated with cultured yeast and allowed actively to ferment before being used to inoculate the main mash (Ralph,

1995). Since the natural distilling water in both Kentucky and Tennessee comes from iron-free limestone, both these techniques of 'backsetting' and 'sour mashing' are perhaps essential in providing the optimum pH for conversion and fermentation of the cooked mash. In traditional distilleries the fermentation takes place in open wooden vats with no temperature control, whereas in the larger modern distilleries the fermentation takes place in closed stainless steel fermenters with strict temperature control.

As with Scotch grain whisky, bourbon is generally double distilled, but the second distillation takes place in a pot still (a 'doubler' or 'thumper' still). 'Low wines' collected from the column still are refluxed in the 'doubler' and the distillate collected as 'high wines', which are then filled into new charred white-oak barrels – the main characteristic of bourbon whiskey. In Tennessee, the new-make whiskey can be further 'smoothened' by filtering through a bed of charcoal before filling. Maturation for at least two years completes the process, although developments with 'single cask' and longer maturation have shown that quality bourbon can compete with Scottish single malts.

Japanese whiskey

Commercial development

Of all the countries with a thriving whiskey distilling industry, Japan is the only one where it was not founded by Scottish/Irish immigrants. Nevertheless, there were and still are strong ties between Japanese and Scottish distillers. These ties go back to the vision and enterprise of one or two individuals who created a substantial market for locally distilled whiskey. The fathers of Japanese whiskey are Shinjiro Torii and Masataka Taketsuru. The latter was an employee of the Osaka company Settsu Shuzo, which had plans to create a whiskey distilling operation in Japan prior to the First World War. Plans were made, and Taketsuru was enrolled at Glasgow University in 1918 to study chemistry and whiskey distilling. This he did assiduously, and he returned to Japan in 1920 only to find that his company had shelved their plans for launching a 'Scotch' type of whiskey on the Japanese market. In 1922 Taketsuru quit his job in frustration and was approached by Shinjiro Torii of the rival wine merchanting business Kotobukiya (later to become Suntory), and was asked to build and manage a new malt distillery that was to be established the following year in the Yamazaki Valley on the outskirts of Kyoto. Within two years the distillery was fully operational, and Taketsuru continued with Torii's developments until, in 1929, Japan's first whiskey, Suntory Shirofuda (now called Suntory White) was launched (Suntory, 2002). It is interesting to note that Torii had a similar background to some of the great Scottish entrepreneurs, such as the Dewars, the Walkers, the Chivas brothers and Arthur Bell, in that he was originally a wine merchant who saw the commercial opportunity for developing his own brand and style of

whiskey. Torii's first whiskey was predominantly his own young Yamazaki malt blended with unmatured neutral spirit. These early Japanese whiskies were therefore unique, and Suntory did not produce a 'Scotch' blend until 1973, when non-cereal neutral spirit was replaced with a matured grain whiskey made from North American maize.

Taketsuru had an even stronger desire to recreate a 'Scotch' malt, and always felt that Japan's northernmost island, Hokkaido, had the geography and climate nearest to those of Scotland and hence that a truly 'Scottish' malt whiskey could best be distilled and matured there. He therefore left Suntory in 1934 to set up his own distilling business in Yoichi, Hokkaido, under the banner of Nippon Kaju KK (later to become Nikka) (Nikka, 2002).

These two companies are still at the core of the Japanese distilling industry, and since the Second World War have both renewed their bonds with Scotland by buying into Scottish distillery operations. Other Japanese businesses and trading houses have also participated in the growth of whiskey distilling, either by setting up joint venture companies (such as Kirin–Seagram) or by importing bulk Scotch whiskey to blend with locally distilled spirit. Japanese marketeers have recently posed the question whether, because their own distillers had striven to replicate a 'Scotch' at ever-increasing expense, would it not be more economic to own a Scotch whiskey operation and export from Scotland? Recent reductions in Japanese import duty have driven this trend, so reducing demand on those Japanese distilleries that expanded so rapidly in the 1970s. This expansion may in itself have contributed to some of the blending problems encountered in trying to replicate a 'Scotch' blend. With only one or two very large malt distilleries from which to draw matured spirit, and with (until relatively recently) a limited supply of grain whiskey, the malt character predominated, so a balanced blend was well nigh impossible. The fact that a very acceptable and distinctive 'Japanese' whiskey resulted from the original development appears to have been ignored in the desire to recreate a 'Scotch'. From the very modest 17 000 litres produced in 1931, volumes of Suntory whiskey increased exponentially even throughout the Second World War. In 1944 Suntory produced 771 000 litres of whiskey, and at its peak in the post-war years it was selling 30 million cases a year. All of these products flourished without hindrance by punitive excise duty and it can be argued that, by imposing high import taxes on both Scotch and North American whiskies in the post-war years, successive Japanese governments fostered the growth of their indigenous whiskies. However, Suntory's sales have now fallen back to around 10 million cases, perhaps owing to imported Scotch and bourbon, but more likely because of a switch in drinking habits in the Japanese market.

Technical development

Both Torii and Taketsuru were convinced that for a Japanese whiskey to succeed, it would have to be of the highest quality. In an early form of best practice, they set out to recreate or procure the best raw materials, distilling

technology, and maturation conditions required to reproduce a 'Scotch malt' type of whiskey.

As in Scotland, the original Yamazaki distillery had its own floor maltings. Locally grown winter barley was windrowed and harvested unthrashed. Bales of barley on the straw were then stored in the distillery and were only thrashed prior to malting. The green malt was kilned in the presence of peat smoke to produce a lightly peated malted barley. With an almost continuous increase in demand, these floor maltings could not cope with the increased capacity of the distillery and were finally closed in 1973. All malt is now imported, with a large proportion coming from Scotland and an increasing amount from Australia.

Malt whiskey was (and still is) fermented in wooden wash backs with cultured yeasts, and distilled in traditional copper wash and spirit pot stills. The original Yamazaki distillery contained only two stills, but these doubled in 1958 and then trebled in 1972. At this time a new malt distillery and a 40-million litre grain distillery were also built near Tokyo. The malt distillery was built with six wash and six spirit stills, and was then doubled in capacity so that it can now produce around 22 million litres absolute alcohol per annum.

Grain distilling is relatively new to Japan, so Suntory was able to make use of the best available continuous distillation technology and combine the production of grain spirit with the distillation of neutral spirit manufactured for other beverages such as gin and vodka. The grain whiskey is made using North American maize, which is mashed and distilled conventionally.

As in Scotland, malt and grain whiskey is matured in oak casks before blending. Originally Taketsuru matured his spirit in very large puncheons coopered from Japanese oak. This entailed long, slow maturation periods. With increased demand in the 1970s, these puncheons have been replaced with barrels made from imported North American oak staves.

The most popular brands are blends, but single malts have been successfully launched by Suntory, which has diversified its whiskey production away from high-volume distillation at principally one malt distillery. In fact, a smaller distillery was built at Nakashu in 1981 specifically to create a range of differentiated malt whiskies. The realization that successful blending is dependent on blenders having a wide range of different whiskies in their palette has brought the relationship between Scotland and Japan even closer. Most Japanese whiskey businesses now fully own distilling, maturation and blending facilities in Scotland, setting a trend that will continue into the new millennium.

Acknowledgement

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Chapter 2

Malt whiskies: raw materials and processing

Timothy C. S. Dolan

Introduction

This chapter describes the raw materials used in the malting and wort preparation stages of the production of malt whiskies, together with the science and technology of their preparation for fermentation. The raw materials used in these stages are air, water, barley, peat and malt. In the later fermentation and distilling stages, described in Chapters 4–6, yeast, copper and wood are employed.

The major raw material is barley, which is made into malt – the basic material used in mashing. The parameters required in barley to make malt of the quality needed for malt distilling are explained, and the malting process is fully described.

Malt whiskies are made in a number of countries, including Australia, Canada, the Czech Republic, England, France, India, Japan, New Zealand, Northern Ireland (NI), Pakistan, the Republic of Ireland (RoI), Scotland, Sweden, Turkey, Wales and the USA (Murray, 1997). The major producers are Scotland, Ireland, India and Japan. All malt distillers use some malted barley as the source of fermentable sugars and required enzymes. Each country has a different definition of what constitutes malt whisky, with corresponding legislation. In Scotland and Ireland the legal definitions are strongly set out (see below). In Scotland, Scotch malt whisky is made from 100 per cent malted barley with no other grains included.

The legal definition of Scotch whisky

Scotch whisky has been defined in UK law since 1909 and recognized in EC legislation since 1989. The current UK legislation relating specifically to Scotch whisky is the Scotch Whisky Act 1988 and the Orders made under it, which came into effect in June 1990 and superseded that part of the Finance Act, as subsequently amended, defining Scotch Whisky. For the purposes of the Scotch Whisky Act 1988, 'Scotch whisky' means whisky:

1. That has been produced at a distillery in Scotland from water and malted barley (to which only whole grains of other cereals may be added) all of which have been
   - processed at that distillery into a mash
   - converted to a fermentable substrate only by endogenous enzyme systems
   - fermented only by the addition of yeast.
2. That has been distilled at an alcoholic strength by volume of less than 98.4 per cent so that the distillate has an aroma and taste derived from the raw materials used in, and the method of, its production
3. That has been matured in an excise warehouse in Scotland in oak casks of a capacity not exceeding 700 litres, the period of that maturation being not less than three years
4. That retains the colour, aroma and taste derived from the raw materials used in, and the method of, its production and maturation, and to which no other substance other than water and spirit caramel has been added.

The Scotch Whisky Act 1988 prohibits *inter alia* the production in Scotland of whisky other than Scotch whisky. The Act and The European Spirits Definition Regulation both specify a minimum alcoholic strength of 40 per cent by volume, which applies to all Scotch whisky bottled and/or put up for sale within or export from the EU.

It can be seen that the only raw material expressly specified is malted barley. Its use is a legal requirement.

This chapter focuses on Scotch malt whisky, referring to the differences pertaining to other malt whiskies.

Raw materials and processing

Air

It is desirable that all distilling operations be carried out in a wholesome air environment. In malting, it is particularly important that the air used in germination and kilning is clean and pure, to ensure the wholesomeness of the malt and to prevent the formation of any unwanted compounds, such as volatile nitrosamines. The consequences of using unsuitable air in kilning are discussed under Malting (see below).

Water

Water is used in malting, mashing and reducing alcoholic strength (production and product water), to generate and distribute heat for cooling, and to clean the plant (process and service water). The entire cycle of making malt whisky is a series of adding and removing water. When barley is sown it takes up water to allow growth. At harvesting some is removed so that the barley can be stored safely. At steeping it is added and at kilning it is removed – in this case for safe storage of the malt. Water is added to the malt at mashing, removed at distillation and added at spirit reduction before bottling. Often water is added by the consumer, who metabolizes the whisky and passes the water back into the environment, from whence it is used by the next generation of barley. It is worth bearing in mind that throughout the entire cycle it is cheap to add water, but expensive to remove it.

Water sourcing

A plentiful supply of water is essential for malting, mashing and distilling, and this is one of the reasons why, in the past, malt distilleries were built in areas that had their own sources, usually in the form of wells or boreholes. These original sources are still used in many plants, while others rely on the local water authority for their supply. Another important factor was that in the days of waterpower, a suitable source of water to drive the waterwheel was required.

The nature of the water source affects the characteristics of the water (see Table 2.1) and thus the quality of the spirit produced, and this fact has resulted in some areas being famous for their spirit. The quality of the water can also affect the efficiency of the processes where it is used, for example in boiler-feed water or in plant-cleaning systems.

Table 2.2 lists the characteristics required for a distillery water supply.

Methods of pre-treatment of water

Some water sources require no treatment, so the selection of a 'perfect' supply is the ideal. However, a water supply that meets all requirements is rare. and usually some form of water treatment is used (see Table 2.3).

De-ionization of water is common in distilleries. Ion exchange columns contain resins that are capable of exchanging the unwanted ions for harmless ones. The resins can be regenerated when exhausted, usually by washing through with mineral acids.

Sand filtration is used for the removal of solids. Carbon filters can be used for the removal of flavour taints caused by chlorine, for ion exchange columns for de-mineralization or de-alkalization, and for membrane filtration or reverse osmosis for the removal of most contaminants, including bacteria. Iron and manganese can be removed using a BIRM filter.

Table 2.1
Sources of water and their characteristics

Source	Mineral salt content	Microbiological content	Taints	Consistency of supply
Boreholes	Contain some soluble material present in the rock strata where the water is held	Likely to be low because the water has been filtered through rock strata	Likely to be low unless the water has been contaminated by surface water, e.g. in built-up areas	Very good over long periods of time
Surface	Likely to be low unless agricultural chemicals are being washed off the land	Likely to be high because of contamination from farm land	Likely to be low unless the water is contaminated by accidental spillage	Can be variable, especially in periods of drought
Public supply	Depends on whether the source or borehole or surface; any anomalies should be known if the supplies alternate	Likely to be low because of treatment by the water authority	Likely to be low because of treatment by the water authority	Very good because of the water authority's legal obligations

Table 2.2
Required characteristics of water for a malt whisky distillery supply

Characteristic	Standard
Appearance	Clear and colourless
Wholesomeness/potability	Freedom from taint
Mineral salt and metallic contents	Contents that meet the mashing, reducing and process requirements
Microbiological standard	Reducing water must be of potable standard
Reliability of supply	Water must be available at all times

Methods of sterilizing water

The method of water sterilization depends on the level of infection and the subsequent use of the water. If it is heavily infected, it may require filtration followed by heat treatment; if it is lightly infected, sterilization by ultraviolet light or sterile filtration is more usual.

Table 2.3
Methods of pre-treatment of water

Quality standard	Treatment
Clear and colourless appearance	Filtration, usually through sand
Wholesomeness/potability, freedom from taint	Filtration, usually through carbon
Mineral salt and metallic contents that meet the production, product and process requirements	Unwanted ions removal by de-ionization
Freedom from micro-organisms that would spoil the production, product or process	Sterilization by ultraviolet light or sterile filtration
Availability of water at all times	An alternative supply is often used; where this is the case, different water treatment may be needed

Water usage

Production (mashing) water

Production water used in the production of wort makes a major contribution to the quality of the spirit that is produced. Salts dissolved in the water affect the wort's flavour and influence the pH of the process and the final product, and they provide essential trace elements for yeast growth. Briefly:

- Sulphates reduce mash pH
- The yeast needs calcium
- Carbonates raise pH and form scale on heating surfaces
- Nitrates indicate surface water or sewage contamination
- Magnesium and zinc are trace elements required by the yeast.

Table 2.4 shows how production water is used in the process.

Table 2.4
Entry routes of production water

Water use	Quality requirements
Mashing	In mashing, the water contains the salts needed to ensure the optimum conditions for effective enzyme activity
Sparging	The water must not dissolve unwanted material from the husk
The water yeast is mixed in	The water must be free of unwanted organisms

Product water

Product water is added to the new-make spirit to reduce its strength to that required for filling into casks. It must be potable water.

Process water

Process water is that water used for:

- Cleaning the production plant
- Cooling in the condensers or worms.

The water must meet the requirements of the process where it is used.

Service water

Service water is that used in boilers to raise steam and in cooling towers as part of the refrigeration plant, as well as that used for general hygiene cleaning.

The main requirements for boiler water are that it does not form scale deposits on the heating surfaces and that it does not corrode the plant. Consequently the removal of carbonates is essential, and often the standard de-ionization process described above is supplemented with further treatment. To prevent corrosion, additives are used to scavenge oxygen from the water and its pH is adjusted.

Several malt whisky distilleries are equipped with evaporative cooling towers. These are required where there is insufficient cold water into which to reject the unwanted heat, or where the volume of diluting water in the receiving watercourse is insufficient. Ever more stringent regulations mean that many distilleries will be unable to meet new discharge consent conditions for cooling water, which may lead to the requirement for other means of heat dissipation, such as a heat recovery system. Water used in cooling towers is prone to the growth of the bacterium Legionella, which is pathogenic and can infect humans through the fine droplets formed in the plant, causing Legionnaire's Disease. The system must be maintained in a clean condition, and the bacteria controlled with bactericides or through electronic methods.

General cleaning water is used for hosing down and general hygiene, and the normal standard supply can be used.

Water conservation

Management of water usage can be improved as in Table 2.5.

Barley

Barley procurement

The art and skill of making barley into malt is as old a technology as whisky distilling itself, and malting specifically for making Scotch whisky grew along-

Table 2.5
Ways of reducing water usage in malting and malt distilling

Water use	Wastage	Conservation
Production water	Excessive drainings from the mash/lauter tun; excessive flushing following product transfer	Measure and control volumes of water used
Product water	Careless reducing	Take care
Process water	Excessive flushes/rinses during plant cleaning; unnecessary dumping of detergent or sterilant water; leaks in cooling system	Automate CIP systems; use several short rinses rather than one long one
Service water	Loss of steam condensate	Plant maintenance
	Leaks	Supervision to ensure careful use
	Overuse of hoses for hygiene	Use of restrictors on cleaning hoses
	Water used for cooling	All cooling water recovered and re-used in the most energy-efficient way

side the craft of distilling. A few distilleries today still retain maltings on site, where they produce a highly characteristic product for that particular distillery. Although the industry is very old, the historic documentation of malting in Scotland is meagre. A paper devoted to malt was written by Sir Robert Moray in1678, in which he states that: 'Malt is there made of no other Grain but Barley, whereof there are two kinds, one which hath four rows of Grains in the Ear, the other two rows. The first is the more commonly used; but the other makes the best malt'. The four-row barley to which Moray referred is the forerunner of the landrace 'bere', which is still grown in Orkney, where it is used to make bere bannocks – a type of scone. These comments confirm that over 300 years ago the selection of appropriate varieties for malting was important. Moray discusses the importance of barley harvesting, storage, grading, steeping, germination and kilning, and, while his comments do not have the support of scientific evidence, much of what he describes is based on sound empirical knowledge. The factors mentioned above are as important today as they were over 300 years ago.

Barley harvesting and drying

Historically malting and distilling in Scotland were local affairs, with the distiller/maltster purchasing locally grown barley for processing. This was the case until the advent of the combine harvester in the late 1940s. Until then, barley was cut by scythe or reaper, stooked to allow final drying out and maturing, stacked, and finally threshed from the stack. Stooking helped to

reduce the final moisture at harvest. The moisture content of the grain in the stack would remain at 15–17 per cent until threshing took place. It was common for such grain to grow mouldy in the stack, and for the threshed grain to have very low germination energy.

The malting of local grain of such mediocre quality limited production capacity, because of the extended germination times required on the floor and the poor spirit yields. When compared with high-quality malting barley grown at that time in England for the expanding brewing industry, Scottish barley was certainly poorer. In addition, cheap barley with good percentage germination counts could be readily imported. In the period 1945–1965, Scotch malt whisky production outgrew the supply of malt from local maltings. The additional malt required came either from new pneumatic maltings (Saladin box type) that used imported English or southern Scottish barley, or as malt bought from English or European maltsters. The English barleys used were initially Spratt and Plumage Archer, followed by Proctor and, later, by the winter barley Maris Otter.

This trend continued until the mid 1960s, when a radical change in barley handling and malting technology once again promoted the malting of local barley (Griffin, 1982). This change involved using the malting plant as a barley drying facility at harvest to reduce the moisture to around 12 per cent, preserving the germination capacity and ensuring storage safe from infestation.

The first combined barley drying and malting plant to be constructed was based on a multiple Saladin box design (Griffin and Pinner, 1965), in which each box acted both as a batch steeping and germination vessel and as a kiln of approximately 50 t capacity for kilning malt or 100 t for barley drying. This plant was followed by larger concrete boxes (200 t malting, 400 t barley drying) and subsequently by circular steel vessels of up to 500 t capacity (Turcan, 1981; Stevens, 1984). In 2001, the largest of these combined drying and malting plants in Scotland could receive and dry over 2000 t wet barley per day.

At the same time as the development of these malting plants, farming technology was also developing very rapidly. With the introduction of larger combine harvesters, freshly threshed grain was available at harvest rather than being stored in stack. This increased the need to dry and store grain at harvest.

Maltsters prefer barley dried in one pass in deep-bed batch driers at low air-on temperatures (40–45°C) to barley dried in continuous cross-flow driers where higher air-on temperatures (60–65°C) are used. Although the superficial germination characteristics from both kinds of drier are similar, there is evidence to suggest that barley dried in deep-bed batch driers recovers from dormancy faster than that from continuous driers (Bathgate, 1982). There is also evidence indicating that drying barley using more than one pass is detrimental to the recovery. The practice of drying the grain from field moisture of 20–15 per cent to 15 per cent and then drying it from 15 per cent to 12 per cent is discouraged by maltsters, who nowadays prefer to have total control of the drying of their barley.

In warm parts of the world, dormancy is not usually a problem for maltsters. In Scottish conditions the winter barleys in use in the mid-1960s–1980s

and bred for southern climes were likely to show severe dormancy periods of up to sixteen weeks. From 1980, the spring barley Triumph gained ground as it gave much improved spirit yields, but it could remain dormant for up to 26 weeks. While dormancy can be reduced by appropriate storage conditions (see later), the constraints of having to dry and store barley on site at harvest are a severe financial limitation to Scottish maltsters. In spite of this, the benefits are substantial. By guaranteeing quality and variety and minimizing drying and storing costs rather than paying a merchant or farmer to perform this critical task, Scottish maltsters have been able to provide consistently high-quality distilling malts. The mild climate and high latitude day length generally produce plump barley with high thousand-corn weight and a relatively low total nitrogen (TN) content (about 1.6–1.7 per cent). On the other hand, the overall growing period is relatively short, with spring barley (generally sown in early March) being harvested in late August to early September.

For around twenty years (1965–1985) Golden Promise (GP) was the major variety in use. Despite being very susceptible to attack by mildew (SAC, 1988) and thus never becoming a recommended variety, GP has several attributes that are still necessary for a variety to be successful in Scotland:

- It is early maturing and more likely to be fully ripe at the end of the short Scottish growing season
- It has a short, stiff straw, and can resist ear loss during the autumnal gales prevalent towards the end of the harvest season
- It is not prone to dormancy
- It has malting quality superior to all of its predecessors either in terms of extract potential or in ease of processing in the maltings and the distillery.

The success of a barley variety can be gauged by its lifetime in the seed market. It is now almost 50 years since the first commercial crops of GP were harvested. As new varieties with higher agronomic and distillery yields, such as Triumph and its successors, were bred and launched into the farming scene from 1980 onwards, GP waned to a very small tonnage. It was, and remains in the second millennium, favoured by a handful of distillers, and in the year 2000 over 10 000 tonnes were harvested. In the 1980s, several new varieties that could outyield GP on the farm and outclass it in terms of malting quality and distillery yield were introduced. In particular, the variety Triumph, which was widely grown throughout Europe, gained in popularity, but it had several drawbacks. It was not as early maturing or as stiff-strawed as GP, and its resistance to mildew broke down. Its greatest weakness was its genetic inheritance of severe dormancy – it could be dormant for up to six months after harvesting. Several hybrids of Triumph, such as Corgi, Natasha and Camargue, became available in the mid 1980s. These varieties had the benefit that they did not inherit the severe dormancy character whilst also having the agronomic properties or earliness of GP. It was reported that early ripening is linked to lower dormancy levels for any variety, and this must be of primary importance in Scotland (Bathgate, 1987). There is no value in barley having

low TN content and high thousand-corn weight if it cannot germinate when required.

The extra storage costs of carrying stocks over from the previous harvest, to cover production requirements during the dormancy period, put a severe financial strain on Scottish maltsters, and in the late 1980s fervent efforts were made by plant breeders to produce varieties that were not prone to dormancy and gave better distillery yields. Other research at that time showed that transferring newly dried barley into storage while it was still warm led to a reduction in dormancy.

In the 1990s plant breeders were being given strong guidance by the Institute of Brewing (IOB) Barley Committee (BC) regarding the desirable attributes in barley destined for malting, (brewing) and distilling (note that the IOB is now called the IGB – Institute and Guild of Brewing), and were responding by breeding with these requirements in mind. In 1995 the malt-distilling sector provided the following consensus view of desirable barley attributes, which still pertains (Brookes, 1980):

1. High priority
   - Resistance to splitting at any stage
   - Mealy, not steely, endosperm
   - Rapid, even water uptake (hydration)
   - The quality of low nitrogen pick-up when growing
   - Vigorous germination
   - High starch content
   - Low glycosidic nitrile potential.

2. Medium priority
   - Good and uniform corn size
   - Good husk appearance
   - Ripens evenly in the northern UK
   - Low dormancy
   - No pre-germination.

Barleys that meet these requirements are much more likely to be bought for malting, as the malt in turn will be more likely to posses the attributes required of it when it is considered as the raw material for mashing.

In the 1990s liaison between malt distillers, maltsters, farmers, other interested parties and breeders became closer, and as a result of all parties understanding the attributes required in malt for producing malt whisky, the breeding programmes were more focused. In the decade, a succession of new varieties appeared that had the desired agronomic and processing qualities. Spring varieties have been the most successful, and examples include Chalice, Chariot, Decanter, Derkado, Optic and Prisma. Winter varieties that have been successful, mainly in southern Scotland, are Melanie, Halcyon and Regina. During the twentieth century, Scotch malt distillery yields increased from around 300 l of pure alcohol per tonne of malt dry weight (abbreviated to l/t (dwb)) to more than 460 l/t (dwb) (see Table 2.6). This was mainly due to increases in the fermentable extract contents of the

Table 2.6
Major improvements in Scotch malt whisky spirit yields – dates and causes

Year	Spirit yield, l/t (dwb)	Spirit yield improvement	Comment
1900	300	–	Chevalier in use
1910	300	–	Spratt Archer in use
			Plumage Archer in use
1950	350	50	Advent of Proctor and Maris Otter
1965	390	40	Advent of Golden Promise
1970	410	20	Effective cleaning and sanitization introduced
1990	430+	20	Advent of Triumph
1995	445	15	Advent of Chariot
1997	460	15	Vintage quality Chariot Harvest
2001	460	–	Next breakthrough awaited

successive varieties, but also to changes in agronomic and processing practices (Dolan, 2001) The malting aspects of the various varieties are discussed below, under Malting.

Regarding barley procurement, the high cost of transport from England and continental Europe, the presence of high-capacity, low-cost maltings near the major malt whisky distilleries, and the ongoing local availability of malting barley with the required attributes should ensure the future of this sector of the malting industry in Scotland.

Barley storage, cleaning and grading

When the barley has been dried to 12 per cent moisture, it is transferred directly to store, without cooling, to facilitate dormancy breaking. The temperature of the grain may be between 18°C and 25°C at this stage, depending on the expected degree of dormancy. In the Triumph era of severe dormancy, it was suggested that the temperature should be increased to around 30°C for a short period as a way of lessening the duration of the dormancy (Palmer and Taylor, 1980). This practice was found to be successful, and has been the industry norm for about fifteen years. During this period of storage samples are taken regularly for determination of germination energy, and once dormancy has fully broken the grain bulk is gradually cooled to an ambient temperature of about 5°C for storage over winter. Volumes of air of around $0.15\,m^3$/min per tonne are normally used to cool barley, applied either by

pressure fan through ducts under the barley or by suction from above the bulk. Suction has the disadvantage of being liable to air leaks in the adjoining structures of the storage vessel, so air could be sucked through the fans without passing through the grain bed. Blowing under pressure suffers the disadvantage that, if the grain is sufficiently warm, the air passing through the grain can absorb additional moisture, which will condense on the roof of the vessel if it is cold. Condensation droplets failing back on to the grain can cause severe spoilage on the surface layer of barley.

The types of storage vessel in use vary, but the cheapest form is flat-floored storage sheds with underfloor ventilation, followed by circular, steel, flat-bottomed bins with sweep auger clearing and underfloor ventilation. Concrete silos were constructed in the 1960s when costs were relatively low, but such silos are presently about five times more expensive to construct than flat stores. The flat stores are roofed with a gable angle the same as the natural angle of repose of the barley they contain, so that the wall height is kept to a minimum. Circular, steel, hopper-bottomed vessels are almost as expensive as concrete silos, but have the advantage of easy discharge. The major disadvantages of flat stores are in the discharge of barley from flat store and the inability to segregate different grades or varieties of barley. Normally barley is discharged by tractor shovel, which is slow and labour-intensive. Barley varieties can be segregated to a limited degree by insertion of temporary walls, but if this is done the capacity of the store is lessened.

In some cases, barley is pre-cleaned prior to drying. This pre-cleaning is usually a rough dressing to remove rubble, straw residues and awns from the green barley. Since wet barley is particularly difficult to sieve, most maltsters clean the barley after drying, when it is *en route* to storage silos, or after storage *en route* to steep. Cleaning prior to storage has the advantage of creating a dust-free environment in the silo, which improves aeration and working conditions generally but raises the problem of disposing of the dressings at harvest. The alternative of cleaning prior to steep allows for sale of the dressings over a longer time period, and this may have some commercial advantages, but the accumulation of dust and small grains within the bulk makes it more difficult for the aeration fans to give an even airflow through the barley.

When the barley is cleaned and graded, the fractionation of the grain size varies between maltsters. Generally, barley is initially sieved over 2.2-mm screens and all material less than 2.2 mm in diameter is sold for animal feed. The EBC recommended method used is Analytica EBC Barley 2.7 (1975). The main bulk may then be fractionated over a 2.5-mm sieve, depending on the final malt quality required. Barley between 2.2 mm and 2.5 mm in diameter amounts to 10–20 per cent of the total, depending on the crop year, and may have a little higher TN content than the head corn. When malted separately the small corns yield between 4 and 5 per cent less extract than the head corns, have a faster water uptake and modify about one day more rapidly than the less than 2.5-mm fraction. It is common to malt small corns separately because of the differences in steeping and germination requirements.

Barley intake quality control

Control of barley variety

The selection of barley varieties for malting depends on availability, cost and end use of the malt. As with barley, in 1995 the IOB Barley Committee drew up a prioritized list of desirable attributes in malted barley destined for the production of malt spirit. There are other attributes in malt destined to be grain spirit (see Chapter 3). The attributes for malt spirit are:

1. High priority
   - High hot water extract (hwe) over a range of TN contents
   - High fermentability content
   - Low gelatinization temperature
   - Rapid, even and complete modification
   - High predicted spirit yield (PSY).

2. Medium priority
   - Low malting loss during processing
   - The required pentose/pentosan balance
   - Resistance to mycotoxins.

The attributes desired in the malt feed back to the attributes desired in the barley (see above), and are strong guidelines for the breeders.

Each year in the UK, a list of recommended cereal varieties is produced by the Cereal Trials Advisory Committee (CTAC) of the Home Grown Cereals Authority (HGCA) (HGCA, 2000). It takes into account the agronomic and, for barleys, the malting performance, which is judged from extensive micromalting studies. From this list, the Scottish Agricultural College (SAC) produces a list for the northeast and northwest regions of the UK, within which lies Scotland (SAC, 2001). Varieties are 'approved' (IGB, 2001)**5 after further extensive micromalting, macromalting and distilling trials by the Institute and Guild of Brewing (IGB) Barley Committee (BC). Growers select suitable varieties from these lists for growing in Scotland and northern England, for acceptance for malting, providing they are of the required quality at harvest.

In 2001 the approved varieties were:

- Winter barleys – Regina
- Spring barleys – Chalice, Chariot, Decanter, Derkado, Optic and Prisma.

Some barley varieties that are on the IGB list and are important in the distilling industry produce cyanogenic glycosides, including methyl glycoside in their growing tips (Riffkin *et al*., 1989a, 1989b, 1990; Cook, 1990; Cook *et al*., 1990). Methyl glycoside is a chemical precursor of the compound ethyl carbamate, which is a carcinogen, and can form during the distilling process. Effective procedures can be adopted to prevent the accumulation of the precursor in new distillate, but the way ahead is for the breeding of barley varieties that do not biosynthesize the precursor. Decanter, which was approved in 2000, is an example.

Similar lists for English- and Welsh-, and for Northern Ireland-grown cereals are published by the National Institute of Agricultural Botany (England and Wales) (NIAB, 2001) and the Department of Agriculture of Northern Ireland (DANI, 2001). Other countries publish their own lists.

In addition, maltsters normally micromalt small batches of new varieties to check that they will produce the types of malts required in terms of adequate levels of diastatic power (DP), α-amylase and fermentable extract. Diastatic power is mainly a measurement of β-amylase activity, but includes some effects of α-amylase.

In the past 30 years, increasing numbers of barley varieties have become available to maltsters. Because of their varying degree of suitability for malting, reliable confirmation of identity is important. The grains of many modern varieties are so similar in appearance, even under the microscope, that traditional methods of identification based on morphological features now have a limited value for barley and are even less useful when applied to malt. In the 1980s the presence of blue aleurone pigment in some varieties (Kym, Atem and Halcyon) helped in their recognition, but the most useful methods available at that time were based on electrophoretic separation of storage proteins extracted from the kernels (Marchylo and Laberge, 1980). Electrophoresis was followed by the more rapid high-performance liquid chromatography (HPLC) (Marchylo and Kruger, 1984). More recently, it has become routine to use DNA fingerprinting (Shewry, 1993). A booklet with detailed descriptions of the cereal varieties is published annually by NIAB.

Control of barley storage, cleaning and grading

Harvested barley is dried to help its keeping properties. Maximum permissible moisture contents for short and prolonged storage are quoted as 14.8 per cent and 13.6 per cent respectively. The ongoing viability of a barley bulk during storage has been reported (Bamforth and Barclay, 1993) as a function of its initial viability, storage temperature and moisture content. A number of prediction equations and nomograms are available based on these parameters (Ellis and Roberts, 1980) – for example, this method indicates that barley with an initial viability of 98 per cent and moisture content of 12 per cent can be stored for over a year at 15°C without the percentage of viable corns dropping below 95.

Advances in cereal grain drying are well documented in the technical literature (Bakker-Arkema *et al.*, 1978; Foster, 1982), and various aspects of grain drying and storage are described in papers published by the Ministry of Agriculture, Fisheries and Food (MAFF) and SAC. Tables of equilibrium humidity are useful in drying calculations, and these are available for barley (Pixton and Warburton, 1971). Determination of grain moisture content is carried out before, during and after drying, and the information is used to monitor and control drying. A range of portable moisture meters is available, and their operational principle is based on the measurement of conductance, capacitance and infrared reflectance, all of which are proportional to moisture content, while specialized balances (where microwave or infrared radiation is

used to drive off the moisture) are also used. Both meters and balances must be calibrated against the standard IGB oven method. The stipulated grain sampling procedure and sample preparation methods must also be used (IOB, 1997).

Information regarding preventative measures against insect infestation and the treatment of grain storage problems is available in the government publications referred to above, and lists of approved pesticides are available (MAFF, 2000). It is essential to choose correct formulations, as otherwise severe reduction of grain viability can result. It is often specified that barley procured for the production of malt whisky must be free from pesticide residues.

The effects of various agronomic practices on grain quality have been discussed in some detail (see, for example, Briggs, 1978). Grain damage may not be obvious in barley received by the maltster, and various methods have been developed to show injuries arising from fungal infection, mechanical effects of harvesting, bad drying etc. Tests that indicate the ability of grain to germinate fully in a rapid and uniform manner are paramount. The most commonly used tests are described in the IOB *Recommended Methods of Analysis* (IOB, 1997).

Control of viability

Germinative capacity (GC) is the percentage of living corns that are present in a sample of grain; the figure is calculated from the percentage of corns that germinate after immersion for three days in a dilute solution of hydrogen peroxide (IOB, 1997). A quicker method is to immerse half-corns in a solution of a tetrazolium salt (triphenyl-tetrazolium trichloride). Visual examination is carried out after several minutes, and kernels with embryos stained to an agreed extent are deemed viable. A disadvantage of this method is that it can indicate viability in heat-damaged corns that are dead but still have residual hydrogen peroxidase activity. Germinative energy (GE) is a measure of the percentage of grains that can be expected to germinate fully if the barley is steeped normally at the time of the test. This test is carried out by germinating 100 corns on filter paper in petri dishes, using 4 ml water.

Control of dormancy

The difference in value between the figures for GC and GE indicates the degree of dormancy of the barley under test. In a variation of the GE test, 8 ml of water is used instead of 4 ml. The figure obtained by subtracting the 8-ml percentage from the 4-ml percentage is known as water sensitivity (WS), and is generally associated with dormancy. The extent of dormancy is a varietal characteristic, and is more common after a long, cool, damp growing season. It has been reported that high temperatures twelve to sixteen days after ear emergence promote dormancy, whereas if these occur at the later mealy stage dormancy is reduced (Reiner and Loch, 1976). When harvesting conditions are around 20°C, with barley moistures dropping to 10–14 per cent in the field, the dormancy period is quite short at around four to six weeks. Scottish weather conditions, with ambient temperatures around 13°C, rarely

lead to barleys with moisture contents below 15 per cent in the field, and so dormancy can sometimes be encountered in susceptible varieties.

Dormancy is a physiological condition that prevents seeds from germinating when they are presented with an otherwise satisfactory set of environmental conditions. If barley is required for malting during this period, the dormancy must be broken by holding the grain at moisture content of 12–13 per cent at slightly elevated temperatures. This procedure sometimes follows drying. WS is overcome by introducing air-rests into the steeping schedule.

The phenomenon of dormancy has not yet been fully explained, and a large technical literature indicates the ongoing effort that is being made to understand it. Some of the major considerations involve the microflora of the barley husks, operation of the pentose phosphate pathway during the early stages of metabolic activity, and the presence of inhibitory levels of endogenous phytohormones such as abscisic acid (Khan, 1977).

As well as occurring as a natural condition dormancy can be induced by certain drying procedures, and when this happens the GC (peroxide) test can yield viability figures that are lower than those obtained in the GE tests. To avoid induced dormancy it is essential to dry barley so that there is always a free transfer of moisture from the moist kernel when it is subjected to drying temperatures above ambient.

The germination properties of dried barley have been reviewed by Palmer and Taylor (1980). Dormancy is less apparent in germination tests carried out at lower temperatures, and the ratio of GE values determined at 16°C and 20°C has been used to distinguish dormant corns from those that have aged during storage, counts on the latter being unaffected by the temperature reduction (Bourne and Wheeler, 1985).

Control of pre-germination

Pre-germination is the term used when the corn starts to grow while the barley is still in the ear. It can occur when a spell of warm, damp weather precedes harvest. Barley containing pre-germinated corns develops unevenly. The affected corns are susceptible to drying damage and may become reservoirs of microbial infection. Barley containing over 5 per cent pre-germinated corns is unacceptable for malting for Scotch malt whisky.

Screening for pre-germination can be carried out by incubating a sample of ground barley with Phadebas reagent (dye-linked amylopectin). The presence of pre-germinated corns as indicated by their α-amylase content can be detected readily by the production of a soluble blue dye (Mathewson and Pomeranz, 1977).

To determine pre-germination on a percentage basis, a suitable number of half-corns are incubated in a solution of fluorescein dibutyrate, after which they are examined under ultraviolet light. Pre-germinated corns possess an enzyme activity (possibly lipase) that hydrolyses the non-fluorescent ester to yield fluorescein, which is easily detectable on the cut surface of the half-corn. This procedure is simpler than the European Brewery Convention (EBC) method, in which agar gels are used (Jensen and Heltved, 1982).

Control of total nitrogen content and corn size, and extract prediction tests

It is a high priority for malt whisky producers that barley procured for this purpose is of low percentage TN content. The starch content of grain, which is the main source of fermentable extract, is inversely related to its protein content. The TN content is measured using standard methods, such as the Kjeldahl and Dumas techniques.

Equipment for analysing large numbers of samples is commercially available, and variants based, for example, on alkali digestion have been introduced to increase the speed of analysis. Near-infrared reflectance (NIR) is popular for intake checks, and for these a choice of equipment exists.

Protein content is calculated as $TN \times 6.25$. The relative amounts of some endosperm protein fractions, separated by electrophoresis, have also been used to predict the suitability of certain barley varieties for malting (Wainwright, 1978).

Barley received at intake is usually subjected to a screening test to ascertain the percentages of corns within various diameter ranges. The procedure described in Analytica EBC (1975) is used. The barley sample is separated into fractions of less than 2.8 mm, less than 2.5 mm, less than 2.2 mm and the screenings, which pass through the 2.2-mm slots.

Thousand corn weight (tcw) is measured using the IOB method. Corn size is a varietal characteristic, and within a variety it is affected by season and location. Several malt properties are related to the size of the corn.

Prediction equations that relate potential hot water extract (hwe) to the TN content of the barley have been used for many years. Early versions involved additional determinations, such as husk content, and included tcw (Pollock, 1962). All included a 'varietal constant', the value of which had to be adjusted from season to season. Recent equations omit the husk content and tcw but still contain the varietal constant, which is derived from the results of micromalting trials using barleys of different TN content. The use of such predictive tests has been reviewed by Lloyd (1983).

Peat

Peat is decayed plant material that has formed over thousands of years, and is generally found in wetland areas. The type of peat formed depends on the plants involved (such as grasses or heathers) and the location (inland or maritime), e.g. Scottish mainland, Islay, Orkney.

Peat is used during barley kilning as a flavour source, not as a heat source. The location at which the peat was obtained, and its method of use, strongly affects the flavour of the spirit produced. During kilning the peat is burned, avoiding flaming, to produce a smoke called peat reek. It is burned during the early stages of kilning, so that the combustion products adsorb to and are absorbed by the malt. These effects happen in the range of 15–20 per cent moisture.**6 Careful control of combustion temperatures is necessary to achieve the desired degree of peating. Peatiness is caused by a spectrum of

phenolic compounds, and the amount within the final malt may be up to 80 ppm. The science and technology of using peat is discussed in the following section.

Malting

Steeping of barley

The science of steeping

The purpose of steeping is to initiate uniform germination and hydrate the endosperm to a level that is suitable for modification. Steeping conditions must subsequently allow satisfactory development of enzymes without encouraging undue malting losses from respiration and growth.

Grain structure and composition

The detailed anatomy of the barley grain and its chemical composition are comprehensively described in the brewing literature (Palmer, 1980; Duffus and Cochrane, 1993).

In a longitudinal section of the barley, two distinctive components of the corn are apparent; the small embryo and the large starchy endosperm (see Figure 2.1). The scutellum separates the embryo and the endosperm. The aleurone layer surrounds the endosperm, and the pericarp-testa and husk enclose the entire kernel. Embryonic axis and scutellum comprise 3 per cent of the grain dry weight. These tissues are rich in lipids (15 per cent), proteins
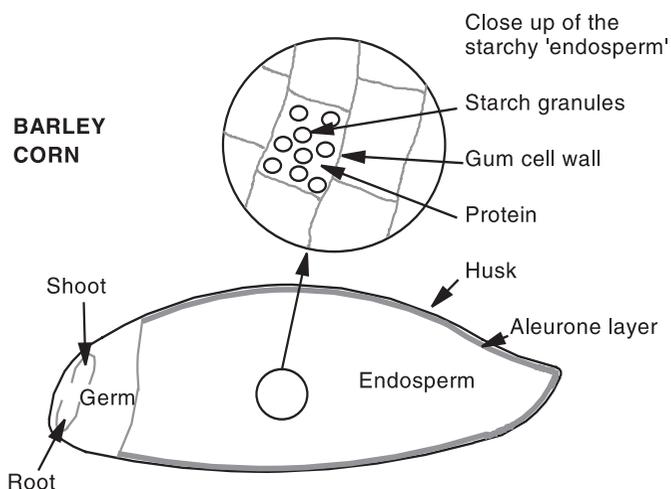
Figure 2.1
Barley grain structure.

(34 per cent) and soluble carbohydrates (23 per cent). The dry matter balance is made of minerals and insoluble cell wall material. Of the dry weight of the kernel 75 per cent is endosperm tissue, and this contains starch (85 per cent), protein (10 per cent) and cell wall material (mainly β-glucan with pentosan). Only 12 per cent of the kernel weight is accounted for by the aleurone layer, over 40 per cent of which is made up of cell wall material (mainly pentosan) and 20 per cent each of protein and triglycerides, while the balance is mineral constituents. Husk and pericarp make up the final 10 per cent of the dry weight. Husk materials originate from dead cell walls and contain high levels of silica.

Water uptake

Before barley is committed to steep it is tested in the laboratory to check that dormancy has been broken, allowing rapid and even germination to take place, and the degree of water sensitivity is ascertained. Before full-scale steeping, the water uptake rate is determined by carrying out microsteeping trials in which moisture content is measured at intervals. A guide to the duration of the first steep, in relation to temperature and the pattern of air-rests required to produce uniform chitting, is thus obtained.

In the first wet, the moisture content of the barley should rise to a value of between 32 per cent and 35 per cent. A subsequent air-rest, with ventilation, enables the corns to absorb the adhering surface film of moisture under relatively aerobic conditions. Further periods of immersion, with or without intermediate air-rests, are carried out to reach the final moisture content.

Water uptake rate, particularly during the initial stages, varies with barley variety, grain size, season and geographical location. The TN and glucan content of the barley appears to have little effect on the rate of uptake. Initially, large corns take up moisture faster than smaller ones. At the end of a prolonged steep, the smaller corns have the highest moisture content. Generally, the most suitable barley varieties for malting are those that take up moisture at the fastest rate.

When the moisture content of the grain exceeds 25 per cent dehydrogenase activity becomes apparent, and at higher moistures changes in the carbohydrate, lipid and protein composition of the embryo are produced by the action of other enzyme systems.

Uptake of water during steeping is generally regarded as a three-stage process (Brookes *et al.*, 1976). Initial adsorption, which is a purely physical process, continues rapidly until a moisture level of about 32 per cent is reached. Within the range 5–35°C water uptake is a direct function of temperature, and at 12°C this stage of uptake can take up to 10 hours. The second phase is a lag phase, which can last for a further 10 hours, during which time the rate of uptake is reduced to almost zero. A third phase, one of active water uptake, continues at an increased rate to produce maximum moisture content after a total steeping time of about 50 hours.

During steeping, the various structural components of the barleycorns take up water at different rates. Soon after wetting the moisture content of the husk rises to 50 per cent, and this value is retained during immersion. The moisture content of endosperm tissue increases steadily during steeping, reaching a maximum value of 38–40 per cent after about 70–80 hours. Embryo moisture level rises rapidly to 60–65 per cent over 24 hours, then, with the advent of germination (chitting), rises to a maximum of about 85 per cent. The moisture content of the scutellum changes in a similar way, but always remains lower than that of the embryo. After 24 hours it reaches 55 per cent, and then slowly increases to a maximum value of 65 per cent. During steeping the bulk of moisture is taken up by the husk and endosperm, which are both relatively inactive in metabolic terms. As a result, the type of steeping employed has no great effect on overall moisture level. Vigorous aeration or spray steeping increases the rate of water uptake by the embryo and scutellum, and thus the rate of development of the germ; this significantly affects the overall pattern of moisture distribution in the steeped corns.

Endosperm tissue must be correctly hydrated before it can be modified. The structure of barley endosperm is non-uniform, and micro-regions exist where packing densities and ease of solvent penetration are different from those of surrounding areas. With some difficult or low-grade barley, the effect of spray steeping is to encourage rapid embryo development before sufficient endosperm hydration has taken place. During germination, the effects of this deficiency are exaggerated by the transfer of moisture from the endosperm and associated aleurone tissue to the embryo. It is better to avoid such malting barley.

Respiration

Shortly after the first wet of the barley, respiratory activity swiftly increases, both in the embryo and aleurone tissue. Oxygen is rapidly removed from the steep water and there is a simultaneous release of carbon dioxide and ethanol until the end of steeping. Ventilation during the immersion and air-resting periods both supplies oxygen, which is essential for germination, and removes carbon dioxide, which has an inhibitory effect on growth. It also minimizes the build-up of ethanol in the grain. Ethanol can retard the germination rate and produce non-uniform chitting. Until the corns chit, oxygen supply to the centres of respiration is limited. For that reason, the respiratory quotient (RQ) can remain above 1.0 for at least the first 24 hours of steeping. Subsequently, it drops below unity. Heat release is linked to respiratory activity, and during the later air- resting periods ventilation has to be stepped up to control the temperature of the grain.

Germination inhibitors

Abscisic acid (ABA) is present in mature, ungerminated barley, the highest concentration occurring in the embryo (Yamada, 1985). During steeping ABA passes into the steep water from the husk, and its overall concentration in the grain diminishes. A fraction of ABA seems to be destroyed so the level of ABA

in the grain is reduced faster when the steep is aerated. The onset of germination coincides with a fall in the level of ABA to a relatively low value. ABA inhibits gibberellin-induced α-amylase synthesis in the aleurone layer, but the role of this compound in the early stages of germination is not yet fully understood. Gibberellin synthesis is delayed until germination has commenced, and at the end of steeping this hormone is present at just detectable levels.

During steeping a number of phenolic compounds, amino acids, sugars and salts are leached from the corns. These materials make up about 0.5–1.5 per cent of the corn's dry matter. Their continued accumulation in steep water can retard the metabolic rate of the grain, but they are removed when the steep water is changed.

When barley is steeped to higher moisture contents, dry matter losses arising from rootlet growth and respiration during subsequent stages of malting increase. Enzyme development and endosperm modification are usually favoured by this condition, particularly if the initial uptake of water occurs rapidly. In immersion steeping, rather than spray steeping, early chitting leads to higher hwe values.

Effect on malt quality

Oversteeping, especially with inadequate aeration, can lead to ragged germination, as each barleycorn has its own specific moisture requirement. The required levels of enzymes can be produced by using multiple wettings, with one or more air-rests, to achieve a moisture content of at least 46 per cent. Generally, pneumatic maltings require higher malt moisture levels than floor maltings. Abrasion and related processes, in which the distal end of the barleycorn is damaged, allowing faster ingress of water, can be used. They have been shown to give rise to faster water uptake rate and modification. In the brewing industry, abrasion is used along with gibberellic acid to speed modification, but this is not the case in Scotch malt whisky production, where additives are forbidden.

Wet casting procedures (hydraulic transfer), in which steeped grain is pumped from the steep to another location, can damage the embryo and upset the course of malting (Yoshida *et al*., 1979). Modification is delayed by approximately a day, some enzyme levels do not recover from the process, and fermentability is reduced. Modern practice favours moving the cast barley on rubber conveyer belts.

After drainage of the final water a film of moisture is retained by the steeped barley, and this restricts gas exchange. The rate at which this film is removed, either by adsorption into the grain or by transfer to the surrounding air, is critical for the onset of respiration and growth. Water in this film can account for 2–5 per cent of the corn's moisture content.

The technology of steeping

Basic steeping requirements are a supply of suitable water at the required temperature and a supply of clean air to remove excess heat, metabolic

waste products (such as carbon dioxide and ethanol) and endogenous germination inhibitors.

The floor maltings still in use demonstrate the limitations imposed on earlier maltsters. The barley was steeped in a single cast-iron cistern with no aeration or carbon dioxide extraction for upwards of 48 hours, and with not more than a single change of water. The water was drawn straight from the stream or distillery reservoir at 5–10°C, and was not heated to a suitable temperature. Under those conditions, germination counts at casting were low and uneven. In this respect, the discovery of water sensitivity (Essery *et al.*, 1954) and how to overcome it was of greater significance to the distiller–maltster than to southern Scottish or English maltsters.

Scottish maltsters and distillers rapidly adopted new concepts in maltings, and newer designs of steep vessels were quickly incorporated into malting plants in the early 1960s. These plants were usually of the conical type, with in-steep aerators and carbon dioxide extraction. As batch sizes in most floor maltings, Saladin boxes and drum maltings were similar (around 20–30 t capacity as barley), this type of steep vessel was quite acceptable. The problems associated with uneven temperature distribution and airflow during air-rests in larger conical vessels was avoided.

Steeping techniques in the newer steeping/germinating/kilning vessels (SGKVs) differ from traditional transferred steeps and, since the steeping and germination phases tend to overlap in such maltings, they are described below in the Germination section.

The steeping schedules employed in conical steeps are similar to those used in the manufacture of brewing malts (Brooks *et al.*, 1976; Axcell *et al.*, 1983). If a moisture content of about 30–35 per cent is required in the first wet, to initiate germination, and an initial air-rest of 12–16 hours at 13–15°C is sufficient to promote an even chit, the subsequent wets and air-rests are adjusted to give a casting moisture of 45–47 per cent, with a uniform chit of over 96 per cent. The casting moisture may vary according to the variety of barley, its TN content, and the degree of steeliness (hardness) or mealiness (porosity) in the endosperm. Given that these properties vary from year to year, and even between different regions in the same crop year, the maltster will always have to make minor changes to steeping cycles to maintain uniform water uptake and germination.

In the 1980s, large flat-bottomed steeps of the Nordon type were introduced. This type of steep is preferred when the barley tonnage exceeds 50 t, because a more even temperature and aeration profile can be maintained over the grain bed. With enough fan power, the first few hours of germination can proceed in the steep vessel before transfer to the germination vessel. Care must be exercised in not damaging the grain if rootlets have started to emerge. The 'Giracleur' discharge on this type of vessel is gentle with regard to physical damage to the grain, but subsequent conveyance to the germination vessel must be carefully managed to avoid too much rootlet loss.

Transferring from conventional conical steep is best done under gravity as a vertical dry discharge to the germination vessel. In older Saladin plants, the steeped grain was sometimes pumped over as aqueous slurry to the germination box. Hydrostatic pressure applied to chitted malt can retard the rate of

germination (Yoshida *et al.*, 1979) just as much as the mechanical damage mentioned above, and should be avoided.

Germination

The science of germination

For malt spirit production, the purpose of germination is to maximize fermentable extract by promoting both endosperm modification and the development of amylolytic enzymes. To achieve these objectives, the progress and uniformity of germination are controlled by managing optimum moisture levels within the corn, supplying air, removing carbon dioxide and excess respiratory heat, and mechanically turning the grain to prevent matting of rootlets.

Penetration of the pericarp-testa by the coleorhiza marks the start of germination (chitting). Development in the embryo leads to the production of rootlets and growth of the acrospire. Enzyme synthesis and activation take place during the germination stage, and the cellular structure of the endosperm is modified by the action of these various enzymes while the grain respires aerobically. Barley variety, TN content, corn size and conditions of steeping all affect the rate of modification.

Before processing starts with any bulk of barley, micromalting trials are carried out to attempt to predict the performance of the barley during germination. Tests may be conducted at different germination temperatures or with malts at different levels of moisture content. Daily sampling and analysis of the growing malt can then indicate the rate at which modification and enzyme development are proceeding. The uniformity of germination and the progress of growth can also be observed, and information gained about malting loss.

The exact site of enzyme synthesis and details of the initial pattern of endosperm modifications have been matters of some controversy for over a hundred years. The situation remains unresolved, but it is generally agreed that the major site of synthesis and secretion is the aleurone layer, under the influence of gibberellin released from the embryo. Scutellar tissue appears to contribute, but it is more likely that in the studies involving isolation of such tissue there has been contamination by aleurone cells. It is agreed that modification of the endosperm proceeds from the area nearest to the embryo. Gibberellin is synthesized in the embryo, by a process requiring oxygen. Studies indicate that the endogenous gibberellin is GA1, and that gibberellic acid (GA3) is present in smaller amounts (Yamada, 1982). Following the migration of gibberellin to the aleurone, a number of hydrolytic enzymes are synthesized and secreted into the endosperm in an orderly sequence (MacLeod, 1979).

Other enzymes, many of which are already in the endosperm, do not require the intervention of gibberellin for their activity. For example, GA is required for the aleurone synthesis of α-amylase and limit-dextrinase, whilst synthesis of barley β-glucanase and α-glucosidase are enhanced by its presence; β-1-3-glucanase and phosphatase, on the other hand, rely on GA only for their secretion. In contrast, β-amylase and carboxypeptidase are present

in the endosperm and have no direct GA requirement. During germination, latent β-amylase is converted into the active form by the action of proteolytic enzymes.

Fermentable extract is almost exclusively derived from the starchy endosperm of the germinated grain. Release of this material, by the process of modification, depends on the combined action of several hydrolytic enzymes on cell walls and matrix proteins. In the endosperm, starch occurs as large (20–30 μm) and small (1–5 μm) granules. Aspects of their release are well covered in the literature (Bourne, 1999; Lynn *et al.*, 1999; Stuart *et al.*, 1999). Although the small granules are more numerous, they represent a minor weight proportion of the total starch. Both types of granule are embedded in a matrix of reserve protein composed of hordeins and glutelins. This conglomerate of starch and protein is contained within a cellular envelope composed of 70–75 per cent glucan, 20–25 per cent pentosan, and other minor components. Of the cell wall glucan, 70 per cent is β1,4 linked, the remainder being linked in the β1,3 position. Modification of the endosperm follows the release of β-glucanases, endo-proteases and pentosanases from the aleurone layer. A significant amount of β-glucan is degraded during malting, along with 35–40 per cent of the reserve protein, but less than 15 per cent of the starch is broken down. Incomplete modification of the endosperm will result in reduced extraction of starch during mashing, and a reduced susceptibility of the starch to enzymic attack.

In terms of modification, much research effort has been made to isolate the factors that distinguish good malting barley. Malting quality is a varietal characteristic, and is not related simply to either glucan or protein content. Malting varieties usually modify faster than non-malting varieties, even when their TN content is higher. Traditionally, mealiness and steeliness in the barley endosperm have been used to judge quality. Steely grains (which malt less readily) generally contain more nitrogen than corresponding mealy grains, but the relationship between endosperm nitrogen and steeliness is not consistent between varieties. It is possible that in poor malting varieties there are limited areas of high-nitrogen endosperm that display an extreme degree of steeliness and modify at a slower rate. The polypeptide composition of reserve proteins (hordeins) in different varieties appears to be related to their malting potential (Wainwright, 1979). High glucan content does not always signify poor malting quality; barleys with relatively high levels of cell wall materials modify successfully if adequate β-glucanase levels can be achieved. In assessing this provision, it is important to distinguish between free enzyme and unsecreted non-functional enzyme (Palmer *et al.*, 1985). The application of electron microscopy has had a limited success in relating endosperm microstructure to malting quality. Within the endosperm, the packing density of the starch-containing cells varies, thus affecting ease of hydration, and differences are apparent between varieties in the density of the small starch granule-protein matrix. In areas of well-modified endosperm, cell walls are absent and, the amount of protein matrix is greatly reduced.

Increasing the temperature of germination initially improves the rate of modification, but final extracts are lower. However, the modification value

for kilned malt, as expressed by the differences between values for hwe using fine and coarse mill settings (fine–coarse difference), can remain unsatisfactory. This indicates that overall modification has been restricted. High germination moistures, on the other hand, increase endosperm hydration, which helps difficult barleys to modify. An adequate supply of oxygen is essential for modification, particularly during the early stages of germination. If carbon dioxide is allowed to accumulate, modification is restricted.

In malt for distilling, the most important enzymes are those involved in the conversion of starch into fermentable sugars. This group comprises α and β-amylases, limit-dextrinase and α-glucosidase.

The endo-enzyme α-amylase specifically attacks α-1,4 glucose linkages; the α-1,6 linkages are bypassed and are not hydrolysed. This enzyme also reduces the viscosity of starch suspensions and produces dextrins containing up to twelve glucose units, with only a slow production of glucose and maltose. Amylose and amylopectin are converted to the extent of 70 per cent and 55 per cent respectively. The α-amylase is absent from ungerminated barley and depends on gibberellin for its synthesis in the aleurone layer during germination. A natural α-amylase inhibitor occurs in the endosperm of ungerminated barley, but the level of this material does not change during germination; its role may be to retard the digestion of intact starch granules in the endosperm. While it is heat sensitive, a proportion may survive kilning and mashing (Munck *et al.*, 1985; Weselake *et al.*, 1985).

By contrast, β-amylase is an exo-enzyme, removing maltose units from the non-reducing ends of α-1,4 linked chains. The substrate viscosity is slowly reduced with the formation of linear dextrins, and maltose is rapidly produced. Amylose will eventually be converted completely into maltose by α-amylase, whereas amylopectin is 75 per cent converted; α-amylase will not attack raw starch granules. The enzyme is present in ungerminated barley mainly in a latent form, which is converted to free enzyme during germination.

Limit-dextrinase specifically attacks the α-1,6-glucosidic linkages in, for example, amylopectin, β limit-dextrins, and starch-derived α limit-dextrins. The debranching enzyme effectively increases the fermentability of worts and subsequent alcohol production. This enzyme is absent from barley, and is synthesized during germination in parallel with α-amylase (Lee and Pyler, 1984).

Maltase (α-glucosidase) liberates β-D-glucose from the non-reducing end of α-1,4 linked chains. It will also release glucose, which is linked terminally in the α-1,6 position. This enzyme occurs in the aleurone layer and embryo of ungerminated barley.

Within the range 12–20°C, higher temperatures increase initial rates of enzyme development but suppress maximum attainable values. Procedures in which germination starts at a relatively high temperature and finishes at a lower temperature appear to have no advantage for the distiller, but higher enzyme levels are usually produced at higher moisture contents. Barley varieties differ in their ability to develop enzymes, and this ability is influenced by seasonal changes and geographical location.

Respiration and growth

Respiration supplies the energy for the synthetic processes that take place during germination. At the start of germination respiratory activity is divided equally between the embryo and the aleurone layer, but as germination progresses a higher proportion of the activity occurs in the embryo. Depending on the temperature of germination, respiration rate steadily increases, reaches a peak after three to four days, and then slowly declines. Initially, respiration is faster at higher temperatures. Carbohydrate is the main substrate, which is converted to carbon dioxide, and up to 4 per cent of the initial barley dry matter is lost by respiration during germination. Respiratory activity is generally higher in barleys germinated at higher moisture levels. If oxygen supply is limited or carbon dioxide is allowed to accumulate, respiration is restricted.

During germination, adequate ventilation is required to disperse the heat produced by respiration. Part of the energy supplied by respiration is used by the barley to produce rootlets and acrospire, which can account for 4–5 per cent of the original barley dry weight. Malting conditions affect growth and respiration in a similar manner, although there is not necessarily a strictly parallel development. Malting loss comprises losses that arise from steeping, rootlet growth and respiration, and it accounts for a proportion of material that otherwise could have been fermented to alcohol.

Moisture control

In pneumatic maltings, an unavoidable loss of moisture takes place from the germinating grain because of respiratory heat release. A thermal gradient is produced in the ventilating airstream, which increases its moisture-holding capacity. Regardless of the initial air humidity, rapid transfer of moisture takes place from the malt to the surrounding air. Within the corns, water is transported from the endosperm to the embryo at an increased rate, and this has an adverse effect on the modification process. Although the former moisture level can be reinstated by spraying, the distribution of moisture will be different because the water is taken up by the rootlets and the outer layers of the corn. Best practice is to achieve the required moisture content by steeping.

Fermentability

The main object of malting is to maximize the production of fermentable extract in the malt. During germination, hot-water extract (hwe) as determined by the IGB method (IOB, 1997) rises steadily to a maximum value, then declines as an increasing proportion of soluble material is translocated to the embryo for growth. Before this maximum is achieved the potentially fermentable fraction of this extract has already started to decline, due to a change in the relative amounts of carbohydrate and nitrogenous material, in favour of the latter. Thus, fermentable extract reaches a maximum value before the peak value for hwe is achieved.

Concomitant with the increase in hwe there is usually a steady decline in the difference between values for hwe using coarse and fine mill settings. This

difference, which is inversely related to the degree of physical modification, continues to decline beyond the point of maximum hwe (Dolan, 1983). If the germination process is continued until minimum difference is achieved, this can result in the loss of extract. Continuing to a lesser degree of modification, for example to the point of maximum hwe, will similarly incur a loss of fermentable extract. At the point of maximum fermentable extract it is likely that an adequate level of modification will have been achieved, as spirit yield predicted from this value correlates with that obtained in distillery practice. Furthermore, by curtailing the length of the germination period unnecessary malting losses are avoided, thus increasing the yield of spirit based on original grain. This same argument does not necessarily apply to the production of malt for grain spirit, where enzyme levels are more important than fermentable extract. In terms of other analytical parameters, malt fermentability is inversely related to cold-water extract and wort soluble nitrogen (Bathgate *et al*., 1978).

Measurement of green malt quality

By tradition, the extent of acrospire development and the ease by which the kernel endosperm can be 'rubbed' between the finger and the thumb is used as a guide to the progress of malting. During germination, grain moisture content and temperature must be monitored continuously. In pneumatic maltings it is unlikely that any deficiency of oxygen or local accumulation of carbon dioxide will occur, but the latter can be checked by use of portable gas analyses. Levels of α-amylase and DP (which is mainly β-amylase) are measured in green malt by IOB methods. Typical values for green malt DP and amylase are 70–80 IOB**7 and 20–30 DU (dextrinizing units) respectively (expressed at 44 per cent moisture). The progress of modification can be followed by treating sectioned corns with a chemical stain such as calcofluor, which specifically combines with β-glucan in the endosperm cell wall. Unmodified areas of endosperm fluoresce under ultraviolet light after treatment with the dye (Aastrup and Erdal, 1980). Other tests, which are applied to kiln-dried malts, are referred to in the Malt section, below.

The technology of germination

The malting plant used to make malt for Scotch malt whisky is as diverse as in the rest of the UK malting industry. Almost every type of plant, from floor maltings to completely automatic SGKVs, is in use. It is useful to compare the effects on the malting grain of three different types of plant: completely separate vessels for each process; combined germination and kilning (GKV); and combined steeping, germination and kilning (SGKV) vessels.

Separate steeping, germination and kilning

This is the commonest type of plant in older maltings, where the germinating vessels are either Saladin boxes, or drums. Some of the first maltings in the UK

to use Saladin plants (1957–1962), Galland drums (1905) and Vickers Boby drums (1968–1972) were in the north of Scotland.

The Saladin plants are of the concrete box type, fitted with either contra-rotating spiral screw or Van Caspel type bucket turners. Humidification and pressure fan ventilation are designed to ensure that grain temperatures of 14–20°C can be maintained across the bed, with no more than 0.5 per cent moisture loss per day. With the facility to spray water on to the grain via booms fitted to the turner gantry, moisture levels can easily be maintained in these vessels; however, spraying is to be avoided.

Control of humidity in open vessels cannot match that in the completely enclosed drum. While the capital installation costs of such vessels are extremely high, the simple drum-turning mechanism and relatively low maintenance costs still make the drum a very effective germination vessel. The inversion of the grain, under very gentle conditions, means even grain temperature, minimum rootlet damage and minimum moisture loss. In addition, heat is dispatched to the ambient surrounding air by conduction through the steel walls; so a lower air volume is required to maintain the temperature differential across the bed. The major disadvantage is the relatively small batch size (30–50 t), a primary aspect considering the high capital and energy costs incurred in moving small batches of malt to and from the kiln. The drum maltings in operation are all of the Boby type, which have improved aeration/humidification control compared with the original Galland design.

Combined germinating and kilning vessels

Some producers of Scotch malt whisky malt operate germination vessels that can also operate as kilns. Generally such vessels are circular in construction, have the capacity for batch sizes of 100–300 t as barley steeped, and are fitted with two sets of fans – one for germination and the other for kilning.

Compared with traditional germination vessels, GKVs are fitted with a larger under-deck plenum, to equalize the pressure of kilning air. During germination the duct introducing kiln air is closed, and it is opened during kilning. Some vessels have turners that rotate on a boom, and some have fixed turners and rotating floors. The rotating turners can be lifted out of the malt during kilning. This is an advantage, because stationary turners constantly set in the malt can result in uneven drying owing to faster airflows around the spirals and localized overheating by thermal conduction through the steel shafts.

Airflows during germination are similar to those used in Saladin boxes (around $15 \, m^3$/min per tonne), and during the final hours of germination the humidification plant may be turned off to allow a partial drying out of malt before commencing kilning. Because of the larger amounts of air employed and the large vessels used in these vessels, humidification control is more difficult and moisture losses are generally higher than in small Saladin and drum plants.

Retention of moisture after steeping is extremely important for proper endosperm modification (Palmer *et al.*, 1985), as additional spray steeping cannot

guarantee that water applied during germination will penetrate the endo-sperm. While final moistures can be adjusted by spraying shortly after casting, later application of water, when the rootlets are fully formed, increases the moisture content of only the culms and the embryo. Spraying late in the germination period also has the disadvantage of maintaining rootlet develop-ment at a stage when withering is useful in limiting malting loss.

In malting for malt whisky, the main requirement is a high spirit yield. Bathgate demonstrated that the fermentable extract in malt is carbohydrate-limited, so if carbon is lost through a high respiration rate then the potential spirit yield from the malt is depleted (Bathgate *et al*., 1978). In other words, distilling malt requires long, cool germination with the minimum heat gen-eration. Germination periods of five to seven days at 15–20°C are common in all malting plants that have separate steeps.

Combined steeping, germination and kilning vessels

Faster throughput can be achieved in a combined SGKV because the steeping and germination phases can be overlapped. To gain maximum use of a sepa-rate steeping vessel, it should be utilized as often as possible during the process cycle. One steep vessel should serve at least two germination vessels, with a maximum 72-hour turn-round in a six-day germination cycle. If the barley batch is steeped in the germination vessel then there is no limit to the time at which water can be applied, other than the period required to build up the required volume of water, and the filling and emptying rates. When a number of SGKVs are employed and they have large capacities, the daily volume of water required can become the limiting factor in the steeping cycle employed. The vast amount of water required is due to the void volume in the air plenum under the deck. This empty space may amount to 30 per cent of the water required, and is water that is never in contact with the grain. Such malting plants were commonly built in Scotland, where water was relatively cheap and disposing of effluent (mainly by pumping out to sea) was not difficult or expensive. Effluent problems can arise with the disposal of sus-pended solids if the under deck area is not kept thoroughly clean after kilning. Malt rootlets (culms) fall through the wedge wire floors of the SGKV during malt stripping, and these must be removed before the plenum is flooded during the next steep. These are minor disadvantages compared with the low capital cost of installation, and the flexibility of the plant in terms of both barley drying and malting and the high throughput rate.

If the barley has high GE, only two immersions in water may be necessary in these plants. The target moisture content of the barley after the first wet is higher than it is for plants with separate steeps. A first wet time of 18–24 hours, giving a moisture content of 36–42 per cent, is common. This relatively high moisture content allows for a long air-rest, during which the full germina-tion airflow can be applied. With flat beds, air distribution and temperature control are first-rate and rapid chitting takes place. The air-rests cannot be differentiated from germination and may extend to up to 36 hours before the second wet, which takes the moisture content up to the required 46–47 per cent.

Kilning

The science of kilning

The purpose of kilning freshly produced or green malt is to stop biological activity when the required enzyme levels and degree of modification have been reached, and to produce a dry storable product that can be milled to the grist required for mashing.

Undesired flavour components are removed during kilning, while other desired ones appear – either from existing precursor chemical compounds or from extraneous sources such as peat smoke.

The physics of malt drying is similar to that of barley, and mathematical treatments of both are often found together in the literature, along with worked examples (Sfat, 1965; Briggs et al., 1981). At the start of kilning the temperature of the green malt rises above ambient, but remains well below that of the air-on temperature because of evaporative cooling. Water evaporates from the malt in an unhindered manner until a moisture content of about 20–25 per cent is reached. This phase of kilning, known as the free-drying stage, ends at the break – the point at which the drying front progressing through the malt bed in the direction of the airstream breaks at the surface. After the break water remaining in the malt is released less readily, and as a result the temperature of the malt rises. This second, diffusion stage is continued until a moisture content of about 10–12 per cent is reached. At this point most of the water is what is termed 'bound' and further moisture must be driven off by increasing the air-on temperature, resulting in a moisture content of 4–6 per cent.

The biochemical activity of the embryo may continue until the malt temperature exceeds $50°C$. During the free-drying stage, modification and enzyme synthesis continue. After the break, enzymic activity declines and some enzymes are denatured, this decline increasing during the final stages of kilning. The more thermolabile enzymes are more susceptible to damage from heat when the moisture content of the malt is high (Narziss and Rusitzka, 1977a, 1977b).

Malt modification continues during the free-drying stage of kilning. Starch, β-glucan and proteins are enzymically broken down and their products are translocated to the embryo, which is still actively respiring. As malt temperature increases, the biosynthetic activities in the embryo slow down and low molecular weight compounds such as sugars and amino acids build up in the grain. These compounds make up the 'cold-water extract' of the malt.

The progress of physical modification can be monitored by staining corn sections with the reagent calcofluor, which reacts specifically with cell wall glucans. The progress of biological malt modification can be monitored by following the fall between values for fine and coarse SE (f/c difference) figures. In the 1980s the friability meter (friabilimeter) gained popularity as a means of assessing the progress of modification, and homogeneity, in a sample of malt (Baxter and O'Farrell, 1983). It is in wide use today, but must be used with caution (Cooper, 1986a, 1986b; Woodward and Oliver, 1990) and calibrated regularly.

It is particularly important in distilling to preserve the amylolytic enzymes during kilning. If kilning is managed correctly the activity of these enzymes continues beyond mashing, because there is no wort boiling stage, and into fermentation. Up to 20 per cent of the total alcohol yield can be due to enzymic activity during this period of secondary conversion (Sim, 1992; Sim and Berry, 1996).

The kilning cycle has a strong effect on the malt's fermentability, and in the 1990s air-on temperatures were reduced once the benefits were realized. A typical air-on cycle is now 12 hours at 60°C, 12 hours at 68°C and 6 hours at 72°C, whereas previously typical air-on temperatures were 58°C, 72°C and 78°C. It is very important that there is no temperature ramping in the early stages of kilning as the thermolabile enzymes are at their most vulnerable then, when the moisture content is high. The advent of indirect kilning, which was widely introduced in the 1980s as a means of preventing nitrosamine formation, also led to malts having higher fermentabilities, because of the removal of the passage of combustion compounds through the malt. Malt produced at a Scottish Saladin maltings that has installed indirect kilning, a mechanical kiln loader that gives an even malt bed, and uses a modern kilning cycle, typically has fermentabilities (%F) of 88–89 per cent. A typical figure prior to the changes was 86 per cent.

The laboratory-determined %F is used along with the value for the coarse hwe (%SE7) value to calculate the percent fermentable extract (%FE), from which a predicted spirit yield (PSY) can be calculated. The laboratory method for determining a malt sample's fermentability can suffer from variations that may be introduced by the yeast, but the prediction equation has been shown to be very accurate (Dolan, 2000). For malts where sulphur has been burned during kilning (see below), fermentability results may be artificially depressed because of the relatively low pHs that are produced in the unbuffered laboratory mash. With these malts, spirit yields generally exceed those predicted in the test.

The range of variation in enzymic activity during kilning is large, as outlined below.

During the early stages of kilning, the activities of α- and β-amylase, endo-β-1,3:1,4 glucanase and the endo- and exo-peptidases continue to increase. In the final malt, because of the relatively mild kilning conditions used in distilling practice, levels of α-amylase and the peptidases may exceed those in the original green malt. Only slight losses occur in the final levels of β-amylase and limit-dextrinase, whereas up to 80 per cent of the endo-β-glucanase activity is lost. Maltase (α-glucosidase) is very susceptible to temperatures exceeding 50°C, and peroxidase and catalase are among the most easily destroyed enzymes. Acid phosphatase, 6-phytase and lipoxidase lose activity even under mild kilning conditions, whereas lipase activity survives.

Concentrations of compounds other than enzymes change during kilning. The amounts of amino acids and some reducing sugars decrease. Formation of melanoidins from these two groups of compounds, a common reaction, is enhanced if the malt is overmodified or is allowed to stew by raising its temperature while it is relatively wet.

Sulphur dioxide

The combustion of sulphur containing fuel oil, which may have up to 3 per cent w/w sulphur, produces sulphur dioxide (SO_2). In directly fired kilns the SO_2 is rapidly taken up by green malt, mainly when the malt moisture content is above 20–25 per cent. Levels are retained by the grain throughout subsequent kilning, even when the initial period of exposure has been less than two hours. Standard laboratory distillation methods indicate only a fraction of the total SO_2 present in the malt. Other techniques show that free, combined and non-distillable forms exist in the grain. The chemical state of combination of the SO_2 present determines its effect on the wort's properties. Direct kilning may also cause the synthesis of oxides of sulphur that can produce a black-flecked appearance, known as 'magpie', on the surface of malt near to the kiln floor. Sulphur residues on malt are detectable chemically as sulphate, and correlate with the pH of the wort. Sulphur dioxide is sometimes introduced into direct gas-fired kilns, where none is produced by combustion, to combat the formation of N-nitrosodimethylamine (NDMA). For low-gravity worts, pH is lowered when malts containing SO_2 are used – as in the laboratory method. Soluble nitrogen levels are increased and the activities of proteolytic enzymes and β-amylase may be enhanced by the reducing action of sulphur dioxide, whereas the activity of other enzymes is reduced (Sim, 1983). Sulphur dioxide causes the colour of malt to lighten, and reduces the level of surface microbial infection in the malt (Flannigan, 1983).

Nitrosamines

Kilned malt is a potential source of volatile nitrosamines, which could ingress at low levels into the distilled spirit – although this is very unlikely, given the present clear understanding of the science and how to prevent formation and entrainment. Nitrosamines are formed when nitrogen oxides, produced by the combustion of fuels during kilning, react with hordenine or related compounds such as dimethylamine present in green malt when the correct reaction conditions exist. The formation of NDMA can be effectively blocked as described above. A review of nitrosamine chemistry was published in 1986 (Wainwright, 1986). When peat is used in malt kilning extreme care must be taken to avoid flaring, to prevent nitrosamine formation.

Flavours

During kilning new compounds are formed and others, which exist in the green malt, are destroyed. These chemical changes affect the sensory quality of the final distilled spirit. The types of reaction considered to be of importance during kilning include those based on the enzymic and chemical oxidation of unsaturated fatty acids, the combination of free amino acids and reducing sugars, and the thermal degradation of precursors such as S-methyl methionine, which are synthesized during germination (Tressl *et al.*, 1983).

The chemistry of the formation of flavour compounds is complex, and becomes increasingly so as kilning temperatures are raised. There are detailed reviews in the literature (Bathgate, 1973).

The distillation stage is of paramount importance, both as a flavour formation process and as one of separation. Quantitatively, the major flavour-potent compounds (congeners) are produced during fermentation. In sensory terms, many compounds that make significant contributions are often present in only trace amounts. Much work remains to be carried out regarding linking whisky character to chemical composition, and tracing the origin of these components (see Chapter 3).

Linoleic acid (18 : 2) is the most abundant fatty acid present in the barley grain, and malt contains two lipoxygenase enzymes that convert this compound to 9- and 13-hydroperoxides, which are then isomerized and modified via ketols to produce trihydroxy fatty acids. These in turn can serve as precursors for the formation of aldehydes such as hexanal and trans-2-hexenal (Gardner, 1975).

A range of important flavour compounds is formed by the Maillard reaction, in which free amino acids and sugars combine and undergo chemical transformation by Strecker degradation and other routes, leading to the production of unsaturated aldehydes, furans and pyrroles etc. The formation of dimethyl sulphide (DMS) is an example that illustrates how a diversity of factors, ranging from barley variety and nitrogen content, storage time, conditions during steeping and germination and, finally kilning conditions, can all affect the level of a compound that will eventually appear in the distilled spirit (Anness and Bamforth, 1982). The mechanisms of DMS formation are better understood than those that produce the chemically related di- and trisulphides, which are of more sensory significance. Their formation appears to be influenced by residual levels of sulphur dioxide present in the kilned malt (Williams and Gracey, 1982).

Peat-derived phenols are among the compounds that contribute to the peaty flavour of distilled spirits. These compounds are complex, and become increasingly so as kilning temperatures are raised. The spectrum of phenols and the sensory properties of the kilned malt can be controlled by careful management of the peat-burning regime. Detailed reviews are available in the literature (Bathgate, 1973; Walker, 1990). The compounds are introduced during kilning by exposing the malt to the smoke (reek) from burning peat. Phenols from peat reek are absorbed efficiently when the malt moisture content exceeds 25 per cent. A minor proportion of the phenol absorbed from peat reek is retained in the peated malt, the balance being neutralized or destroyed by changes (possibly enzymic) that take place in the malt during kilning. Quantitatively, the most important phenols are phenol, isomeric cresols and the xylenols. Other phenols such as guaiacol, although present in smaller amounts, can make a very significant sensory contribution. The combustion temperature of the peat affects both the yield and the relative amounts of phenols produced; raising this temperature from 400°C to 750°C increases phenol and cresol concentrations several-fold but, diminishes the yield of guaiacol. Methods for the determi-

nation of phenols in malt have been reviewed in the brewing literature (Thomson, 1983).

The effect of kilning on malt bacteria

Many of the bacteria that infect spirit fermentations originate from malt. Levels of bacteria are too low in kilned malt to have a deleterious effect, but, given the correct conditions, these bacteria can become established in the distillery plant and ultimately enter the worts. Bacterial infection of malt is greatly reduced by the presence of sulphur dioxide during kilning. In malt distilling, the absence of wort boiling makes it imperative to observe plant hygiene and sanitation. Heavy bacterial infection early in the fermentation can reduce spirit yield, and introduce unwanted flavour components or their precursors.

Yeast nutrients

Malt supplies soluble amino nitrogen, which is essential for yeast growth and rapid fermentation. It is also capable of at least partially satisfying other requirements for materials such as unsaturated fatty acids, sterols and vitamins, which enable the yeast to function under anaerobic conditions. Nitrogen is a major requirement, but in an all-malt wort there is no shortage, even when the TN content of the malt is as low as 1 per cent. A concentration of 100–150 mg α-amino nitrogen per litre of wort at a specific gravity of 1.040 is generally considered sufficient. The spectrum of malt amino acids that is available to the yeast and is modified during kilning influences the content of volatile components present in fermented wash.

Malt rootlets (culms)

Malt rootlets produced during the germination process constitute about 3–5 per cent of the original barley dry matter. They are removed after kilning, contributing considerably to overall malting loss. They have a spirit-producing potential of over 150 litres of alcohol per tonne, but are normally sold for cattle feed because of their high protein content (*c*. 25–30 per cent). They are rich in vitamins and have relatively high levels of basic materials including hordenine (Liu and Pomeranz, 1976). Rootlets are capable of acquiring relatively high levels of nitrosamines, phenols and sulphur dioxide during kilning if conditions are conducive to the formation of these compounds. The sulphur dioxide content of rootlets, for example, can be 100–200 times that of the parent malt, thus emphasizing the importance of their efficient removal from it.

The technology of kilning

In traditional malting, distilling malts were (and still are) kilned in natural-draught kilns with distinctive pagoda-shaped roofs. The malt beds are shallow at around 30 cm, airflows are low, and temperature control is erratic. The original fuel was peat, and later a mixture of peat and anthracite was burned in the hand-stoked furnaces. The adsorption of peat smoke on to the malt is

the most distinctive feature of most distilling malts made for malt distilling, and its use as a raw material is almost unique to Scotch malt whisky. The levels of peat reek vary from area to area. A few natural-draught kilns are still in operation on Orkney, Islay and the mainland, but, with the centralization of malt production and the introduction of modern malting plant, alternative means of applying peat smoke have been developed.

Malt destined for malt distilling is distinctive in that it is kilned using low air-on temperatures to preserve the diastatic enzymes essential for highly fermentable extract, and high airflows (40–70 m^3/min per tonne) for quick drying to about 5–6 per cent moisture content.

The equipment used for kilning is determined by the type of malting plant – separate dedicated kiln, GKV or SGKV, each of which is discussed below.

Separate kilns

Most separate kilns are of concrete construction following the 'Winkler' design. In these vessels, the green barley is loaded by mechanical spreader on to a wedge wire floor to a depth of approximately 1 m. The square floor is either self-tipping or is stripped by means of a traversing auger. The kiln air is heated directly by firing into the airstream or, more commonly, indirectly by passing the air through a bank of heat exchangers. Directly fired kilns were the norm, and peat, anthracite or coke was used as the primary fuel. When oil became a cheaper option, the kilns built in the late 1950s and early 1960s were fired with light gas oil or heavy fuel oil. By the late 1970s, many maltsters had converted their kilns and were using natural gas. The discovery that direct firing, particularly with gas, led to high levels of nitrosamines in malt (Spiegelhalder *et al.*, 1979) led to a rapid change to indirect firing, where steam or thermal fluids were used as the heat-exchange medium. Light gas oil, heavy fuel oil, natural gas and coal are currently all used in indirect kilning, and many maltings now have the ability to change fuels should this be economically expedient.

With the rapid rise in fuel costs, energy management became as important as control of quality on the kiln. The kilning cycles employed were a compromise between those thermally most efficient and those producing the optimum malt quality. In the 1990s, the need for optimum malt quality took precedence and energy management became less dominant. To optimize malt quality, it should be dried at as low a temperature and as high airflow as possible. Because the electrical power consumed by the kiln fans varies as the cube of the volume delivered, kilning power consumption can be very high for high-capacity fans. Similarly, the greatest thermal efficiency is achieved at as high an air-on temperature as possible – completely opposite to the quality requirement. The compromise position is to kiln to the break point (i.e. during the free drying period when the air leaving the kiln is saturated) at a temperature of 60–65°C and then to increase the temperature to 70°C to the finish with a reduced fan speed. In this way the maximum drying is achieved at as low a temperature and as high an airflow as possible, and

only when the moisture level is below about 10–20 per cent is the volume dropped and the temperature increased.

GKV and SKGV kilns

The general principles described above also apply to GKVs and SKGVs. The major differences lie in the nature of the malt bed. In loading a separate kiln with green malt, great care is taken to have the malt bed evenly and loosely packed so that the passage of drying air is equally distributed over the whole piece.

The problems usually associated with combined germination and kilning vessels are unevenness in bed depth occurring as the barley germinates and expands, areas of compaction caused by mechanical turners, and problems of uneven drying round the turner spirals.

If the bed is uneven and has compacted areas, the air will take the line of least resistance, leaving wet patches of malt on the surface of the bed. When this occurs little can be done to remedy the situation, apart from turning the malt when it is partially dry (e.g. at the break point). Turning at this late stage has a minimal effect on malt quality, but the high costs incurred in drying an unevenly loaded kiln are the main concern. Thus, great care has to be exercised in loading and turning right through the process, to achieve efficient kilning.

Nitrosamine control

Nitrosamine contamination is particularly associated with the use of natural gas as a kiln fuel in directly fired kilns. The way in which the gas is combusted can be altered by use of low NO_x burners. In these burners, the ratio of gas to air is carefully controlled to give a very lean mix. This procedure causes reduction of the flame temperature and in the amount of atmospheric nitrogen oxidized to nitrogen oxide (NO) or nitrogen dioxide (NO_2), which are necessary precursors for the formation of nitrosamines.

Reactions between NO_x gases and hordenine can be blocked by the application of sulphur dioxide (SO_2) to the malt. Raw sulphur is burned and the SO_2 produced is fed into the hot airstream during the early stages of kilning.

By the early 1990s most kilns were indirect, and this has continued to the present time, with a handful of direct ones equipped with low NO_x burners. There remain a handful of traditional directly fired pagoda kilns, where skilful management can produce unique malts with no nitrosamines. In some locations, where there are high amounts of ambient NO_x gases, sulphur has to be burned and the SO_2 fed into the indirect airstream to avoid nitrosamine formation.

Peating

The traditional fuel when each malt distillery had an integral maltings was local peat, together with faggots of heather and other available combustibles, but Moray (1678) reported that faggots of broom should be avoided as they

impart an unpleasant aroma. The procurement of fuel was a central part of the malting scene, and was as important as assuring the supply of barley. In the non-malting season of late spring and summer, when the weather was too warm for floor malting and distilling, the distillery workforce was employed in digging and raising peats for autumn and winter malting. This practice remains part of the working schedule in Orkney and Islay. Peating is now an ancillary process, and peat is no longer used as a primary fuel. All kilns were originally hand-stoked, so that as each addition of peat was made to the fire there was a short period of intense smoking of the fuel before high temperature combustion. Peat, even in a dry state, may contain between 25 per cent and 60 per cent moisture and is a 'smoky' fuel. When used in this way as the direct fuel for heating kiln air, the peat not only dried the malt but also imparted a strong characteristic aroma as some of the smoke was adsorbed by the malt. Later, and especially when oil and gas burners were introduced, separate peat furnaces were installed as a secondary operation to produce smoke rather than primary heat. As long as the primary fuel contained elemental sulphur (e.g. anthracite, light gas oil or heavy fuel oil) the NO_x gases produced in the secondary burning of peat could not react with the malt to produce nitrosamines.

It was only when gas was used as a primary fuel that the dual effect of nitrogen oxides from both the gas burners and the peat furnaces was recognized (Ladish, 1981). The peating of malt is a ready source of nitrosamines, arising from residual nitrogenous material in the peat. Great care must be exercised in stoking the fire to prevent flaming and excessive heat development, and in applying the appropriate amount of oxides of sulphur to prevent nitrosamine formation. When direct gas burners or indirect firing is employed, then rock sulphur must be burned or gaseous sulphur dioxide applied so that about 10–30 ppm of sulphur dioxide is detectable on the peated malt to prevent the formation of nitrosamines. If an indirect heat recovery system is employed partially to heat the incoming air through a heat exchanger, then less primary fuel and so less sulphur is applied to the malt. Given these safeguards on burning and sulphuring, peated malt can be made with almost undetectable levels of nitrosodimethylamine (NDMA) and can be marketed with the same guarantees as other forms of malt.

The adsorption of peat smoke on to the malt is not a simple operation, and is governed by the temperature of combustion of the peat, the intensity of the smoke and the rate of malt drying. Peat smoke will not readily adsorb on to malt when there is a surface water film, i.e. during 'free drying', and adsorbs only slowly when the surface is very dry; greatest adsorption takes place when the malt is hand dry (15–30 per cent moisture). Therefore, peat smoke must be applied intensely during the early period of kilning right up to and beyond the break. The level of peating and the chemical spectrum of phenolic compounds is measured using HPLC. In industry parlance, the relative intensity of peating has three categories: lightly peated (about 1–5 ppm total phenols); medium peated (5–15 ppm); and heavily peated (15–50+ ppm). The last category varies considerably according to individual requirements, the heaviest being the Islay malt.

Malt

Malt specification for Scotch malt whisky

The major parameters of a modern malt specification are set out in Table 2.7, and approved malting barley varieties for 2001 are listed in Table 2.8. The reasons for these parameters are explained below.

Each distiller has a slightly different specification, and may have additional clauses. All analyses are carried out using the recommended methods of the Institute and Guild of Brewing (IOB, 1997), unless otherwise agreed.

Table 2.7
Typical malt distilling malt specification (2001)

Barley varieties	Chariot, Decanter, Optic
Moisture (%)	4.5–5.0
Soluble extract (0.2 mm, dwb)(SE2)(%)	> 76
Soluble extract (0.7 mm, dwb)(SE7)(%)	> 75
Fine/coarse SE difference (%)	< 1.0
Fermentability (%)	> 88
Friability (%)	> 96
Homogeneity (%)	> 98
Phenols content (ppm)	0–50
SO_2 content (ppm)	< 15
Nitrosamines content (ppm)	< 1.0

Moisture

In the last twenty years the moisture content specified has risen from approximately 4 per cent to approximately 5 per cent, for two main reasons. First, it was realized that gentler kilning would help preserve the activities of the more thermolabile enzymes, which could continue to work during the mashing and fermentation, releasing more fermentable carbohydrate. Secondly, there was a saving in energy. Malt is hygroscopic, and there is normally a moisture pickup of around 0.5 per cent between maltings and distillery. Therefore, if the malt is required to be 5 per cent, the off-kiln moisture should be 4.5 per cent. Where there are adjacent maltings and distillery, it can be dried to 5 per cent. Malt with moisture content of 6 per cent or more is difficult to mill, and there is the

Table 2.8
Institute and Guild of Brewing list of
approved malting barley varieties (2001)

Season	Scotland and the northern regions	Southern regions
Winter		Fanfare
		Halcyon
	Regina	Regina
		Pearl
Spring	Chalice	
	Chariot	Chariot
	Decanter	
	Delibes	
	Derkado	
	Optic	Optic
	Prisma	

possibility that 'green-malt' sensory notes may enter the spirit from lightly kilned malt.

Soluble extract

The units used to describe soluble extract (SE) are litres per kilogram (l/kg) or per cent extract (%) (IOB, 1997) The two units are related by a factor. Both units are in use, depending on distiller's preference. The second unit has been criticized (Bathgate 1989) as not being a 'pure' unit, but it has gained wide acceptance and usage.

The SE2 figure is the maximum extract that is obtainable from the malt, as milling at this 0.2-mm setting (fine grind) mechanically breaks up the cell wall material, releasing the starch granules. This figure varies little with the degree of modification of the malt. Milling at the 0.7-mm setting (coarse grind) releases only the starch granules that have been made available because of matrix breakdown during malting, so it is a measure of the degree of modification of the malt. A fine–coarse difference of about 1 per cent is normally specified. Some distillers prefer to use a coarse SE figure obtained by milling the malt sample with a mill gap setting of 1.0 mm, but the vast majority use 0.2 and 0.7 mm SEs.

The SE7 figure was chosen in 1977 (Hudson, 1976) when the Miag laboratory mill became standard, because its values were closest to the distillery's mash tun extract (mte) when the traditional mash tun was the norm. In the last 33 years there has been a gradual change in Scottish malt distilleries to lauter-type mash tuns, which have higher extraction efficiency. These tuns can achieve SE2 levels of extract, but it is the SE7 figure that has been shown to be the most reliable figure for predicting spirit yield (Dolan, 2000). Distillers currently specify SE7 figures in the 85 per cent area – very different from the around 75 per cent that is normal for Golden Promise.

Friability

This analysis is commonly used in malting as a check on the endosperm's modification of the malt, to determine the homogeneity of the modification and to measure the percentage of corns that have not grown. It is widely used in the plant as well as in the laboratory. The information obtained from the friabilimeter, while a useful early indicator of the quality of malting, must be taken as being complementary to laboratory results. In recent years there have been severe problems with calibrating these instruments, and results should be treated with caution, as they are also variety- and operator-dependent. Typical specification (F) figures are friability 96 per cent, homogeneity 98 per cent.

Fermentability

This laboratory test is used to determine the percentage of the SE7 that is fermentable, and allows calculation of the fermentable extract (FE7).

The true method of determining the wort's fermentability (real fermentability) is by conducting a laboratory fermentation and distillation, and calculating it using the original (OG) and residual (RG) gravities obtained, but it was shown (Dolan, 1983) that the figure arrived at by using the laboratory final gravity (FG) (apparent fermentability) and an empirically derived factor gave acceptably close results. At present figures of up to 88 per cent are specified, this high figure being obtainable due to advances in kilning technology and practice. These improvements in malt fermentability appear to carry through to concomitant increases in distillery spirit yield.

A factor can be applied to a malt sample's FE7 to predict the spirit yield with a reasonable degree of accuracy (Dolan, 2000).

The predicted spirit yield (PSY) equation is:

$$\text{PSY (l/t, dwb)} = \text{FE7 } 6.06$$

The empirical equation (Dolan, 1976) is reported to be reasonably accurate over the normal range of malt distillery yields – say 390–450 l/t (dwb). Dolan reported the back-calculated factors (bacfac) for a number of distilleries,

stating that in consistently run distilleries the PSY factor was very stable (Dolan, 2000). In the most efficient plants, with lauter mash tuns, the bacfacs are close to 6.06, while in plants with traditional mash tuns the bacfacs are slightly lower. It is important to note that the PSY equation is not designed to be a measure of distillery efficiency, but a yield predictor.

Phenols

Malt bought for making Scotch malt whisky can have a specification for no phenols when the malt is kilned using no peat, and in a kiln not normally used for making peated malt. Other malts may have a specified content of 1–50+ ppm phenols. For malts in the low range (1–10 ppm), unpeated and peated malt are blended to achieve the required level. For higher levels, such as for malts used in some Islay and other distilleries, the malt is made in a dedicated kiln where there is intense application of peat reek (see above). The level and spectrum of phenols is generally measured using HPLC, which in the last ten years has replaced unreliable colorimetric methods.

Sulphur dioxide and nitrosamines

The SO_2 clause was inserted to check that the malt has been made on an indirect kiln, although the fermentability is a good indicator. In some areas where indirect kilning is used, if there are excessive ambient levels of NO_x gases sulphur must be burned to prevent the formation of nitrosamines. While unpeated malt is generally indirectly kilned, peated malt obviously must have the direct application of peat reek. It is essential to ensure an absence of nitrosamines, and this is achieved by careful control of peat burning and use of sulphur.

Other parameters

Other parameters might include:

1. Diastatic power (DP). This is a measure of the main carbohydrases in malt. With the trend to gentler kilning, lack of these enzymes is unlikely, and in any case the fermentability figure is a more meaningful parameter.
2. Total soluble nitrogen (TSN). Sometimes a minimum TSN is specified when a distiller wishes to make certain that there is enough free α-amino nitrogen in the wort to ensure yeast nutrition. It is unlikely in the modern malting age that there will be a lack. A free α-amino acid level of greater than 90 ppm at a specific gravity of $1.040°$ is sufficient.

McGregor (1999) has set out pointers for future understanding of the science and technology in malt distilling, and the prospect of improved yields and the inclusion of new parameters.

Milling, mashing and wort recovery

The science of milling, mashing and wort recovery

The purpose of milling and mashing is to create malt grist that will readily mix intimately with water during the mashing process, allowing the desired gelatinization, enzymolysis and dissolution to take place as efficiently as possible. The purpose of wort recovery is to produce and separate from the spent grains wort of maximum obtainable fermentable extract for fermentation. Scotch malt whisky distillers use batch infusion mashing, and carefully control the amount of water used and its temperature so that the mash produced has a final temperature of 64.5°C and has a thick porridge-like consistency. The temperature is held constant for 30–60 minutes. During this stand time, the grist particles begin to hydrate and swell, starch granules gelatinize, simple low molecular weight compounds dissolve, and enzymes attack mainly starch and its degradation products (amylolysis) and nitrogenous materials (proteolysis). The carbohydrases break the starch and large degradation products into fermentable sugars, such as glucose, maltose and maltotriose, and unfermentable sugars such as maltotetraose and other linear or branched dextrins. Careful malting, especially kilning, with the aim of maximizing the amount and range of enzymes that survive into mashing and beyond, has a major effect on the efficiency of this conversion process.

It has been shown that minimizing stand time has a beneficial effect on the amount of fermentable sugars obtained at the mashing stage and during fermentation, when surviving enzymes continue to degrade dextrins, releasing fermentable sugars that the yeast can metabolize. This effect has been noticed and exploited as the types of mash tuns use have gradually changed from traditional to lauter-type in the last three decades.

The technology of milling, mashing and wort recovery

After malt has been rested for several weeks at the maltings, it is prepared for despatch and checks are made to ensure that it meets the required specification (Table 2.7). It is then delivered, and is stored in airtight bins. Before use in the distillery, a sample for the first operation – milling – is tested (using a standard sieve or other test) and the mill roller settings are adjusted accordingly. Most malt distilleries are equipped with standard four-roller mills, while a few have six-roller mills, although these have been shown to be unnecessary with well-modified modern malts. The milling requirement varies from distillery to distillery, depending on the efficiency of the mash tun, but a target of about 20 per cent husk, 70 per cent grits and 10 per cent flour is normal practice for traditional mash tuns, while the grind is finer for lauter mash tuns.

After the malt has been milled into grist, it is fed into a Steel's masher and mixed with water that is controlled to ensure is at a final temperature of 64.5°C and at a thickness of about four parts water to one part malt. Usually between five and ten tonnes of malt are mashed per batch.

Over the last three decades there has been a gradual change in mashing technology, with traditional mash tuns being progressively replaced by lauter or semi-lauter mash tuns (Barnes and Andrews, 1998). Both types of mash tuns are used as reaction and separating vessels.

Traditional distillery mash tuns contain large rotating mixing paddles and no sparge systems. In this traditional method the mash is thoroughly mixed for about 20 minutes during mashing-in, and after a total stand-on time of about one hour the first worts are run off until the mash bed is almost completely dry. A second water is then mashed in and mixed at a higher temperature, typically about 70°C, before again running off wort. This procedure is repeated for a third and sometimes a fourth time, with temperature increments to around 80°C and 90°C respectively. The volumes of water used at each mashing operation vary considerably, but approximate ratios of first, second, third and fourth waters are $4:2:4:2 \, m^3$ per tonne of malt mashed. The first and second worts are pumped in turn through a heat exchanger, where they are cooled to around 19°C, to a fermenting vessel or washback. The third and fourth runnings are combined to provide a portion of the mashing liquor for the subsequent mash. Distillers use variations of this format, with usually three mashes and slightly different volume ratios, aiming to produce worts of consistent OGs in the range 1.050–1.065. Consistent OGs lead to more consistent flavour production during fermentation, and aiming for a consistent desired-flavour profile is more important than energy saving.

The mashing technology that has gained ground over the last 33 years is to use a Steel's masher directly with a shallow-bed lauter tun, fitted with above-bed knives and a sparging arm. While a traditional mash bed may be 1.0–1.5 metres in depth, a lauter tun may only be 0.5–1.0 metres deep, providing a shorter drainage path for sparge water. Consequently a finer grind can be employed, and instead of separate mashes a continuous sparge, with an upward temperature profile, can be applied. A 'semi-lauter' system (which means operating a lauter tun using a traditional infusion mash protocol) can yield up to 1 per cent more fermentable extract. The most traditional of distilleries will achieve 100 per cent of a standard Institute of Brewing (IOB) laboratory extract, but the target for most mashing operations is to achieve the equivalent of the IOB fine-grind extract. In both mashing systems the wort run-off is controlled by means of an underback, which is a balancing tank that is used to ensure that the grist is not drawn down on to the slotted floor. Worts run-off times vary from 1 hour in a modern lauter plant to up to 6 hours in a traditional plant. The wort coming from a lauter plant is generally more fermentable, as the carbohydrases survive better owing to the shorter time at temperatures unsuitable to them during the stand and run-off, but the extraction may be less efficient because of the faster turnaround time. The efficiency of both extraction and sparging is checked using the standard laboratory methods. The total available extract (% dwb) should be greater than 1.0, and the sparge loss 0.5–1.0 per cent.

It is difficult at wort running to check the quality of the wort, as its chemistry is dynamic, because the wort is not boiled as in brewing. HPLC analysis

of the fermented wash gives an excellent indication of the combined efficiency of mashing and fermentation, and can be used as a tool to indicate what adjustments to make to improve subsequent mashes

It is widespread practice that, during the pumping of the cooled first worts to the fermenter (wash back), yeast that has been slurried in water is bled into the stream to minimize competitive infection during fermentation. The yeast may be added in other ways (see Chapter 4).

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Chapter *3*

Grain whisky: raw materials and processing

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Introduction

The production of Scotch whisky has been the subject of an impressive volume of references, both as layman's guides for consumers (e.g. Murray, 1977) and as more technical accounts for people directly involved in the distilling industry. However, most of these are focused on the production of malt whisky. These often provide an almost romantic backdrop to the marketing of Scotch, with a strong emphasis on traditional methods of production, but tend to obscure the relative importance of grain whisky. Unfortunately, while a number references give some information regarding grain distilling, this tends to be more as an afterthought to relatively detailed accounts of malt distilling.

Exceptions to this include some informative accounts of grain distilling, such as those of Pyke (1965), Rankin (1977), Bathgate (1989), Wilkin (1989) and Piggot and Conner (1995). However, with recent changes in the industry this is an appropriate time to review the contribution of cereal processing in the production of Scotch grain whisky.

Scotch whisky has two major and distinct forms, malt whisky and grain whisky. Malt whisky is produced wholly from barley malt as the cereal source (see Chapter 2). On the other hand Scotch grain whisky is produced from unmalted cereals. These are processed together with a small amount of barley malt, which supplies the enzymes to convert starch to fermentable sugars. The production of grain whisky can be regarded as a large-scale extraction and

fermentation process, which is adapted to the continuous operation of Coffey or Patent grain stills.

The production of Scotch grain whisky, like that of malt whisky, is constrained by the requirements of the Scotch Whisky Act 1988 and the Scotch Whisky Order 1990 (Statutory Instrument 1990), which were enacted to protect the exclusive generic nature of Scotch whisky from adulteration by other spirits. One of the other main effects of this legislation is to limit the use of some processing options that may be available for other types of grain spirits, such as commercial enzyme preparations, processed cereals and adjuncts, such as flaked maize, and nutrient supplements for yeast.

According to this legal definition of Scotch whisky, only endogenous enzymes from the malt can be used to convert the starch and additional commercial enzyme preparations are not permitted. However, commercially produced exogenous enzymes can be used in the production of neutral spirits that are not destined for the Scotch whisky market.

There is a common misconception that in the production of Scotch whisky there are large differences in the processes used for malt whisky, while all grain distilleries tend to be the same. In fact, it can be argued that the distillery processes used in nearly all malt distilleries are relatively similar, compared with the differences seen between the grain distilleries.

One of the problems with describing grain distillery processing is that although there are just a small number of grain distilleries, these represent a very broad range of processing options. In addition, the flexibility of these distilleries, within the confines of the Scotch whisky definition, means that the processes used in individual grain distilleries are continually developing as demands to improve efficiency and competitiveness are incorporated. Additionally, the fact that the few distilleries are in direct competition with one another means that some aspects of individual processes are regarded as being confidential to the parent company and are not disseminated widely, even within an industry as compact as the Scotch whisky industry.

This makes it very difficult, if not impossible, to provide a definitive account of grain distilling that will not immediately become obsolescent. The aim of this chapter is to describe the major stages of the processing of cereals for the production of Scotch grain whisky, such as milling, cooking, and the conversion of starch into fermentable sugars. These processes will also be placed into context with the fundamental biochemistry governing them, which includes grain structure, starch composition, gelatinization, retrogradation, and the importance and actions of starch-degrading enzymes.

It should be mentioned that although the main focus of this work is on the production of Scotch grain whisky, grain distilleries can also produce neutral spirit for use in the manufacture of vodka, gin and other products, and the processes for these other products have many common features with grain whisky production.

Grain whisky is by far the most important form of Scotch whisky in terms of alcohol production, and comprises about 60 per cent of the total alcohol produced by the Scotch whisky industry. This large proportion of alcohol is produced by a very small number of grain distilleries.

Grain whisky forms the basic matrix for blended whisky, which accounts for more than 90 per cent of the Scotch whisky that is shipped throughout the world. This represents a very high level of annual export earnings, at around £2 billion per year (based on figures for 2000, published by the Scotch Whisky Association), and makes this sector one of the most important earners in the UK economy. This emphasizes the importance of ensuring that production in grain distilleries is carried out as cost-effectively and efficiently as possible, and distillers have a strong interest in ensuring that as much alcohol is produced from the raw materials as is possible.

There are currently seven***12 grain distilleries in Scotland, producing somewhere in the region of 200 million litres of alcohol per year. Unlike malt distilleries these tend to be situated in the lowlands of Scotland, largely in the central belt bounded by Glasgow, Edinburgh and Perth, fairly close to industrial and urban centres. The exceptions are Invergordon, which is situated to the north of Inverness; Girvan, which is on the southwest coast near Ayr; and Loch Lomond, which is situated at Alexandria, near Loch Lomond.

The grain distilleries that are currently in operation are The North British Distillery, Cameron Bridge, Port Dundas, Girvan, Invergordon, Strathclyde and Loch Lomond. Dumbarton distillery was taken out of service during 2002. This means that the remaining distilleries are under increasing pressure to ensure that they are as efficient as possible and able to meet the future needs of the industry. It is thus ever more important that the principles behind the process are thoroughly understood, so that the implications of modifying or replacing important parts of the process can be assessed by those who are implementing these changes.

However, it is important to realize that this chapter is not a manual to be read for the optimum operation of grain distilleries; current processes are too diverse for this. Each distillery has its own set of control parameters, which determine the efficiency of the process. This is more a way of summarizing information from a diverse range of sources in a form that is relevant to the production of Scotch grain whisky.

Raw materials

Maize

Traditionally, until about 1984 the main cereal raw material for the production of Scotch grain whisky was maize *(Zea mays)* (Brown, 1990), which was originally sourced from North America as US grade 3 (Lyons and Rose, 1977). This is of lower quality than US grades 1 and 2 maize, which are controlled for use in the manufacture of US spirits (Lyons, 1999). US maize was originally attractive because it was relatively inexpensive, being the cheapest cereal grain available in the USA (Bronsky and Schumann, 1989), and because of the high levels of starch associated with this cereal, which it was considered could be readily extracted and converted into fermentable sugars (Lyons and Rose,

1977). Since then, as result of economic factors such as increases in import duty and other measures designed to improve the competitiveness of European (EU) produce, the use of maize has declined substantially in favour of wheat. However, some distilleries still use maize, at least periodically, although this is now obtained almost exclusively from the south of France. This is generally of the 'yellow dent' type, although occasionally white corn (which has been considered to give a higher alcohol yield) has also been used (Lyons, 1999).

Maize is well suited for the production of Scotch grain whisky, since it has a higher starch content than wheat (Walker, 1986), gives a higher spirit yield and has been associated with fewer processing problems than wheat. This is because maize contains much lower levels of viscosity-inducing materials such as pentosans (Bathgate, 1998) and glucans (Walker, 1986).

than wheat. However, maize starch has a higher gelatinization temperature than wheat and it is essential that it is cooked at high temperatures in order to gelatinize and release the starch efficiently prior to conversion of the starch to fermentable sugars (Bathgate, 1989).

In the past maize for Scotch whisky distilling has been purchased primarily on the basis of its moisture content and specific weight (bushel weight); however, since the advent of genetically modified (GM) maize in the USA Scotch grain whisky distillers have had to use supplies certified as being non-GM, such as those traceable through an Identity Preservation Scheme (Robson, 2002).

It is believed that maize (or corn) originally came from central Mexico about 5000–6000 years ago, and it is now cultivated throughout the world. There are several types of maize, which include dent and flint types. The most important of these are the American dent types, which dominate the agricultural markets (Watson, 1984). A variant with considerable industrial importance is waxy maize, in which nearly all the starch is in the form of amylopectin. The flint types, which were once important in southern Europe, Turkey, Argentina and South Africa are now disappearing (Pendered, 1983). Historically the major countries exporting maize have been the USA (which dominates the market), Argentina, South Africa and France. Originally the USA was the major supplier to the Scotch whisky industry, but for the reasons outlined above maize for Scotch whisky production is now sourced from accredited sources in the southwest of France (Robson, 2002).

An ear (cob) of maize is made up of a central stem on which up to 1000 seeds (kernels) develop. These mature about 60 days after pollination and, in the Northern hemisphere, are harvested in the late summer or early autumn, when the kernel moisture is below 30 per cent (Watson, 1984). These are further dried to about 14 per cent moisture, and the rapid drying process used in the USA results in the kernels becoming fragile and liable to shatter (Pendered, 1983), resulting in a high proportion of damaged corns.

Overall mature maize kernels contain, on average, about 71–72 per cent starch and 10 per cent protein. The mature maize kernel has four principal parts. The most important part for distillers is the endosperm, which comprises about 82 per cent of the grain. This contains about 98 per cent of the total

starch in the kernel and about 74 per cent of the total protein. The other major parts of the kernel are the germ, which accounts for about 11 per cent of the kernel weight; the pericarp or hull (5 per cent), which is the smooth dense outer covering of the kernel; and the tip cap (0.8 per cent), which is the remnant of the pedicle that attaches the seed to the cob (Watson, 1984).

The mature endosperm of maize consists of a large number of cells packed with starch granules embedded in a continuous surrounding protein matrix. The whole cell contents are in turn surrounded by cellulosic cell walls. Mature dent corns contain a central core of soft floury endosperm surrounded by a harder glassy region known as horny (or hard) endosperm; the ratio of soft to hard endosperm varies but is generally in the region of 1 : 2, and is largely dependent on the protein content.

Different types of corn contain different proportions of floury and hard endosperm, and these are important in determining the processing characteristics of the maize. Floury endosperm is characterized by larger cells containing larger starch granules in a thinner protein matrix, which ruptures easily during drying. Thus floury maize kernels with a higher proportion of floury endosperm process relatively easily. On the other hand, in kernels with a high proportion of hard endosperm the protein matrix is much thicker and does not rupture during drying, and the starch granules are compressed into an angular, close-packed configuration with a hard, glassy structure. Hence maize kernels containing a high proportion of hard endosperm, such as flint maize, are much harder to process and it is more difficult to extract starch efficiently (Watson, 1984). This is one reason why it is essential that grain distilleries must process maize at a high temperature and pressure.

The use of maize for distilling has declined considerably over recent years, and the use of wheat has dominated the industry since the 1980s. However, some individual distilleries still prefer to use maize because their processing systems are more suited to this cereal and because there is a perception that maize contributes to the flavour characteristics of the final spirit (Robson, 2002).

Wheat

Since around 1984, soft winter wheat *(Triticum aestivum)* has been the major starch source for the production of grain whisky in Scotland (Brown, 1990). Of the average UK wheat crop between 1995 and 2001, which accounted for 15.1 million tonnes, around 3 per cent was used by the Scotch whisky industry (Home Grown Cereals Authority, HGCA, 2001). The predominance of wheat in recent years is mainly due to economic factors resulting from higher levels of import duty on maize and substantial restitution payments on whisky exports, which made it more attractive economically to use home-grown wheat (Palmer, 1989).

Although the change from maize to wheat has been generally considered not to have affected the quality of the final spirit, some processing adjustments

have been necessary (Nicol, 1990) and some distillers believe that wheat spirit is lighter-bodied than that distilled from maize.

Wheat and maize have different processing characteristics, and these have important implications for the handling of distillery co-products such as spent grains and spent wash. Most grain distillers dry their spent grains and spent wash, then combine them to produce animal feed. Co-products deriving from maize dry more easily and efficiently than those of wheat, since maize spent wash is less viscous than that of wheat. As a result wheat has been reported to cause more processing problems, since spent grains and spent wash deriving from wheat can cause a residue build-up on heat exchangers and evaporators, which reduces efficiency and may cause processing down-time (Newton *et al.*, 1994). Wheat is also associated with viscosity problems, which are believed to be due to a number of different factors, such as gluten content and pentosan polymers such as arabinoxylans. Wheat endosperm walls contain around 75 per cent arabinoxylans, while maize contains about 25 per cent (Newton *et al.*, 1994).

The wheat grain is the seed or fruit of the wheat plant. Structurally the kernel can be divided into three distinct parts; the endosperm, which comprises around 83 per cent; the bran, which accounts for 14 per cent; and the germ or embryo, which makes up about 3 per cent of the kernel (Bushuk, 1986).

The endosperm is the most important part of the kernel in terms of distilling, and consists of more than 80 per cent carbohydrate (mostly starch), approximately 12 per cent protein and 2 per cent fat with 1 per cent minerals and other constituents (Bushuk, 1986). Starch is the main reserve carbohydrate of the wheat grain. The amount of starch in wheat is generally inversely related to the nitrogen content, and can be a reliable index of the anticipated yield of alcohol. The cultivar, or variety of wheat, is also an important factor in determining the suitability of wheat for distilling.

Wheat specifications

One of the advantages of wheat is that there are a large number of different varieties to choose from, although only a few soft wheats (such as Riband, Consort and Claire) are currently regarded as suitable for distilling (Brosnan, 2001). Some of the main factors used to distinguish wheat varieties are hardness or softness of the grain, winter or spring habit, and (more recently) protein content. Within these classes, wheat can further be described according to specific weight, contamination with foreign materials (including other cereal grains), the level of screenings, the absence of sprouted grains (degree of soundness), the moisture content, and measures of properties of their doughs as well as other measures of processing quality. Some of these properties are influenced primarily by genetic factors and are inherent in the varieties of wheat produced, while others are governed by environmental factors such as soil fertility, rainfall, and temperature, both during the growing season and at harvest (Orth and Schellenberger, 1988).

In the past cereal buyers in distilling companies tended to avoid specifying too many parameters for wheat, since this would incur a price premium to suppliers as more parameters were specified (Brown, 1990). However, nowadays it is normal in purchasing wheat suitable for distilling to specify not only moisture content, specific weight, hardness and nitrogen content, but also, in some cases, wheat variety (Brown, 1990; Nicol, 1990).

The types of wheat best suited for alcohol production are soft white or red winter wheat varieties. Durum and hard red spring wheats are generally not suitable for alcohol production, owing to the lower starch content and the resultant low yield of alcohol (Stark *et al.*, 1943).

Winter wheat is the highest yielding cereal in Britain, and comprises the largest acreage sown. In Scotland, winter wheat is sown in late September to November and is harvested the following September. The optimum seed rate depends on when the crop is sown; a low seed rate (300 seeds per square metre) is suitable for early sowing, whereas a high seed rate (exceeding 450 seeds per square metre) is suitable for November (winter) sowing. Nitrogen applications are carefully controlled during crop growth, as nitrogen applied to crops nearer harvest accumulates in the grain and gives an increased final nitrogen content, which is not a favourable trait in wheat used for distilling although it is desirable in bread wheat (David Cranstoun, Scottish Agricultural College, personal communication).

Distillers prefer to use soft wheat varieties because they tend to give fewer processing problems in distilleries. Hard wheats are associated with higher viscosity worts than soft wheats, irrespective of protein content (Brown 1990), and tend to increase problems in important rate-determining areas of the process, such as the transfer of worts and centrifugation and evaporation of spent wash.

Specific weight is the weight of a known volume of grain (expressed in kilograms per hectolitre, kg/hl). This was originally referred to as the bushel weight and is broadly similar in concept to thousand corn weight, which is used for barley. The higher the specific weight the more starch (and protein) it contains, and grain distilleries usually set specifications at no lower than 72 kg/hl (Brown, 1990). The size of kernels or screenings may also be specified as this may be important in some distillery processes; if the grains are too small they can pass through mills and enter the process whole, which might mean that not all the available starch is extracted, leading to decreased alcohol yields.

The differences in hardness of wheat appear to relate to adhesion between starch granules and storage proteins (Wrigley *et al.*, 1988). Hardness of grains is often measured by a simple milling test, with samples that take the longest to mill being classified as the hardest and *vice versa*. The hardness or softness of grain texture is linked to the way in which the starch is bound up within the protein matrix, and is thus related to the nitrogen content. The inverse relationship between grain nitrogen level and alcohol yield is well known, and simply relates to the relatively low starch content of higher nitrogen wheats (Brosnan *et al.*, 1998). This has been confirmed by distilling trials, where it has been found that there is a direct link between nitrogen content and spirit yield.

This relationship has been found to be stronger in some harvest years than in others (Scotch Whisky Research Institute, SWRI, unpublished data). Work by Brosnan *et al.* (1998) suggests that other factors that are relatively independent of nitrogen content, such as the proportion of A and B starch granules and the way they can be densely packed together, may also have an important role to play in determining the maximum alcohol yield of wheat.

Distillers require inexpensive soft wheat that is easy to process, with high levels of starch and low protein content. This specification is similar to feed wheat, and therefore distillers are not in competition with bread millers for their wheat (Brown, 1990). In contrast, for bread production millers require hard wheat with high nitrogen and gluten contents and low starch contents, which could cause 'stickiness' problems in distilleries and give a low alcohol yield. This complementary requirement for the 'best of the rest' (Brown, 1990) that the bread manufactures do not need has enabled distillers to buy suitable wheat at more economically favourable 'commodity' prices.

Wheat breeding

In the nineteenth century, Gregor Mendel established the basic principles of plant genetics when he discovered that certain inherited traits were transferred from one generation to the next. The plant breeder's aim is to reassemble these units of inheritance, known as genes, to produce crops with improved characteristics. In practice this is a complex and time-consuming process. Each plant contains many thousands of genes, and the plant breeder seeks to combine a range of desirable traits in one plant to produce a successful new variety. Conventional breeding involves crossing selected parent plants, chosen because they have desirable characteristics such as high yield or disease resistance. The breeder's skill lies in selecting the best plants from the many and varied offspring. These are grown on trial plots and tested over a number of years. Typically this involves examining thousands of individual plants for different characteristics, ranging from agronomic performance to end-use quality. Developing a new variety of wheat can take up to fifteen years.

However, plant breeding is not an exact science and often unexpected side effects can occur, sometimes resulting in undesirable characters – for example, varieties containing the 1B1R rye gene from early wheat/rye crosses tend to have higher *in vitro* viscosity than non 1B1R hybrids. In addition, soft wheat varieties tested containing the 1B1R translocation gave alcohol yield results at the lower end of the expected range for distilling wheat, and are thus not classed as good for distilling.

There is also some evidence that insertion of the 1B1R translocation causes an unexpected reduced nutrient digestibility of feed varieties (Short *et al.*, 2000), which could have a detrimental effect on chickens. This suggests some similarity between distilling wheat and poultry feed wheat.

Although the cultivar Riband was not originally produced specifically for distilling, it has been the market leader for many years. Plant breeders are still

unsure what made Riband so much better than other available varieties, and why it gives the consistency of alcohol yield with which it is associated.

Assessment of new wheat varieties

For many years, discussions between suppliers and end users of wheat regarding the desirable quality characteristics for distilling wheat have been coordinated by the Scottish Agricultural College. This collaboration is an ongoing one, and is useful and successful for both distillers and growers (Brown, 1990).

A recommended list of wheat varieties is now produced on an annual basis by experts from the Scottish Agricultural College (SAC, 2001), the Home Grown Cereals Authority (HGCA) and the National Institute for Agricultural Botany (NIAB, 2002), who have given guidance to the farming community on the selection of suitable wheat varieties for planting in Scotland and the rest of the UK. The harvest weather in Scotland tends to produce wheat that is inherently low in nitrogen content (Brown, 1990). The Scottish Agricultural College has been able to reconcile the complementary requirements of farmers and distillers so that suitable varieties for distilling, such as Riband, Consort and Claire, continue to be available, and new varieties are continually being evaluated.

The Scottish Agricultural College carries out wheat trials at various sites each year, in many different locations. This allows them to assess new wheat varieties that have been put forward for testing, as well as named varieties in the final stages of the recommendation process that may have gained provisional approval. The aim of the trials is to select up and coming wheat varieties that are suitable for growing in Scotland. New varieties are not approved until they have completed at least three years of trials (SAC, 2001).

Decisions regarding whether new wheat varieties progress through the recommendation process are largely made on the basis of the results of agronomic tests, such as agronomic yield, disease resistance and straw strength. However, to maintain a strong link between distillers and the work carried out by the SAC, each year the Scotch Whisky Research Institute obtains samples of soft wheat varieties from these trials and assesses them for their distilling potential by measuring and comparing their alcohol yield. This work has shown that certain wheat varieties have a consistent tendency to give higher potential alcohol yield results than other varieties.

Over the last decade or so the cultivar Riband has been the dominant variety in Scotland, and it was fortuitous for distillers that the changeover from maize to wheat for distilling in the middle 1980s coincided with the SAC listing of this variety (Brosnan, 2001), since Riband has shown itself to be uniquely suited to grain distilling. The strength of this soft wheat variety for distilling has been in its consistency in producing low nitrogen grain that provides high alcohol yields relative to other varieties, coupled with good agronomic performance (Brosnan *et al.*, 1998). However, Riband is now becoming an 'old' variety, is costing more to grow and maintain, and is being outclassed in terms of agronomic performance by other varieties. This is resulting in Riband increasingly losing its market share. In recent years, some distillers have spe-

cified Riband wheat from their suppliers in order to ensure an adequate supply of this variety for distilling use. However, more new varieties will have to be developed to fulfil the distilling market requirement, and research is continually going on to find new varieties suitable for distilling end use. Varieties such as Consort and Claire have been put forward as possible alternatives, but neither of these are considered to be quite as good as Riband (Brosnan, 2001).

Consultations between the distilling industry and plant breeders to find new varieties for distilling are continuing, and research to evaluate the alcohol yield potential of new varieties of wheat as they come along enables grain distillers to inform farmers and plant breeders about which varieties are suitable for distilling, as well as identifying those that are unsuitable.

Barley

Although it can be relatively inexpensive compared to maize and wheat, unmalted barley has been rarely used in grain distilleries because of the processing problems associated with high levels of gums such as β-glucans (Walker, 1986).

In grain distilleries barley is generally used in the form of malt, and its primary function is as a source of enzymes to convert cereal starch from unmalted cereals such as wheat or maize into fermentable sugars. According to the legal definition of Scotch whisky, all of the enzymes in a mash must come from the malt, and no other externally added enzymes are permitted.

It is essential that barley malt for grain distilling, which is often described as high diastase malt, contains high levels of starch-degrading enzymes. Enzyme levels in barley malt are normally defined in terms of dextrinizing units (DU), which is effectively a measure of α-amylase and diastatic power (DP). DP is essentially the total enzyme (α- and β-amylase) activity, as measured using standard methodology (Institute of Brewing, 1997).

Barley for grain distilling must contain high levels of β-amylase and have the potential to produce high levels of α-amylase, limit-dextrinase and α-glucosidase. Generally barley malt with a DP of 180–200 units and a DU above 50 units is considered to be the standard requirement for grain distilling malt (Bathgate, 1989). Since barley malt is a relatively expensive component of the production cost in grain distilleries there is a continuing drive to reduce costs by limiting the amount of malt used, and this has resulted in malt inclusion rates falling to less than 10 per cent in some cases. The main effect of this is that it is now more important than ever for grain distilling malt to meet high enzyme specifications.

The process of preparing malted barley for distilling is covered in detail in Chapter 2. However, there are some differences both in the process used and in the type of barley that is suitable for malting. The main features of the process of malting grain distilling barley are that the barley is allowed to germinate for a longer period, typically five to six days, and that a gentler kilning regime (50–60°C) is used (if at all) in order to develop and preserve

enzyme activity. The use of green malt preserves around 35–50 per cent of the enzyme activity that would otherwise be lost on kilning (Bathgate, 1989).

Barley for grain distilling generally has a higher nitrogen content (1.8–2.0 per cent) than that for pot still (malt distilling) barley (Bathgate and Cook, 1989). This is because grain distillers are less interested in the amount of starch that is present (although this does contribute a small but significant proportion of alcohol yield) than in developing the highest possible enzyme potential.

Originally, unkilned green malt was largely used for grain distilling, and malting was generally carried out at the distillery using local or domestic barley. Green malt was generally cheaper to produce, due to lower energy costs, and gave higher enzyme levels than kilned malt, so that a lower dosage rate was required to achieve efficient of conversion of starch. However, these advantages were offset by higher transport costs and a shorter shelf life (Walker, 1986).

The use of green malt has gradually declined in favour of commercially produced kilned malt, although some grain distilleries still continue to use green malt. However, in mid-2002 the last on-site production facility was replaced by commercially produced green malt (Robson, 2002).

In the past, high diastase grain distilling malt has been imported from places such as North America and Canada (Bathgate, 1989) and latterly from Scandinavia (Sweden and Finland). Barley malt deriving from Scandinavian grown barley has had a reputation for high levels of enzymes, and at one time was in great demand in the Scotch whisky grain distilling sector. However, over the last fifteen years or so the use of these barleys has declined in favour of domestic supplies, amid concerns over unacceptably high levels of the precursor of ethyl carbamate (urethane) in some barley. As part of their overall efforts to minimize ethyl carbamate levels, there have been moves on the part of the distillers to encourage the production of new barley varieties that do not produce this precursor (Cook, 1990).

Recent domestic barley varieties suitable for grain distilling, such as Derkado, Maresi and Decanter, have emerged through the assessment process for malting barley, which has been jointly run by the Institute of Brewing Barley Committee and the Scottish Agricultural College. The development of new barley varieties for Scotch whisky distilling is proceeding, on an ongoing basis, to ensure the continuing availability of suitable barley for the production of grain distilling malt.

Grain distillery processing

General overview of process and principles

The technology of cereals processing for Scotch whisky production has been reviewed in the past by such authorities as Pyke (1965), Rankin (1977), Bathgate (1989), Wilkin (1989) and Piggot and Conner (1995). Developments

in the basic process and technology have occurred over the years in response to requirements for improved efficiency, changes in raw materials, and environmental protection legislation.

In grain distilling, the processing of unmalted cereals has two main objectives: to release the starch from the grain, and to convert this into fermentable sugars. Generally the first of these aims is achieved by cooking the cereals at high temperatures or pressures, which gelatinizes the starch so that it can be released and solubilized. Enzymes from high-enzyme malted barley can then convert the starch to fermentable sugars, which are in turn fermented to alcohol by yeast.

Figure 3.1 illustrates the main features of what might be regarded as a 'typical' grain distillery process.

It represents a very generalized view of cereals processing in grain distilleries as used in the Scotch whisky industry. In practice there are a number of options for each part of the process. Table 3.1 gives an indication of the range of options in use in Scotch whisky grain distilleries.

Each of these has its own advantages as well as disadvantages, and each system is designed to fit in with individual company aims and objectives,
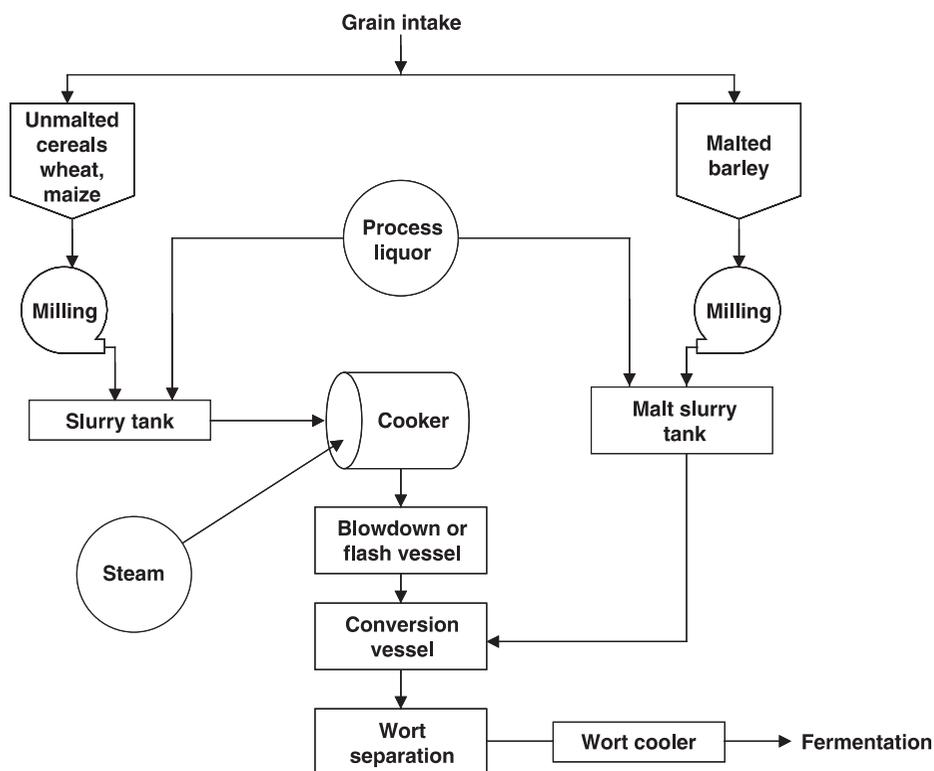
Figure 3.1
Schematic layout of grain processing in a 'typical' grain distillery.

Table 3.1

Processing options used in Scotch whisky grain distilleries

Milling	Process	Process liquor	Pre-malt	Cooking	Conversion	Wort separation
Hammer milling	Batch	Water	No pre-malt	High pressure	Dried malt	No filtration
Coarse 'cracking'	Continuous	Weak worts	Pre-malt	Atmospheric	Green malt	Wort screens
Whole grains		Backset		Continuous		Wort filters

which have mainly evolved as a result of their individual experience with particular plant design and technology. In some cases the systems are also modelled on features from other grain spirit production processes that have been developed in the international market.

Before looking at some of these processes in more detail, it is important to be aware of the basic biochemistry of cereals processing and how this is applied to the processing of raw materials in the distillery. The main areas of interest are the structure and composition of starch, starch gelatinization and retrogradation, and starch-degrading enzymes and their actions. All of these have a direct impact on the way that cereals are treated during cooking, and subsequent conversion of starch into fermentable sugars.

Starch structure

The efficient conversion of starch into ethanol is the major determinant of distillery efficiency, since the primary objective of grain distilling is to produce as much alcohol as possible from the raw materials. The cost of the raw materials is a major component of the overall costs of running a distillery (Hardy and Brown, 1989; Nicol, 1990), and maximizing spirit yield is of fundamental importance to distillers. In order to achieve the maximum potential of the raw materials, distillers must have a good understanding of the structure and properties of cereal starch and the implications these have for processing this material into a fermentable substrate that can be converted into alcohol.

The structure and functions of starch have been well understood for many years, and in the present work there is no intention to provide a comprehensive review of what is a very broad subject area, since this has been well documented and reviewed in many fundamental sources (see for example Whistler *et al.*, 1984; Pomeranz, 1988; MacGregor and Fincher, 1993). The purpose of this chapter is to provide a very brief overview of aspects of starch that are particularly relevant in defining processing characteristics of cereals used in the production of Scotch grain whisky.

The major sources of starch used for making Scotch grain whisky are wheat and maize (corn), although barley, triticale and rye have also been used on

occasion (Lyons and Rose, 1977). The composition and structure of starch can vary for different cereals, and this has important implications for the ways that these cereals are processed in the distillery both in terms of conversion to fermentable sugars and in terms of maximizing the efficiency of cereal throughput.

After cellulose, starch is the most abundant form of carbohydrate produced in plants. Starch is a condensation polymer of glucose, is found in all the major organs of plants, and is the primary form of storage carbohydrate, providing a reserve food supply for periods of dormancy (Swinkels, 1985). In cereals such as wheat, barley and maize, reserve starch is mainly stored in the starchy endosperm, where it is embedded in a protein matrix. Starch is laid down as granules (diameter range 2–200 μm), which accumulate in organelles called amyloplasts in the endosperm cells. Wheat starch mainly comprises two major populations of starch granules: large lenticular 'A' type granules (20–35 μm) and small spherical 'B' type granules (2–10 μm). Normally there are very many more small granules than large granules. However, although the large granules only account for about 12–13 per cent of the total number, they contain more than 90 per cent of the total starch (Bathgate and Palmer, 1973; Shannon and Garwood, 1984).

In contrast to wheat, maize starch granules are irregular and polyhedral in shape and are generally smaller – on average up to 15 μm in diameter (Lynn *et al.*, 1997). Maize starch granule sizes of 3–26 μm have been reported in the literature (Swinkels, 1985). However, maize starch granules have a single size distribution (Cochrane, 2000), rather than the bimodal distribution associated with wheat.

The size and configuration of the starch granules have an important influence on parameters, such as the gelatinization temperature, that determine the temperature required to process the cereal efficiently. The starch in small B granules is more tightly bound, has a higher gelatinization temperature, and thus requires more severe conditions to extract the starch fully than in the larger A granules, where the starch is more accessible. The small granules tend to remain ungelatinized at normal mashing temperatures (Bathgate *et al.*, 1974).

While the small granules contain only a relatively small amount of starch these are still significant in terms of overall yield, and it is essential that they are utilized efficiently to obtain acceptable alcohol yields. The relatively small granule size in maize means that higher temperatures are required to gelatinize and release the starch granules.

Starch granules also contain non-carbohydrate components such as proteins and lipids (Cochrane, 2000), which have potential implications for cereals processing. It has been suggested that the presence of lipids can reduce the susceptibility of the starch to amylolytic breakdown (Palmer, 1989). The presence of lipids is also an important factor influencing the propensity for starch to retrograde after cooking (Swinkells, 1985).

Starch itself is composed of two major fractions. Amylose is a linear molecule comprising long chains of α(1-4)-linked glucose units, and generally accounts for about 15–37 per cent of the total starch. Amylopectin, which

has a highly branched structure, comprises a large number of relatively short α(1-4)-linked chains linked to α(1-6)-linked branches, and makes up the bulk of the starch. Amylose is largely considered to have a regular left-handed helical structure, while amylopectin takes a largely crystalline form (French, 1984; Lineback and Rasper, 1988)). The properties of starch are influenced by the relative amounts of both amylose and amylopectin (Fredriksson *et al.*, 1998).

Amylose in wheat has a molecular weight of 10^5–10^6 Daltons (Da), with a chain length of about 2000 glucose units (Barnes, 1989). Currently it is considered that amylose contains a relatively small number of α(1-6) branches (about 2–4 chains per 1000 glucose units, and 2–8 branch points per molecule; Hoover, 1995) but shows behaviour characteristic of a linear polymer (Lineback and Rasper, 1988). On the other hand, amylopectin has one of the highest molecular weights associated with naturally occurring polymers (up to about 10^8 Da), and this is of the order of 1000 times that of amylose (Barnes, 1989). Amylopectin has a highly branched structure, made up of a large number of relatively short glucose chains (10–60 units, with an average length of about 20–25 units; Cochrane, 2000), but overall the total chain length can be as high as 2×10^7 glucose units. The branch points account for about 5 per cent of the total glucose units that are present (Swinkells, 1985), giving about 1 branch point for every 20–25 residues (Hoover, 1995). Amylopectin is considered to be a major factor in determining the physical and chemical properties of starch (Tester, 1997).

The relative amounts of amylose and amylopectin are fairly constant for starches from a single source. Most starches contain 20–30 per cent amylose and 70–80 per cent amylopectin (Jane *et al.*, 1999). However, different sources of starch have varying amylose to amylopectin ratios, with cereals such as wheat, maize and sorghum having a significantly higher ratio of amylose (about 28 per cent) compared with starches deriving from tubers and roots (potato, tapioca and arrowroot), which contain about 20 per cent amylose. Some starches, such as waxy maize, contain little or no amylose. More specialized cereals, such as amylomaize, can contain as much as 80 per cent amylose (Swinkells, 1985).

The relative amounts of amylose and amylopectin and the distribution of A and B granules have a strong influence on the physical and chemical properties of starch, and are important factors affecting the processing characteristics of cereals, such as gelatinization temperature, viscosity, and the tendency for retrogradation or recrystallization, which are a result of high levels of amylose. These can have a serious effect on processing efficiency, as well as on alcohol yield.

The composition of the starch has an important influence on its breakdown by starch-degrading enzymes such as α- and β-amylase, which by themselves are unable to degrade α(1-6) glycoside links, and the breakdown of amylopectin by these enzymes results in the presence of α and β limit-dextrins as well as fermentable sugars such as maltose and maltotriose. The α(1-6) links in α and β limit-dextrins are degraded by limit-dextrinase.

Gelatinization

After deposition in starch granules, the starch is partly crystalline and is largely insoluble in water. In order to utilize the starch it is necessary to disrupt the granular structure so that it can absorb water (Evers and Stevens, 1985). Gelatinization has been defined by Zobel (1984) as the process of swelling and hydration of starch granules so that the starch can be solubilized. Normally this is achieved by slurrying the starch in water and heating until the starch begins to melt. Ultimately this results in a suspension containing amylose and amylopectin fragments, which are then amenable to the action of amylolytic (starch-degrading) enzymes that can convert the solubilized starch into fermentable sugars (Palmer, 1989).

Gelatinization takes place in several stages (see Figure 3.2). Initially when dry starch granules are exposed to excess water at low temperatures (0–40°C) they undergo limited reversible swelling (the swelling or amorphous phase). As more heat is applied the crystalline starch begins to lose its integrity (the melting phase) and, after combining with the non-crystalline fraction, under-
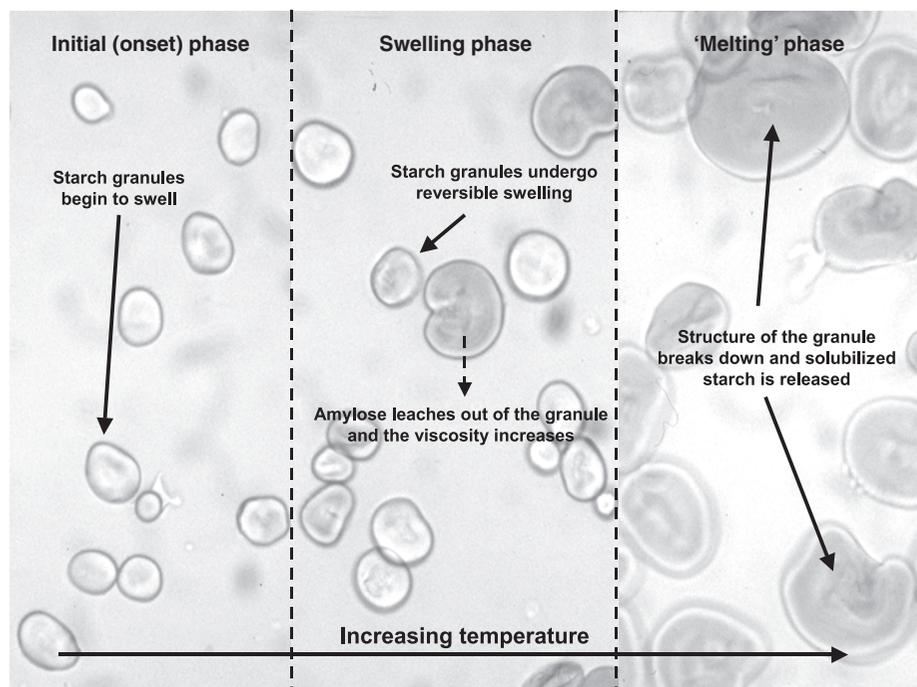
Figure 3.2
Composite photograph showing the phase changes that occur in cereal starch granules as they undergo gelatinization. As the temperature rises the starch granules begin to swell and become less crystalline as they become disrupted (prepared from a set of photographs supplied by M. P. Cochrane, used by permission).

goes irreversible swelling and hydration (French, 1984). This is associated with a substantial increase in viscosity, which has been attributed to the leaching of amylose from the granules. The swelling is accompanied by the disruption of the molecular order inside the starch granule, which is manifested as loss of birefringence (Atwell *et al.*, 1988; MacGregor and Fincher, 1993), which is a measure of the degree of order (or crystallinity) of starch granules when viewed under polarized light (Cochrane, 2000). The degree of swelling has been largely attributed to the effect of amylopectin (Fredricksson *et al.*, 1998).

According to French (1984), gelatinization of starch granules results in the dissociation and uncoiling of the helical regions of amylose and break-up of the crystalline structure of amylopectin, allowing the hydration and swelling of liberated amylopectin side chains. This causes the starch granule to swell, first allowing linear amylose to diffuse out of the granule and then ultimately resulting in the complete disruption of the granule structure. There appears to be a negative correlation between the proportion of amylose and the onset of gelatinization (Fredriksson *et al.*, 1998), thus as the relative amount of amylose increases the temperature of the onset of gelatinization decreases.

In the past, the standard method for measuring gelatinization temperature of cereals has been differential thermal analysis (Zobel, 1984) or differential scanning calorimetry (Atwell *et al.*, 1988). However, the progress of gelatinization has also been studied using various other techniques such as the Brabender Visco Amylograph (Zobel, 1984), which measures the changes in viscosity (pasting) as a cereal slurry is subjected to a programmed temperature cycle. Pasting has been defined as the granular swelling and exudation of molecular components following gelatinization (Atwell *et al.*, 1988). A more modern variant of the Brabender Visco Amylograph is the Rapid Visco Analyser$^{®}$ (Calibre Control Inc., **13). Figure 3.3 shows an idealized RVA amylogram, or pasting curve, which is designed to illustrate the main stages in the gelatinization (pasting) process.

The increase in viscosity observed as the temperature is increased to about 95–100°C shows that starch granules do not gelatinize at the same rate, but do so over a large temperature range. This is dependent on the degree of crystallinity of areas within each granule, which results in considerable variation between different granules. In addition, small granules appear to gelatinize at a higher temperatures and over a wider range (MacGregor and Fincher, 1993). Jane *et al.* (1999) suggest that amylopectin with longer branch chain lengths gives an increase in gelatinization temperature. As the temperature is maintained the viscosity begins to fall as the gelatinized starch is solubilized. When the temperature is reduced the viscosity begins to rise to a much higher level than the previous peak as the gelatinized starch begins to agglomerate and recrystallize to form a resistant gel. This phenomenon is known as setback or retrogradation.

Gelatinization temperatures for cereals are highly dependent on the method used to measure them (MacGregor and Fincher, 1993); however the gelatinization temperatures of maize and wheat are very different, with that for maize
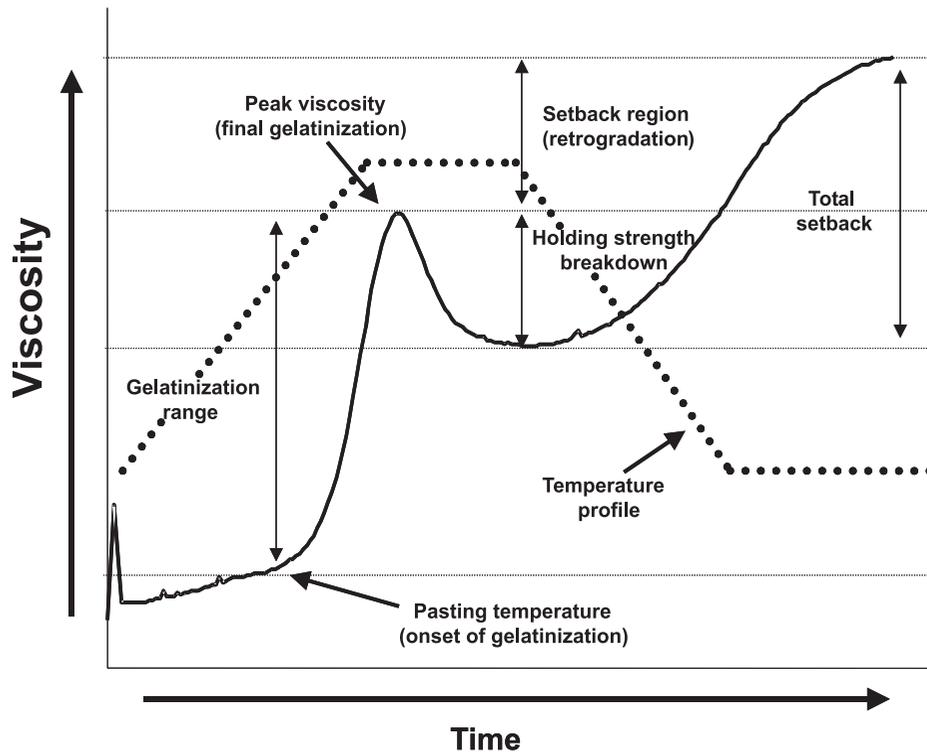
Figure 3.3
A schematic representation showing the main features of a pasting curve produced by a Rapid Visco Analyser (®Calibre Control Inc.)?. This contains some terminology from Dengate (1984).

(70–80°C) being considerably higher than that for wheat (52–54°C; Palmer, 1989). This has important implications for the production of Scotch grain whisky, since this means that high temperature conditions are required to process maize efficiently in the distillery (Bathgate, 1989).

The cooking process

The cooking process has been defined by Kelsall and Lyons (1999) as the process that begins with the mixing of the grain with liquor and ends with the delivery of the mash to the fermenter. In practice this is made up of four different components: milling, cooking, blowdown and conversion. The first three are discussed below, while conversion is discussed in the next section.

Milling

Normally when cereals are processed in the distillery the first stage after intake is to mill the grains, although some distilleries prefer to process whole grains. While the processing of unmilled cereals has declined in recent years, at least one grain distillery still does this. The choice is largely made taking into account the balance between the cost of milling and the energy saved through reduced cooking times (Piggott and Conner, 1995). Nowadays hammer milling of cereals is the norm rather than the exception.

The main purpose of milling is to break up the structure of cereal grains in order to facilitate water penetration of the cereal endosperm during subsequent cooking (Kelsall and Lyons, 1999). Fine milling also has the effect of mechanically damaging starch granules, which promotes the absorption of water (Evers and Stevens, 1985) and facilitates the mechanical release of starch from the protein matrix of the grain and reduces the gelatinization temperature (Lynn *et al.*, 1997). Milling also helps to break down gums (such as arabinoxylans and β-glucans) and other cell wall materials and promotes solubilization of proteins later in the process.

In the Scotch whisky industry two main forms of mills are used; roller mills and hammer mills. Pin mills (disintegrators) have also been used. In some instances wet milling can be used, but this is mainly for processing green malt.

Roller mills are generally used in the production of malt whisky, but can be also used in the context of grain distilling and are particularly suited to the grinding of small-grain cereals such as barley malt and wheat. In a roller mill, cereal grains are compressed as they pass between sets of rollers (normally three sets of two). In some cases the sets of rollers operate at different speeds in order to provide a shearing force to give more efficient grinding of the grain. Roller mills provide a relatively gentle separation of the grist and leave the husk fraction relatively undamaged, which makes them particularly suitable for use in processes requiring the separation of wort in a lauter tun, since the husk is able to act as a filter bed during mashing (Kelsall and Lyons, 1999).

However, hammer mills are normally used in grain distilleries because these can break the grains up into very fine and homogeneous flour that can be handled relatively easily. Hammer milling also enables grain distillers to use short-term cooking and mashing processes, and is particularly suited to continuous processes (Wilkin, 1983).

In a hammer mill, cereal grains (maize, wheat) are fed into a grinding chamber and crushed to a uniform flour by a number of rotating hammers. Control of the grist size is achieved by using a fixed-size retention screen (typically 0.3 cm (1/8") or 0.5 cm (3/16"); Kelsall and Lyons, 1999), which retains larger particles until they are broken down to a uniform size. When using a hammer mill it is important not to grind too finely; this can result in 'balling', which allows small amounts of unprocessed starch to pass through the process. Grinding too finely will also have an adverse effect on the solids content of the post-distillation stillage (spent wash) and put an extra load on evaporators, giving potential downstream processing problems.

Maize is currently not de-germed before milling for Scotch whisky production because this would now be contrary to the legal definition of Scotch whisky, which states that the whole of the grain must be used. Additionally the corn oil is considered to give positive benefits, particularly during fermentation.

The fineness of the grind also has an impact on the yield of alcohol from the process, and an increase in coarseness of about 0.2 cm has been reported to result in a reduction in spirit yield of about 7.5 per cent (Kelsall and Lyons, 1999). If the grind is too coarse, there is a greater potential for the starch to be incompletely gelatinized during processing.

Grain distillers generally prefer to mill cereals prior to processing, although at least one distillery still processes whole cereals. While the use of whole grains can be efficient and cost-effective there can be a cost penalty in extended cooking times (Bathgate, 1989) However, using whole grains has the advantage of potentially reducing the degree of browning reactions, which can result in improved alcohol yield.

Cooking

Principles of cooking

The main function of the cooking process is to break up the hydrogen bonds linking starch molecules and separate the starch from the protein matrix, thus breaking up its granular structure and converting it into a colloidal suspension (Kelsall and Lyons, 1999).

In cereals such as maize, the gelatinization temperature is substantially higher than the temperatures at which the enzymes involved in the conversion of starch to fermentable sugars are able to function (62–67°C; Palmer, 1989). Thus before the starch can be utilized the cereal generally has to be cooked (Wilkin, 1989). The degree of cooking is very much dependent on the cereal used, and this is generally determined by the gelatinization temperature. Maize, which has a substantially higher gelatinization temperature than wheat, requires cooking under more rigorous conditions (Bathgate, 1989). According to Swinkells (1985), true solubilization of starch molecules occurs when a starch paste is cooked at temperatures of 100–160°C.

Although maize is still occasionally used in some distilleries, the major cereal used for the production of Scotch grain whisky is now wheat (Brown, 1990). While in theory wheat would appear to require little or no additional cooking, in practice the experience of distillers has indicated that the cooking of wheat gives improved access to the starch and more complete disintegration of the grain (Bathgate, 1989). In addition, since the economics of the process could conceivably in the future promote a return to the large-scale processing of maize many distillers have maintained the capacity to process maize as well as wheat and have thus retained their traditional cooking processes rather than making substantial changes and adopting cold cooking processes as suggested by Wilkin (1989) and Newton *et al*. (1994). This means that they can respond to changes in the raw materials market. The

modern design of grain distilleries also integrates heat recovery effluent reduction systems that are intrinsically linked to the technology designed for high temperature cooking of cereals.

One of the problems with wheat has been that it contains substantial amounts of cell-wall material such as arabinoxylans, which can cause serious problems with throughput in the distillery both during processing and regarding the recovery of spent grains/wash after distillation. The practical opinion of distillers is that this problem can be alleviated by cooking.

Although unmalted barley has on occasion been used in grain distilleries, since it can be cheaper than other cereals, it gives causes very severe problems with viscosity, owing to the presence of high levels of β-glucans, both during cooking and in the recovery of co-products (Bathgate, 1989) and hence has not been used widely in recent times.

Cooking in the grain distillery

There is a relatively wide spectrum of cooking options used in the production of Scotch whisky, ranging from batch to continuous processes, pressure cooking to atmospheric processing, hammer-milled cereals to unmilled grain, and wheat or maize. Each of these has its own individual features and advantages and disadvantages in relation to the available technology. A number of references describe cooking for grain whisky production: Pyke's (1965) article still provides the definitive account of batch pressure cooking of maize; Rankin (1977) describes the evolution of grain distillery processing from more traditional processes to more modern ones similar to those in use today; and Wilkin (1983) provides an account of both batch and continuous processes, describing the use of both wheat and maize.

Cereal flour (or unmilled grains) is generally mixed with process liquor in a slurry tank Typically the slurry contains about 2.5 litres of liquor per tonne of cereals (Piggott and Conner, 1995). The process liquor is usually water, but can also be recycled stillage (backset) or weak worts or sparge recovered from any mash filtration or separation.

Backset is a recycled portion of the stillage from the distillation from which most of the solid matter has been removed, either by centrifugation or screening (Travis, 1998), and is used in certain cases as a supplement to the process liquor. Although backset can be quite acidic, when it is used properly it is thought to provide important benefits, particularly during fermentation (Travis, 1998). Backset can provide nutrients that are essential for yeast growth, but using too much can result in oversupply of certain minerals and ions (such as sodium and lactate), which can suppress fermentation (Kelsall and Lyons, 1999).

Normally the contents of the slurry tank are mechanically mixed thoroughly to avoid balling of the grist, which can result in lost extract and the presence of unconverted starch – this, as well as resulting in lost alcohol yield, can give problems later in the process. The initial slurry can be carried out at ambient temperature, but is often done at about 40°C (or higher) using waste heat from the process. The higher temperature helps to hydrate and condition the grist as

well as reduce the energy input required for cooking. In some processes, particularly continuous processes, a small amount of barley malt is added as a pre-malt. The purpose of this is for enzymes in the malt (amylases, proteinases and β-glucanases) partially to hydrolyse starch, protein and gums such as β-glucans, in order to reduce viscosity and facilitate pumping of the slurry through the process.

The slurry is then passed forward into the cooker, which is generally a cylindrical pressure vessel fitted with stirring equipment (Pyke, 1965). Thorough mixing during cooking is essential to avoid sticking and subsequent burning (caramelization). Steam is then injected into the cooker to heat the slurry to the required temperature to gelatinize, liquefy and release the starch until the cereal is cooked. Normally the cooker is programmed to operate over a fixed cycle that has been optimized for a particular process and cereal, and cooking temperatures and times can vary for different distilleries. In practice the temperature is programmed to ramp up to the maximum (usually 130–150°C), and is maintained there for only a relatively short time. Some distilleries operate several cookers in parallel in order to maintain sufficient production capacity to support distillation in continuously operating Coffey or Patent stills.

Normal batch cooking is energy intensive and requires relatively long cooking times, which can result in excessive browning reactions and give a reduced yield if the process is not controlled correctly. However, with batch cooking the wort is more likely to be sterile. The batch process is also more adaptable for use with a wide range of cereals.

Table 3.2 gives an indication of the range of cooking temperatures operated at different distilleries.

In the 1980s continuous cooking was seen as having enormous potential in the production of Scotch grain whisky (Wilkin, 1989). However, for various reasons (including process delays, energy efficiency and changes in the market for whisky) continuous processes have not generally found favour in the Scotch whisky industry.

In continuous cooking the finely milled cereal is normally slurried with a small amount of malt (pre-malt) in order to reduce viscosity prior to cooking. The slurry is then heated through a temperature gradient to about 90°C by

Table 3.2
Range of cooking conditions operated at different
Scotch grain whisky distilleries

Cooking process	Maximum temperature (°C)
Atmospheric	95–100
Pressure cooking (batch)	125–150
Pressure cooking (continuous)	120–130

pumping it through a series of precooking tubes (to gelatinize the starch) and it is then passed through cooking tubes at up to 130°C. After about five minutes in the cooker the cooked slurry is discharged into a flash cooler, and is cooled to about 68°C prior to mixing with malt and pumping to a set of conversion tubes. Wilkin (1983) gives an example of the layout of a continuous cooking process.

Continuous cooking has the advantage that cooking time is relatively short, which allows the starch to be gelatinized thoroughly while minimizing the amount of thermal degradation. This reduces the amount of caramelization of the starch through browning reactions. However, because of the reduced processing time the slurry may not be exposed to the high temperature for sufficient time to ensure that the starch is properly cooked. It is also possible that the cooked slurry will not be sterile, which could lead to problems with infection later in the process. The continuous cooking process has the additional disadvantages that it is constrained by the capacity of the fermentation process, which is a batch process, and is particularly vulnerable to interruptions in the process resulting from downstream process problems.

Overall, the cooking process represents a delicate balance between the gelatinization and release of starch and its thermal degradation into undesirable products. If the temperature is too low some starch granules will remain intact and the starch will not be fully gelatinized, resulting in lost alcohol yield. On the other hand if the temperature is too high or the starch is cooked for too long a period, browning (or Maillard) reactions will take place. These reactions remove amino acids, proteins and sugars from the degrading starch, which can result in loss of alcohol yield.

Maillard or browning reactions are a highly complex series of reactions that take place between sugars and amino acids or proteins and result in a range of dark pigmented products (Adrian *et al.*, 1998). These reactions have been extensively reviewed in the literature (Hough *et al.*, 1982; O'Brien, 1998), and there is no attempt to cover this topic in any detail in this work. However, Figure 3.4 gives an idea of the complexity of the processes involved.

Mlotkiewicz (1998) describes the main stages in Maillard reactions. Initially, reducing sugars combine with amino acids to form products (Amadori or Heynes rearrangement products) that are in turn degraded, through a complex series of reactions, into a large number of flavour intermediates and other flavour active compounds. One of the key routes is Strecker degradation, where amino acids also react with dicarbonyl compounds called reductones to form products that are also ultimately converted into brown-pigmented polymeric materials. The final products of Maillard reactions are a mixture of low molecular weight colour compounds containing two to four linked rings, and melanoidins, which have much higher molecular weights. A large number of other flavour and aroma active products, such as furans, pyrroles and cyclic sulphur compounds, are also produced. The development of Maillard products is enhanced by increasing temperature, heating time and pH (particularly above pH 7).

The occurrence of Maillard reactions during cooking is important because they result in the uptake and degradation of both fermentable carbohydrate
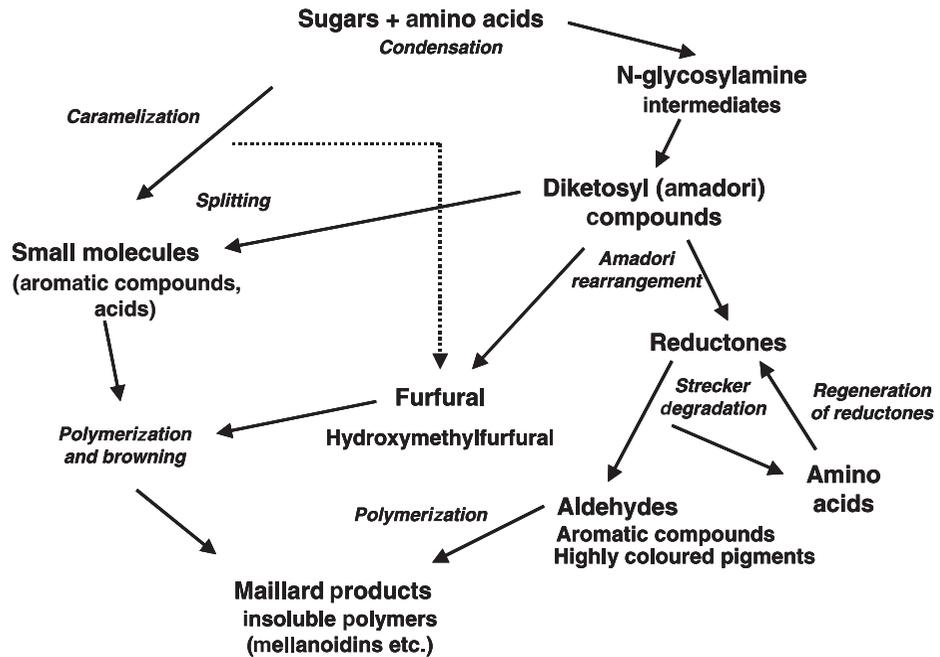
Figure 3.4
Summary of the processes involved in Maillard (browning) reactions.

and amino acids, and could have a significant impact on spirit yield (Figure 3.5). They also have some implications for downstream processing, and the potential to cause problems with co-products.
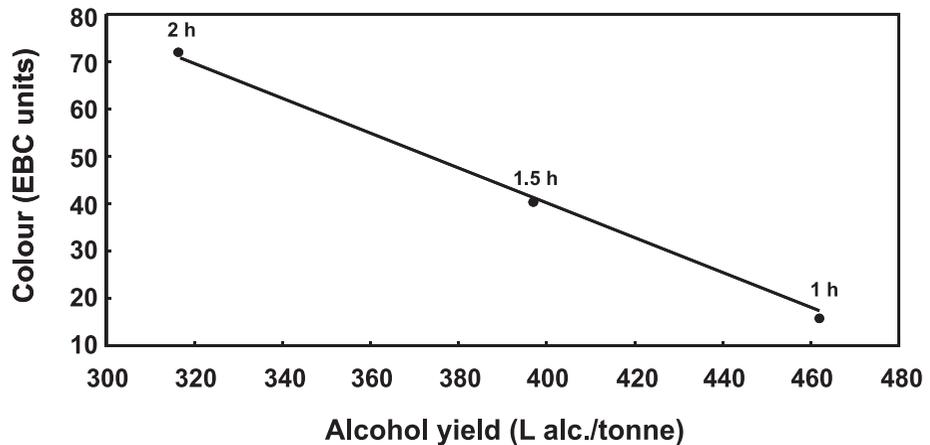

Figure 3.5
Laboratory data showing the inverse relationship between wort colour and alcohol yield for extended cooking times for wheat (1, 1.5, and 2 hours at 145°C) (SWRI data).

The optimum cooking time and temperature depends very much on the cereal used and the process employed. Maize generally requires higher temperatures and/or longer cooking times than wheat. The degree of milling is also important; finely milled cereals require shorter cooking times than unmilled cereals, which can require as long as two hours (Brown, 1990).

According to Bathgate (1989), the pressure cooking of unmilled cereals can be both efficient and cost-effective provided it is economical to use longer cooking times. In addition, when the pressure is released on blow-down the grain endosperm is thoroughly disintegrated as it is passed forwards to the conversion stage. It has been suggested that whole-grain processing is less prone to problems with overcooking or browning. Huskless cereals such as wheat and maize are more suitable for whole-grain cooking, since they disintegrate more thoroughly (Walker, 1986).

Blow-down and retrogradation

When the cooked cereal slurry is discharged from the cooker (generally into a flash cooling vessel or expansion tank) there is a rapid drop in pressure, known as blow-down. This has the effect of mechanically releasing any remaining tightly bound starch from the grain matrix (the popcorn effect). When cooked whole grains are discharged, the pipework associated in the transfer from the cooker is instrumental in ensuring the disintegration of the grains (Walker, 1986).

Blow-down is a critical part of the cooking process, and is normally associated with rapid but carefully controlled cooling. Poor temperature control causes serious problems with retrogradation (setback), resulting in the cooked slurry forming a gel that is resistant to enzymic breakdown (MacGregor and Fincher, 1993). The gel also causes subsequent processing problems owing to high viscosity (Swinkells, 1985) and low filterability, resulting in a poor alcohol yield (Jameson *et al*., 2001).

Retrogradation has been defined as a change from a dispersed amorphous state to an insoluble crystalline condition (Swinkels, 1985) that occurs when heated, gelatinized starch begins to re-associate on cooling (Atwell *et al*., 1988), resulting in gel formation and precipitation (Figure 3.6). The process of retrogradation is very complex (Swinkels, 1985), but is now considered to be to be predominantly influenced by the relative amount of amylose present in the starch (Sasaki *et al*, 2000). Other important factors that are involved in retrogradation are the cooking conditions, starch concentration, cooling procedure and pH. Both wheat and maize (corn) starch, each containing 26–28 per cent amylose, are prone to retrogradation. Waxy maize, which is effectively all amylopectin, is much less likely to retrograde.

The length of the amylose chain is now considered to have a major influence on the processes that take place during retrogradation. This can affect parameters such as the propensity to form precipitates or gels, and the gel strength. Longer chain lengths (>1100 units) show a stronger trend towards gel forma-
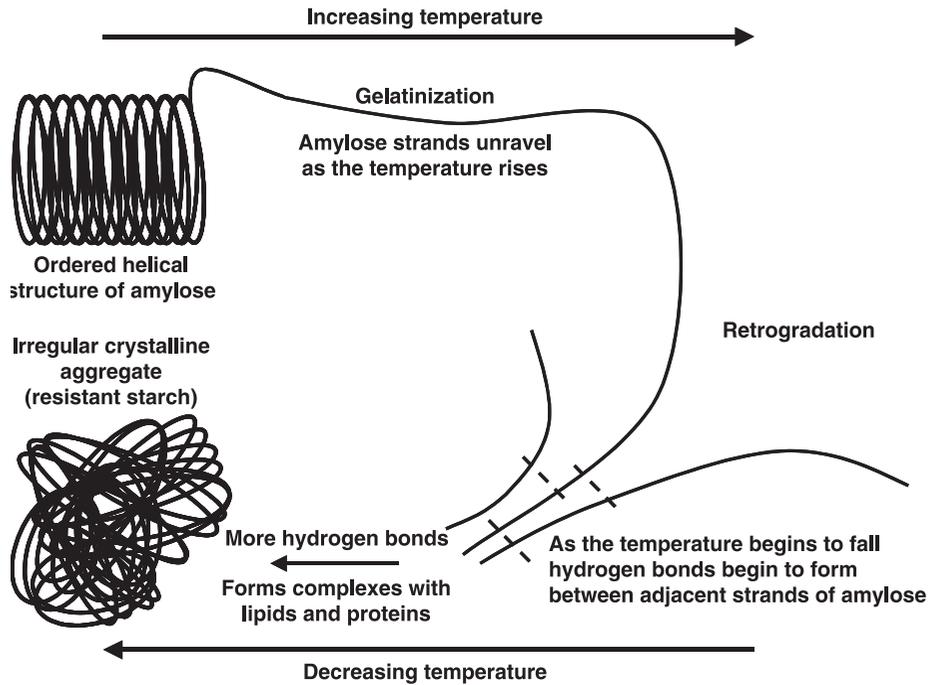
Increasing temperature

Gelatinization

**Amylose strands unravel
as the temperature rises**

**Ordered helical
structure of amylose**

**Irregular crystalline
aggregate
(resistant starch)**

Retrogradation

More hydrogen bonds

**Forms complexes with
lipids and proteins**

**As the temperature begins to fall
hydrogen bonds begin to form
between adjacent strands of amylose**

Decreasing temperature

Figure 3.6
Retrogradation of starch.

tion, due to the alignment and cross-linking of adjacent chains to form ordered structures (Hoover, 1995).

Normally retrogradation occurs as a result of hydrogen bonding between chains of adjacent amylose molecules, which become bound together irreversibly to form aggregates. This cross-linking probably involves a number of regions within a single amylose chain, leading to the formation of a macromolecular network (Hoover, 1995). The aggregated material entraps liquid within a network of partially associated starch molecules, leading to the formation of a gel (Swinkels, 1985). The rate of retrogradation is highest between pH 5 and pH 7. Retrogradation does not occur above pH 10, and only proceeds slowly below pH 2. Retrograded amylose is not readily degraded by α-amlyase (Miles *et al.*, 1985a), and thus once starch has retrograded it is extremely difficult to solubilize again. Retrogradation of amylose is considered to be irreversible, even at high temperatures (greater than 100°C; Miles *et al.*, 1985b).

Amylopectin is much less susceptible to retrogradation than amylose, and the presence of this polymer has been regarded as a moderating influence on this process (Swinkells, 1985). However, under extreme conditions such as high starch concentration and very low temperatures amylopectin can also undergo retrogradation, but this can be to some extent reversible (Miles *et al.*, 1985a, 1985b). Amylopectin from maize has a higher propensity for retro-

gradation than either barley or wheat, and also retrogrades more quickly. This has been attributed to the higher degree of crystallinity of maize amylopectin and a greater proportion of chains of DP 15–20. Wheat amylopectin is less susceptible to retrogradation than that from barley (Hoover, 1995).

While normal retrogadation occurs when starch is cooled, it can also take place at high temperatures (75–95°C) when starch solutions are stored. This takes the form of a precipitate of regularly sized particles. The phenomenon appears to be related to the presence of lipid or fatty acid material forming complexes with amylose. These complexes are not formed above 95°C (Swinkells, 1985).

Jameson *et al.* (2001) observed that extended holding of cooked maize or wheat in a grain distillery at temperatures up to 100°C prior to mashing can result in the formation of resistant starch, which can lead to subsequent problems in the distillery process.

Usually during blow-down the temperature is dropped very quickly to about 70°C, which is very close to the normal striking temperature of malt. This allows the malt to be added in such a way that the malt enzymes begin to hydrolyse the cooked starch before retrogradation takes place. This represents a very delicate balance, and the process must be carefully controlled. If the malt is added too late, or the striking temperature is too low, the cooked starch will begin to retrograde and become inaccessible. If the malt is added too early the striking temperature will be too high and the malt enzymes will be damaged, also resulting in lost yield.

Problems with cooking

There are several main sources of problems during the cooking process. Undercooking occurs when the cooking time is too short or there is insufficient heat to gelatinize the starch properly. This causes significant losses in alcohol yield, since the ungelatinized starch is not readily accessible to enzyme hydrolysis. This can be a particular problem with continuous processing but can also be related to variations in cereal quality, such as wheat nitrogen content and milling performance. When the slurry is overcooked excessive browning (Maillard) reactions occur, which remove both fermentable substrate and proteinaceous material from the wort and result in reduced alcohol yield. Overcooking can also cause physical problems with sticking and burning as the cooked slurry caramelizes. Higher levels of cell wall material and gums such as β-glucans and arabinoxylans, associated with wheat, can also cause problems with viscosity during cooking, but these tend to have more serious downstream effects, particularly on the recovery of co-products after distillation. Retrogradation can be a problem if the cooking and blow-down process is not controlled carefully. Once the starch retrogrades it becomes inaccessible to enzyme hydrolysis and will not be available to produce fermentable sugars, resulting in significant losses in alcohol yield.

Conversion

Principles of conversion

Once the cooker is discharged, the cooked slurry is added to a malt slurry that has been held at a particular temperature (often about 40°C but sometimes closer to mashing temperatures). In some cases the cooked cereal is discharged directly into a vessel containing the malt (drop tank) after cooling to a suitable striking temperature, and in other cases the malt slurry is either added to the cooked cereal directly into the process stream (continuous process), or added to the vessel. The actual procedure used depends on the individual process, and each has advantages and disadvantages. The choice is largely dictated by the technology in place and the economics of the individual process.

The main function of conversion is the breakdown of gelatinized starch into fermentable sugars and small dextrins (starch hydrolysis). It is also essential to degrade proteins into amino acids and low molecular weight nitrogen-rich fragments, which are necessary to provide nutrients for the yeast during fermentation. Since no additives are permitted in the production of Scotch grain whisky, both of these processes must be accomplished wholly by the endogenous enzymes from the malt. In the production of neutral spirit, the use of added commercially produced food-grade enzymes is permitted.

The malt used for conversion is normally commercially produced high-enzyme malt that has been germinated and carefully dried (kilned) to produce high levels of α- and β-amylase (DU/DP). Some green (unkilned) malt is still used, but has in many distilleries been superseded by kilned malt. Green malt is normally cheaper to produce than kilned malt and can potentially be used at a lower dosage (malt inclusion rate), but has a shorter shelf-life and higher transportation costs than dried malt (Walker, 1986).

Prior to mashing, the malt is milled (usually using a hammer mill) into a fine flour, which is then slurried with process liquor (usually water). This is usually conditioned for a short period (20–30 minutes) at a suitable temperature (normally around 40°C) prior to mixing with the cooked cereal slurry. Green malt is normally wet-milled into a slurry before mixing with the cooked cereal. Typically the amount of malted barley used in a Scotch whisky grain distillery (malt inclusion rate) is around 10–11 per cent of the overall grain bill (on a dry weight basis), although this can vary for different distilleries and is up to 15 per cent in some cases.

It is essential that the malt is added as soon as possible after the cooker is discharged so that enzymes (mainly α-amylase) from the malt can begin to degrade the solubilized starch, reducing the viscosity, before it begins to retrograde. As described above, extended storage of cooked starch will result in retrogradation and the formation of resistant starch, which will not be efficiently degraded by the enzymes.

In many cases the malt slurry and cooked cereal slurry are mixed together in a mash tun or some other conversion vessel, and are held at mashing temperatures (62–65°C) for up to 30 minutes to convert the starch into fermentable

sugars – mainly maltose. In a continuous tube converter the residence time can be considerably shorter, due to the increased heat transfer and the more intimate contact between the malt enzymes and the cereal slurry. Robson (2001) describes a continuous conversion vessel in a grain distillery where the residence time is about 20 minutes on average. The conversion time should be sufficient for the complete conversion of starch to fermentable sugars.

The main problem associated with conversion is the incomplete conversion of starch into fermentable sugars, which is manifested by high levels of oligosaccharides and dextrins in the wort. This can be a result of poor temperature control during conversion, insufficient conversion time, or the presence of insufficient enzymes from the malt. This may be because the malt inclusion rate is too low or the malt is of poor quality. In addition, the pH levels may be too low for optimum enzyme performance (for example, as result of using too high a proportion of backset). One of the features that distinguishes grain distilling from brewing is that the wort is not boiled prior to fermentation, and this allows the enzymes to maintain their activity during fermentation (Sim and Berry, 1966). While the malt enzymes are still active and will continue slowly to degrade dextrins during fermentation, they are unable to degrade any substantial quantities of undegraded starch remaining after conversion if the conversion process is carried out inefficiently.

Starch hydrolysis

The major enzymes involved in the degradation of starch are the α- and β-amylases (Robyt, 1984), which degrade the α(1-4) links in the α-glycoside chains of starch. The first of these, α-amylase is the most important of the starch-degrading enzymes, and is largely responsible for the degradation of starch into lower molecular weight dextrins and sugars (Muller, 1991). The major form of this enzyme is heat stable up to 70°C and can operate at pH 6, in the presence of calcium ions, although its activity declines at temperatures above 67°C (Briggs et al, 1981).

The second enzyme, β-amylase, is essential in breaking unfermentable dextrins and oligosaccharides down into fermentable sugars, primarily maltose and to a lesser extent maltotriose. It is less heat stable than α-amylase and is denatured at normal mashing temperatures, with its activity completely disappearing after 40–60 minutes at 65°C (Briggs et al, 1981). A temperature of 67°C will leave substantial levels of dextrins instead of efficiently converting them to maltose (Palmer, 1989).

These facts underline the importance of careful temperature control during the conversion stage of grain distillery production.

Alpha-amylase is an endo-enzyme that rapidly degrades the α(1-4) bonds of starch molecules at random within the chains, producing a large number of progressively smaller oligosaccharides and dextrins. Alpha amylase can attack ungelatinized starch granules, but will only do so very slowly. The linear smaller oligosaccharides and dextrins are in turn degraded into

maltose by β-amylase, which is an exo-enzyme. This degrades starch residues by the stepwise release of maltose units from the non-reducing ends of the chains.

Figure 3.7 illustrates the actions of α- and β-amylase on starch. Neither of these enzymes can degrade the large number of α(1-6) branch points associated with amylopectin, and the significant amounts of branched residues or limit-dextrins remain in the mash (MacGregor *et al.*, 1999). These limit-dextrins are degraded by a third enzyme, limit-dextrinase, which specifically attacks the α (1-6) branch points of amylopectin and branched oligosaccharides to produce smaller, more linear components, which can be further degraded by α- and β-amylase. Although it is considered that limit-dextrinase will only degrade amylopectin itself very slowly and to a limited extent (Stenholm, 1997), the enzyme acts rapidly on limit-dextrins produced by the action of α-amylase (MacGregor *et al.*, 1999). More recent work (Walker *et al.*, 2001) suggests that the bulk of the limit dextrinase passes through the mashing stage in an inactive bound form and is released later on in the process, during fermentation.

The three major enzymes, α- and β-amylase and limit dextrinase, work together at mashing temperatures (62–65°C) to degrade starch progressively, first of all into large oligosaccharides, which are in turn degraded to a mixture of fermentable sugars, primarily maltose and maltotriose, and (mainly) branched dextrins. High levels of unconverted dextrins in the wort normally indicate problems with enzyme hydrolysis.
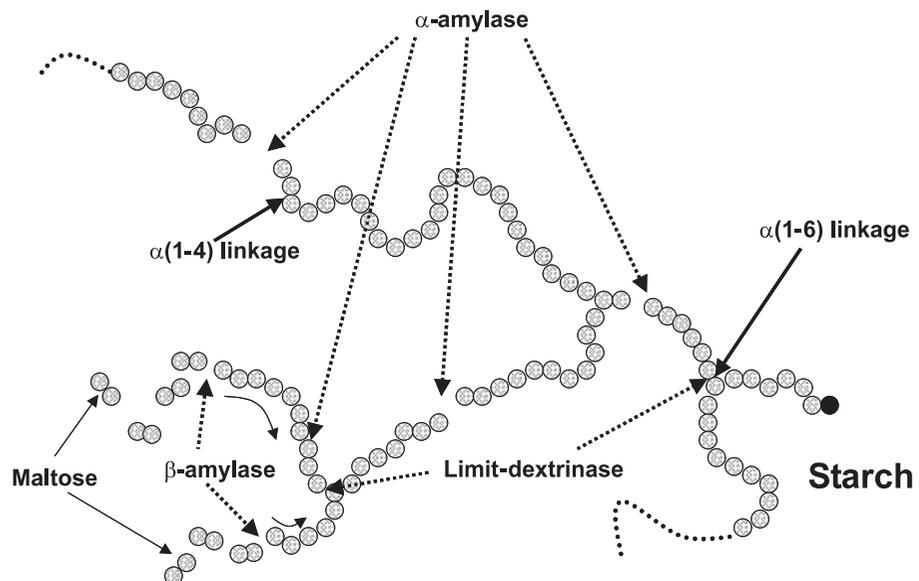

Figure 3.7
Degradation of starch by α- and β-amylase.

A fourth enzyme, α-glucosidase, is generally associated with the metabolization of starch during germination (Sun and Henson, 1992) but may also have a minor role in mashing (Agu and Palmer, 1997). This enzyme can release glucose units from a variety of other α-glucosides and small dextrins (Fincher and Stone, 1993); however, the contribution of this to the degradation of starch during conversion has not yet been fully established (MacGregor, 1991).

Unlike in brewing the wort is not boiled and the starch-degrading enzymes are able to survive into fermentation, where they have an important role in the further hydrolysis of dextrins, thus maximizing the amount of fermentable substrate potentially available for conversion into alcohol (Bringhurst *et al*, 2001).

Proteolysis

Another major function of conversion is that of proteolysis, by which proteins are broken down into amino acids and other low molecular weight nitrogen-bearing protein fragments (Boivin and Martel, 1991). These supply a source of essential nutrients that can be readily utilized by yeast, in order for it grow and operate efficiently during fermentation.

The area of proteolytic enzymes, particularly the endopeptidases, is probably the least well understood part of the mashing (conversion) process (Bamforth and Quain, 1989). However, it is generally accepted that the formation of soluble nitrogenous material in wort is primarily a result of the actions of heat-stable endoproteinases working in conjunction with heat-stable carboxypeptidases (Briggs *et al*., 1981; Boivin and Martel, 1991), which are able to tolerate the temperatures (up to 65°) associated with mashing and conversion (Bamforth and Quain, 1989).

The proteolytic enzymes include a mixture of proteinases, which are endo-enzymes, and peptidases, which are exo-enzymes. Endoproteinases (endopeptidases) break the internal peptide links of polypeptides (proteins) at random to produce smaller molecules, and these are in turn broken down by carboxypeptidases, which are exo-enzymes that remove amino acids step-wise from the carboxyl end of the chains (Bamforth and Quain, 1989).

However, many proteolytic enzymes (such as aminopeptidases) as well as some endoproteinases are heat labile above 55°C and are inactivated by the conditions encountered during mashing (Briggs *et al*., 1981; Jones and Marinac, 2002), and do not contribute significantly to the overall soluble nitrogen content of the wort.

Wort separation

Traditionally in grain distilleries the worts were separated in a mash tun after conversion and sparged several times with liquor at increasing temperatures

to provide a clear, filtered wort for fermentation, much in the same way as in malt distilleries (Rankin, 1977). However, this process is complex and time consuming and can be inefficient in relation to the production demands of a modern grain distillery.

One of the main features of modern grain distilleries is that generally the wort separation stage has been eliminated, to reduce wort processing times, so that after conversion the whole mash is transferred directly to the fermenter (Rankin, 1977) following cooling and pitching with yeast. This allows the use of higher original gravities (up to 1070°) than would otherwise be the case.

However, the use of unfiltered worts can result in problems with the fouling of stills during distillation, and may have serious downstream effects – particularly on the efficient operation of the evaporators that are used to collect co-products. Problems with the evaporators can lead to substantial process delays, thus affecting the efficiency of the whole grain distillery process. These problems can to some extent be alleviated by removing solid matter from the spent wash after distillation.

In some grain distilleries, as a result of processing constraints it has been considered necessary to have some sort of separation process before pumping the wort to the fermenter. The main advantage of using a wort separation process is that a liquid wort is pumped to the fermentation vessel, which gives fewer problems during subsequent distillation and processing of co-products. However, the filtration process itself can lead to delays in the production cycle, which can have serious implications for plant efficiency (Jameson *et al.*, 2001). There is also a greater probability of losing residual starch in the spent grains, which could lead to significant losses in alcohol yield. Additionally, since only about 10–15 per cent of the total protein is solubilized during mashing (Boivin and Martel, 1991), the bulk of the remaining unhydrolysed protein remains with the spent grains. This means that there may be a potential shortage of free amino nitrogen (FAN) to sustain an adequate level of yeast fermentation.

Several different means of separation have been employed in Scotch whisky grain distilleries. Originally worts were filtered using a lauter-type filtration system (Pyke, 1965), but these have now largely been superseded by other filtration systems that are adapted to more-or-less continuous operation. In such systems the mash is filtered through a series of filters or sieves (hydrosieves; Robson, 2001) and the grains sparged to remove soluble material; first and second worts are pumped to the fermenters while the recovered sparge (weak worts) is recycled to the process.

These operate largely in a similar way to the traditional malt distillery wort separation process, although the equipment used is completely different. Figure 3.8 shows the layout of a typical wort separation system.

The requirement for wort separation has largely been determined by the efficiency and economics of the particular process, and by the experience of individual companies with the technology.
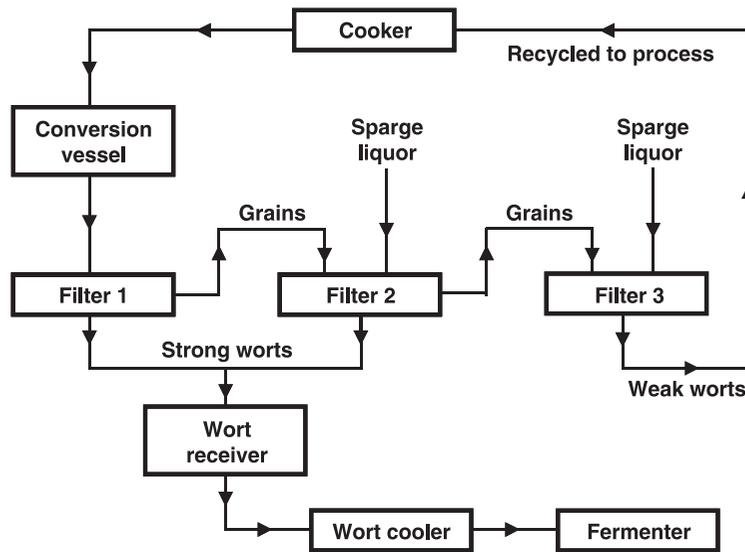
Figure 3.8
Wort filtration in a grain distillery.

Future developments

As this chapter shows, grain distillery processes are rather more adaptable to changes in technology and lend themselves more easily to technical innovation than malt distilleries, within the constraints of the legal definition of Scotch whisky. This degree of flexibility allows the use of a wide range of potential raw materials, the choice of which is essentially driven by market forces. In addition, the economics of grain distilling can promote the diversification from grain whisky to other neutral spirits for use as vodka and gin, in order to supply as wide a range of products as possible. Essentially the technology for producing these products is identical to that for grain whisky, with the possible exception of the distillation stage, which is not constrained by the limitations imposed by the Scotch Whisky Act (1988) and the Scotch Whisky Order 1990 (Statutory Instrument 1990). It is also possible to use commercial starch-degrading enzymes as an alternative to malt, which is relatively expensive, for products deriving from neutral spirits such as vodka and gin. However, in distilleries producing diverse streams of products there is a strict separation of the Scotch whisky production stream from that for other spirits to avoid contamination of the Scotch whisky process, thus maintaining its integrity.

So what are the likely developments for the future production of Scotch grain whisky? At present the technology used is largely determined by the scope of existing processes and the economics of how these can be developed,

in terms of the relative capital cost of modifying the existing process and that of adopting new technology, in response to market pressures defining the products in demand as well as the raw materials available.

The main raw material of choice for grain whisky is still soft winter wheat, although this may be sensitive to changes in the market for both wheat and maize. In addition there is concern that recent changes in the European Union intervention refunds for the wheat used by the Scotch whisky might some extent undermine the economic advantage of wheat over maize. This is one reason why grain distilleries may, where possible, wish to retain the capability to process maize as well as other cereals. However, wheat is still an attractive option for grain whisky production, and advances in the development of new wheat varieties that are suitable for distilling should ensure that the future of wheat is secure as an important raw material in its production.

As Bathgate (1989) suggests, because of the relatively low gelatinization temperature of wheat in relation to maize it should be possible to process wheat at lower temperatures than maize, and this might make it possible to use a low temperature cooking process to extract wheat starch. According to Newton *et al*. (1994), such a process could potentially give higher alcohol yields as a result either of increased protein breakdown at the lower temperatures or of the reduction of losses arising from the browning reactions that take place at higher temperatures.

Some 'no or cold cooking' processes were reviewed by Wilkin (1989), and these were used with varying degrees of success as long ago as 1980. In these processes cereal and tuber starches were used to produce substantial alcohol yields at temperatures as low as 55°C. However, the raw materials had to be milled 'ultrafine', and this used significant amounts of the energy saved by reducing the cooking temperature. Additional problems were encountered with the incomplete release and saccharification of the starch granules, which could only be resolved by resorting to the addition of exogenous enzymes such as proteases and hemicellulases, and as such could not be used for the manufacture of Scotch whisky as it is currently defined. It is possible that with the advent of suitable raw materials adapted for this type of process, such as specially selected new cultivars of wheat and barley, it might eventually be feasible to use such processes economically in the future.

An obvious direction for Scotch grain whisky would be to modify the process to make it truly continuous (Rankin, 1990), although it appears that the current trend is in the opposite direction towards batch production, in response to shifts and fluctuations in the market for Scotch whisky.

Whitby (1995) suggests that in the long term distilleries may eventually become fully integrated, with the maximum production of alcohol by the more efficient utilization of the raw materials and minimum use of energy. However, it is difficult to say whether the Scotch whisky industry is prepared to move fully in the direction of whole crop utilization, as described by Petersen and Munck (1993). This approach is very similar to the Latvian model described by Bekers *et al*. (1997), where the production process in the distillery is a part of a closed biotechnology system, allowing the complete utilization of the raw materials to produce a wide range of marketable co-

products as well as ethanol (whisky) – effectively becoming a biorefinery as postulated by Audsley and Sells (1997). Thus ultimately the future direction of distilling will probably be as processors of grain rather than exclusively as alcohol producers (Lyons, 1999).

Potential future technological developments for the production of Scotch grain whisky are largely already in place in other sectors, and are currently in use for the production of a wide range of other cereal, fermentation and distillation products for industries ranging from brewing to pharmaceuticals and fuel alcohol. However, the adoption of new technologies for cereal processing into Scotch grain whisky will be dependent on market pressures and other economic factors facing the Scotch whisky industry during the twenty-first century. The challenge facing the Scotch whisky industry will be how to tap into this new technology while maintaining its market position, but without losing the distinctive quality of its products.

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Chapter *4*

Yeast and fermentation

Iain Campbell

Introduction

Compared with the quantity of literature on the biochemistry and microbiology of beer and wine fermentations, or of popular literature on whisky itself, there is little scientific information regarding Scotch whisky fermentations. The reviews by Berry and Ramsay (1983) and Korhola *et al*. (1989) are among the few specifically concerned with that stage of whisky production. Fortunately the progress of fermentation is similar in a brewery and a distillery, and much of the information for a general understanding of fermentation is provided in literature related to brewing (see, for example, Hough *et al*., 1982; Young, 1996; Boulton and Quain, 2001; Slaughter, 2002). These reviews already provide authoritative explanations of the alcoholic fermentation of cereal-based wort, so this chapter is more concerned with the practical differences between brewery and distillery fermentations, and makes no attempt to discuss distilled products other than Scotch whisky.

In both malt and grain whisky distilleries the principal sources of the congeners in new-make spirit are:

- The malt, and the cereal of grain distilling
- Volatile structural components of the yeast
- Metabolic products of yeast growth
- Microbial contaminants of fermentation.

Although the more efficient rectification of continuously distilled spirit reduces the flavour contribution of these four factors in comparison with the effect of subsequent maturation, both the yeast itself and the progress of fermentation are important contributors to the character of these two types of Scotch whisky. In fact, that is a legal obligation: the Scotch Whisky Regulations require whisky to retain the aroma and taste from fermentation by yeast of an all-malt or malt-hydrolysed cereal mash.

Yeast for alcohol fermentation

The generic name *Saccharomyces* (Latin: sugar fungus) was first used by Meyen in 1838, but modern taxonomy of yeasts of the alcohol fermentation industries is largely derived from the work of Hansen at the Carlsberg Laboratories in Denmark in the 1880s (Barnett *et al*., 1990). Hansen allocated different specific names to the head-forming yeast of British ales and the similar beers of Belgium and northern Germany (*S. cerevisiae*), the non-head-forming yeast associated with the lower temperature range of lager fermentations (*S. carlsbergensis*), and the more alcohol-tolerant wine yeast with characteristic ellipsoidal cells (*S. ellipsoideus*). Since then the classification has been frequently revised, with the present result that all three of these former species are now named *S. cerevisiae*. While this is justified by the rules of botanical (including fungal) taxonomy, it is inconvenient that yeasts of such different industrial properties do not have official names to differentiate them (Campbell, 1996a). The starch-fermenting wild yeast and brewery contaminant initially isolated as *S. diastaticus* is also now included in *S. cerevisiae*.

Originally bakers and whisky distillers operated reasonably successful fermentations with the excess yeast generated by the brewing industry, but the breeding of hybrids with improved properties for the baking industry stimulated equivalent development of specialized distilling yeast (Fowell, 1967). Obviously ale yeast was an important component of the hybrid, but, because of its amylolytic properties, *S. diastaticus* was also used. Unlike the almost spherical brewing yeast, the distilling hybrid has an obviously elliptical shape with approximate dimensions of $10\,\mu m$ long by $5\,\mu m$ in diameter. In much later genetic manipulation to develop amylolytic brewing yeast, the 'phenolic off-flavour' transferred to the brewing hybrid from *S. diastaticus* was a serious problem (Tubb *et al*., 1981; Hammond 1996). The problem was caused by decarboxylation of cinnamic acids, e.g. ferulic acid to 4-vinyl guaiacol (4VG), but, being related to the flavours derived from peated malt, 4VG and similar phenolic notes are a desirable property for a distilling hybrid. Of the sugars derived from hydrolysis of starch, brewing yeast ferments only glucose, maltose and maltotriose. In practice, the fermentative contribution introduced into the distilling hybrid from *S. diastaticus* is limited to fermentation of maltotetraose and several otherwise unfermentable starch-derived di- and tri-saccharides. Although it is theoretically possible to prepare a fully amylolytic yeast, to do so would contravene the part of the Scotch Whisky Regulations that stipulates that the hydrolysis of the cereal starch must be entirely by the enzymes of malt. The essential properties of distilling yeast are:

- Good flavour production
- Complete and rapid fermentation of wort sugars
- Tolerance of the osmotic stress of the initial sugar concentration in the wort, in modern practice at least 16 per cent and possibly 20 per cent
- The ability to complete the fermentation in the final 8–10 per cent ethanol content of the wash

- Lack of flocculence and minimal frothing
- The ability to grow well above 30°C.

Korhola *et al*. (1989) reported distillery yeasts capable of fermentation at 46°C, with the advantage of reducing the energy requirement of heating the wash for distillation. However, such strains are not used in Scotch whisky production, where the maximum growth temperature is about 35°C.

Yeast biochemistry

The most obvious effect of fermentation is the production of ethanol and carbon dioxide, with ethanol produced to approximately half of the initial concentration of fermentable sugar. However, the biological function of the fermentation is to provide energy for yeast growth, and therefore inevitably part of the fermentable sugar is converted to yeast mass rather than the commercially desired ethanol. The primary concern of the distiller is maintenance of spirit quality, but an important economic consideration is an acceptably high spirit yield from the raw materials – and spirit yield falls with increased yeast growth.

Various organic compounds can be metabolized by *S. cerevisiae* under aerobic conditions (Barnett *et al*., 1990), but for anaerobic fermentation glucose or another fermentable sugar is essential as a source of both energy and the carbon-containing structural materials of new cell material. Although almost all yeasts are capable of growth on ammonium salts as the sole source of nitrogen requirements for synthesis of proteins (mainly enzymes) and nucleic acids (Barnett *et al*., 1990; Slaughter, 2002), in distillery fermentations the amino acids and simple peptides of wort function as the nitrogen source.

For growth and fermentation to occur, the yeast requires suitable pH, temperature and nutrients. Like fungi in general, *S. cerevisiae* prefers acid pH. The optimum pH is about 5.0–5.2, but distillery yeast is capable of good growth over the pH range 6.0–3.5 approximately. For distillery yeasts used in Scotland, the optimum temperature (in the sense of fastest growth and therefore most efficient fermentation) occurs at about 30–33°C. Although distillery yeasts are capable of growth over the range 5–35°C, the rate of growth at temperatures below about 25°C is too slow for whisky fermentations.

Various mineral salts are also required, particularly phosphate and sulphate, and many metal ions are required in trace amounts as enzyme cofactors – iron, potassium, manganese and zinc being the most important (Walker, 1998; Walker and Birch, 1998). Normally the ionic content of grain or malt distillery wort is adequate, but any deficiency will result in a sluggish fermentation since mineral or vitamin supplements cannot be added as in a brewery. Biotin is the only organic growth factor (equivalent to the term vitamin in human nutrition) that is certain to be required by the yeast, but it is present in adequate concentration in the wort of malt and grain distilleries. Otherwise, most strains of *S. cerevisiae* are capable of synthesizing their own

supply of the vitamins that humans require in their diet: pantothenate, ribo-flavin, thiamine, etc. However, under the anaerobic conditions of fermentation, brewery and distillery yeasts are unable to synthesize the unsaturated fatty acids and sterols that are essential structural components of cell membranes. Since the amounts naturally present in the wort are insufficient for anaerobic growth, aeration of wort at the time of pitching the yeast allows the yeast inoculum to synthesize a reserve of these compounds under aerobic conditions.

Flavour congeners are produced as by-products of the fermentation of sugars to alcohol. Although only the principal types of congener are shown in Table 4.1, Nykanen and Suomalainen (1983) listed approximately 400 flavour metabolites formed during alcoholic fermentation by yeast and contributing to the flavour of beers, ciders, wines and distilled spirits. These compounds vary in their importance to the quality of the final product. For any alcoholic beverage the actual amount of each compound is less important than its aroma threshold – the concentration detectable by a trained sensory panel. However, volatility is also important in the case of distilled beverages, and the volatility of flavour metabolites influences the amount collected in the spirit fraction for malt whisky or at the spirit plate of a continuous still.

Table 4.1
Products of yeast fermentation (modified from Suomalainen, 1971)

Alcohols	Acids	Esters	Others
Ethanol	Acetic	Ethyl acetate and any other combination of acids and alcohols on the left	Carbon dioxide
Propanol	Caproic		Acetaldehyde
Butanols	Caprylic		Diacetyl
Amyl alcohol	Lactic		Hydrogen sulphide
Glycerol	Pyruvic		
Phenylethanol	Succinic		

Fermentability of wort

Part of malt analysis, measurement of fermentability and prediction of spirit yield are also relevant to the fermentation stage. A standard hot-water extract (method 2.15 of Baker, 1991) is used as experimental wort, which is not boiled in order to preserve the activity of malt enzymes during the test fermentation by the M strain of distiller's yeast. This is derived from the original distilling M strain of 1952 (see Fowell, 1967), but since improved strains introduced later were still named M, its properties have not been constant over the last 50

years. The standard method specifies the use of commercial M yeast cake, but since that is not necessarily a pure culture there is some advantage in using a guaranteed pure culture obtained by plating out commercial M yeast to obtain single colonies.

From the measured fall in gravity over 44 hours' incubation at 33°C, a percentage fermentable extract in actual distillery practice can be calculated (Bathgate, 1989; Bringhurst *et al.*, 1996; Dolan, 2000). From that figure a prediction of the spirit yield per tonne of malt can be derived – an essential aspect of analysis of malt and cereal, but a figure largely dependent on the efficiency of starch hydrolysis during both mashing and fermentation.

Yeast structures

The outermost layer of the cell is a rigid wall of fibrous β(1-3)- and (1-6)-glucans with an outer, more amorphous, layer of mannan, mainly α(1-4) but with α(1-2) and α(1-3) side chains. Since the scars left by separation of successive buds are composed of chitin, a polymer of N-acetyl glucosamine, an increasing proportion of chitin develops in the walls of older cells. The cell wall constitutes 15–25 per cent of the dry weight of the cell, and has many functions (Stratford, 1992): physical protection, osmotic stability, support for wall-bound enzymes (e.g. invertase), cell–cell adhesion (e.g. flocculation), and as a selective permeability barrier. Although it is convenient to regard the wall as porous to nutrients and metabolites, larger polysaccharide and polypeptide molecules are blocked by the pore size of the wall (de Nobel and Barnett, 1991).

It is the cell membrane (cytoplasmic membrane) that prevents the leakage of soluble components of the cytoplasm into the surrounding medium, but it is equally effective in the opposite direction and prevents the free diffusion of nutrients into the cell. True diffusion across the membrane, at a rate determined by molecular size, solubility in membrane lipids and concentration difference between the culture medium and cytoplasm, is limited to a few simple compounds such as ethanol and carbon dioxide (Slaughter, 2002). For all other compounds, specific permease enzymes are required for the import of nutrients and export of metabolites. Transport may be by facilitated diffusion, where the energy is provided by the concentration difference across the membrane, or by active transport, requiring energy input by the cell in addition to the specific transport system (Young, 1996; Slaughter, 2002).

Saccharomyces species grow by budding. Only one bud scar is shown in Figure 4.1: if that actually is the only scar on the cell, it represents the 'birth scar' left after detaching from its mother cell. If so, the first bud produced by the new yeast cell will be at the other end – not necessarily exactly opposite the birth scar, but more probably near the point where the nucleus is closest to the cell wall, to control bud initiation. The first stage is local softening, by precisely directed β(1-6)-glucanase, of the glucan that gives the cell wall its rigidity. Osmotic pressure within the cell causes swelling, the first microscopically visible evidence of bud development. Subsequently, synthesis of new cell
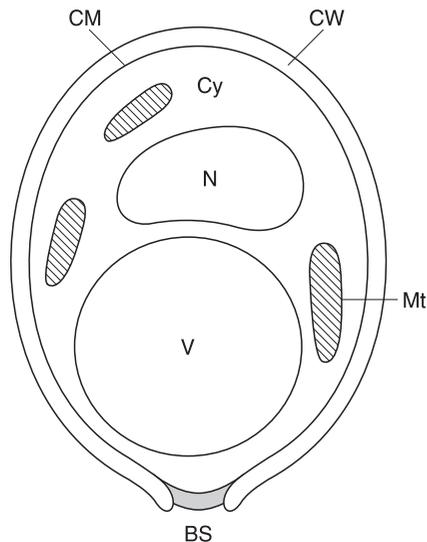
Figure 4.1
Principal structures of an aerobically grown cell of distilling yeast *Saccharomyces cerevisiae*. BS, bud scar; CW, cell wall; CM, cytoplasmic membrane (cell membrane); Mt, mitochondrion; N, nucleus; V, vacuole.

wall, membrane, and nuclear and cytoplasmic material is precisely programmed over the generation time of the cell and the fully formed daughter cell detaches from its mother for both to start their next cell cycle independently. The process was illustrated by Matile *et al*. (1969) in an excellent succession of drawings of electron micrographs. Although parts of their text have been overtaken by more recent research, the drawings are still perfectly valid to complement Wheals' (1987) review of the cell generation cycle.

Various organelles exist within the cytoplasm (Rose and Harrison, 1991). The most important are shown in Figure 4.1: the nucleus, mitochondria and vacuole. These also are enclosed within membranes of the same structure and function as the cytoplasmic membrane. In the resting stage depicted in Figure 4.1 the vacuole exists as a single organelle, the largest within the cell, but during the generation cycle the vacuole fragments and some units migrate to the developing bud before the entry of the divided nucleus. Obviously it is essential that the nuclear genetic material is replicated accurately during cell division, but it is equally important that units of divided mitochondria and vacuole enter the developing bud at the appropriate stage of cell division (Matile *et al*., 1969). Among the biochemical functions of the vacuole are the recycling of proteins and storage of glycogen as an energy reserve, but it also appears to have the mechanical function of forcing into the developing bud its share of the replicated nucleus.

Mitochondria are essential organelles for the oxidative growth of yeast (Nagley *et al*., 1977) and are well developed in aerobically grown baking or

distilling yeast (Figure 4.1). Among other activities, these organelles are associated with the enzyme systems of oxidative metabolism (Nagley *et al*., 1977; O'Connor-Cox *et al*., 1996). The importance of mitochondria to fermentation is discussed further below, under oxygen metabolism, but Rose and Harrison (1991) provide more general biological information on these and other organelles of the yeast cell.

Carbohydrate metabolism

There are three important aspects of carbohydrate metabolism during fermentation:

1. Uptake of the fermentable sugars from the wort
2. Production of ethanol and flavour congeners within the yeast cells
3. Excretion of the metabolites as flavour of the wash and ultimately of the whisky.

Table 4.2 shows the composition of all-malt wort, but it has been realized since the 1960s that these sugars are not utilized simultaneously. Although present in the greatest amount, maltose is not utilized initially by the yeast. The specific permease for glucose is a constitutive enzyme that is immediately available at pitching, whereas the maltose transport system is inducible.

Table 4.2

Approximate sugar composition of all-malt wort, expressed as percentage of total carbohydrate (Palmer, 1989). Carbohydrate constitutes approximately 90% of total solids of wort

Monosaccharide (hexose)	
Fructose	1%
Glucose	10%
Disaccharide	
Maltose	46%
Sucrose	5%
Tri- and tetra-saccharide	
Maltotriose	15%
Maltotetraose	10%
Wort carbohydrates not fermentable by distilling yeast	
Maltopentaose and higher dextrins	13% (at collection in washback; reduced by continuing enzymic action during fermentation)

Similar figures apply to grain distillery worts (Pyke, 1965).

Inducible enzymes are synthesized only in the presence of their substrate, but in addition, synthesis of maltose permease is not activated until the glucose concentration falls below a threshold concentration. Similarly, maltotriose transport is not induced until maltose concentration in the wort has fallen sufficiently. Therefore the uptake of fermentable sugars is normally as shown in Figure 4.2.

The mode of operation of the transport system also varies with different sugars. The uptake of the main carbohydrate nutrients of a typical malt wort is shown in Figure 4.3. Glucose and fructose are transported intact through the cell membrane (shown in this diagram correctly as a double layer of lipid) by facilitated diffusion with their appropriate permease, which is located within the membrane (Meaden, 1993). Before reaching the cell membrane, sucrose is hydrolysed within the cell wall by a wall-bound invertase system, and it is the resulting glucose and fructose that are transported into the cell. Maltose and maltotriose enter the cell by specific energy-requiring active transport systems, which phosphorylate these sugars. A detailed explanation of modern concepts of the structure and mode of operation of permease enzymes in yeast is provided by Slaughter (2002).

Aerobically the sugar is fully oxidized to carbon dioxide and water, releasing the same amount of energy as obtained by chemical combustion of glucose, but in a biological system the energy is released and stored in non-lethal
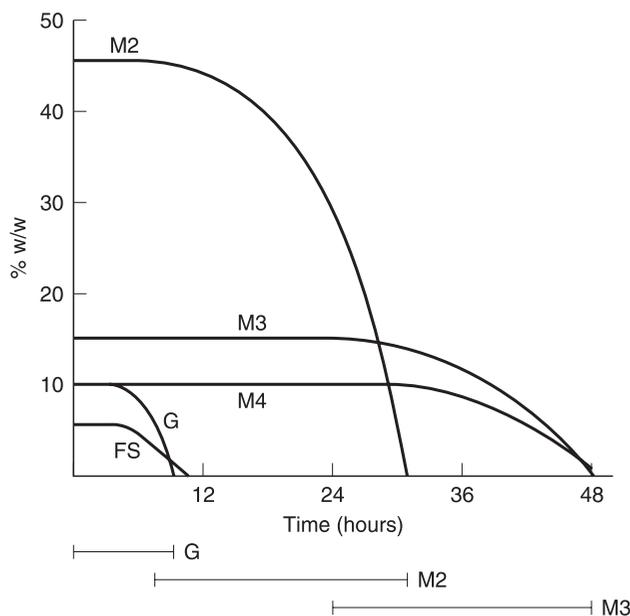

Figure 4.2
Utilization of principal wort sugars by distillery yeast. G, glucose; FS, fructose + sucrose; M2, maltose; M3, maltotriose; M4, maltotetraose. The horizontal bars show the duration of the relevant permease activity.
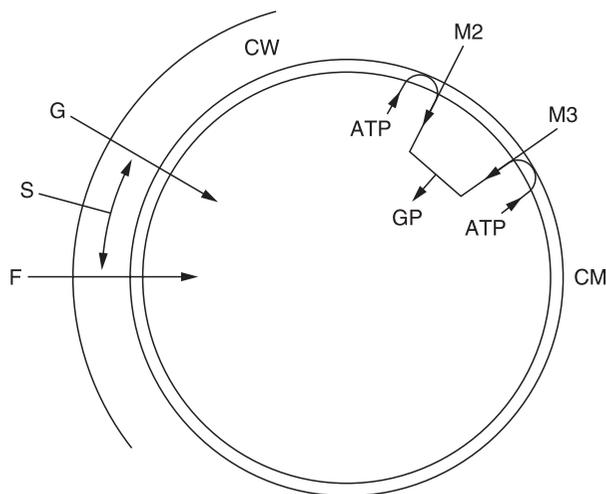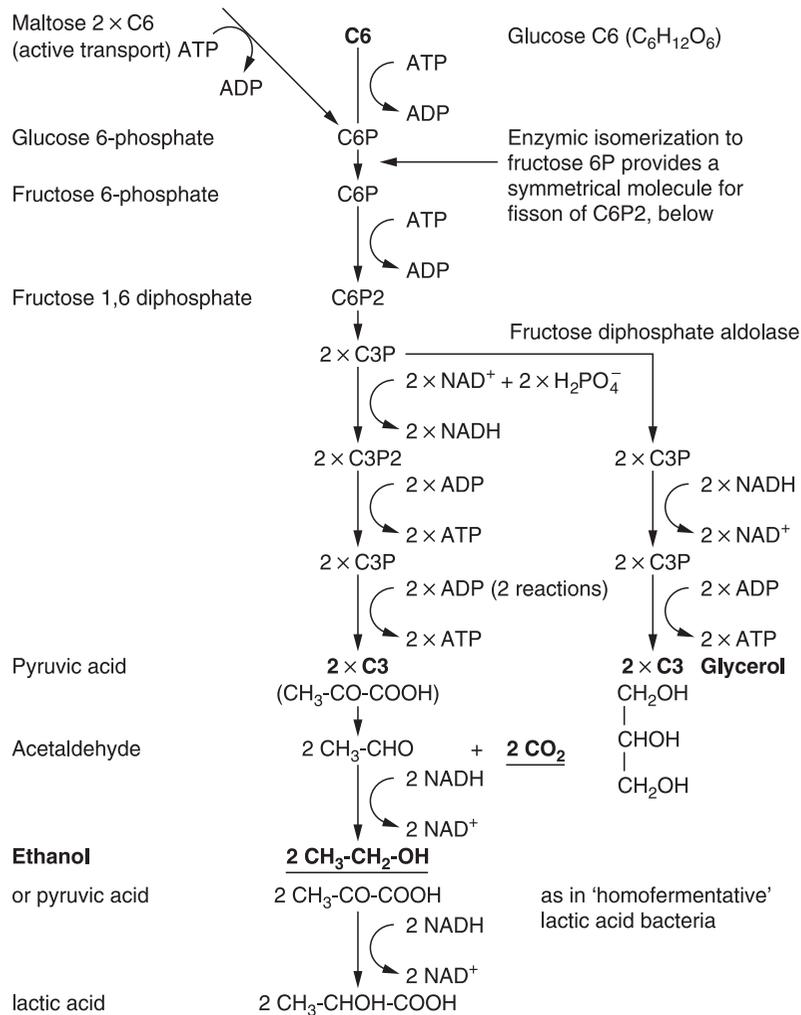
Figure 4.3
Transport of fermentable sugars into *Saccharomyces cerevisiae*. F, fructose; G, glucose;
GP, glucose phosphate; M2, maltose; M3, maltotriose; S, sucrose; CW, cell wall; CM,
cytoplasmic membrane.

amounts as energy-rich compounds. Although others are possible, the normal
energy store is the energy-rich bond coupling a third phosphate group to
adenosine diphosphate (ADP) to form the triphosphate, ATP. Anaerobically
only partial oxidation is possible, to pyruvic acid. Details of the fermentative
metabolism of hexose sugars are available in all biochemistry textbooks, and
only a basic outline of the Embden–Meyerhof–Parnas metabolic pathway is
shown in Figure 4.4.

For simplicity, few of the enzymes of the co-ordinated sequence of the path-
way are named in Figure 4.4. The first steps of the pathway involve phosphor-
ylation of sugar, an energy-requiring process requiring two molecules of ATP
per molecule of hexose. However, maltose is already phosphorylated during
transport and before hydrolysis to glucose, so the first phosphorylation step of
the metabolism of hexose is unnecessary. Also, whatever the original structure
of the hexose, it must be isomerized to fructose-6-phosphate. Only with the
specific structure of fructose can the 1,6 diphosphate of the following step be
split to two identical molecules of triose phosphate (glyceraldehyde 3-phos-
phate).

Phosphorylation does not necessarily require the energy of ATP; later in the
pathway phosphorylation by inorganic phosphate occurs with the reduction of
nicotinamide adenine dinucleotide (NAD) to $NADH_2$. (An alternative name
for NAD, as used in some textbooks and research papers, is diphosphopyr-
idine nucleotide, DPN.) In Figure 4.4 the reaction is shown more correctly as
NAD^+ reduced to NADH as glyceraldehyde 3-phosphate is phosphorylated to
1,3-diphosphoglycerate. Subsequent reactions involve transfer of phosphate to
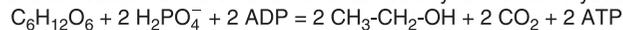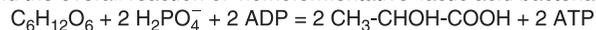
Figure 4.4
Summary of Embden–Meyerhof–Parnas metabolic pathway (modified from Hough *et al.*, 1982).

ADP and isomerization to prepare the triose phosphate for release of its phosphate, generating a total of four ATP molecules for each molecule of hexose. Since two are required to 'pay back' the initial use of two ATP molecules, the overall effect of the pathway is a net gain of the energy of two ATP molecules, which is sufficient for the biosynthetic activities of the growing cell.

In fermentative yeasts, subsequent re-oxidation of NADH to NAD$^+$ by acetaldehyde is required to allow metabolism to continue with the limited quantity of NAD within the cell, and the two hydrogen atoms removed in the re-oxidation of each molecule of NAD reduce acetaldehyde to ethanol. Decarboxylation of pyruvate and formation of acetaldehyde and ethanol is only one of many possible methods for recovery of NAD$^+$: in many lactic acid bacteria, for example, a single step of reduction of pyruvic to lactic acid provides for re-oxidation of NADH.

The branch route to glycerol normally represents only a small part of carbohydrate metabolism. Pyke (1965) reported 0.25 per cent w/v in grain distillery wash, substantially above the levels of higher alcohols and esters, but its volatility is too low for glycerol to appear in the spirit. The flavour congeners directly produced by the main route of the EMP pathway are pyruvic acid and acetaldehyde, but many other acids, alcohols and esters are produced as by-products of biosynthetic activities of the yeast (Table 4.1). However, the majority of these flavour congeners are by-products of biosynthesis of lipids, nucleic acids and proteins, as shown later in this chapter.

EMP is only one of the possible pathways of energy-yielding carbohydrate metabolism, but is the important pathway under the anaerobic conditions of fermentation. Hough *et al*. (1982) provided an adequate account of the hexose monophosphate (or pentose phosphate) pathway, krebs cycle and other metabolic activities relevant to aerobic growth of brewing yeast (and therefore also the aerobic propagation of distilling yeast), but a more detailed explanation can be found in any biochemistry textbook.

Nitrogen metabolism

The amino acids required for yeast growth are produced by hydrolysis of malt protein during malting, and the amount of α-amino nitrogen in the wort is partly related to the degree of modification of the malt. The higher the α-amino nitrogen content of the wort the greater is the amount of yeast growth (ultimately above about 150–180 mg/l), reducing spirit yield by conversion of sugar to cell mass rather than ethanol. Also, higher nitrogen levels mean proportionally lower carbohydrate content in the barley and malt. Given the relatively low nitrogen content of maize and wheat, this is unlikely to be a problem in grain distilleries, and a higher nitrogen content of barley is usually associated with higher enzyme activity – an important property of grain distillery malt.

It is impossible for the limited space of the yeast cell membrane to contain individual permease enzymes for each of the twenty structural amino acids of proteins. Only a few amino acids have specific transport enzymes, and most have to share. Although a few of the amino acids of protein structure were not mentioned by Jones and Pierce (1964) or Pierce (1987), Table 4.3 shows their comparison of the rate of uptake of individual amino acids – effectively a summary of their transport systems. Only aspartic acid/asparagine, glutamic acid/glutamine and lysine have their own specific transport enzymes. Since
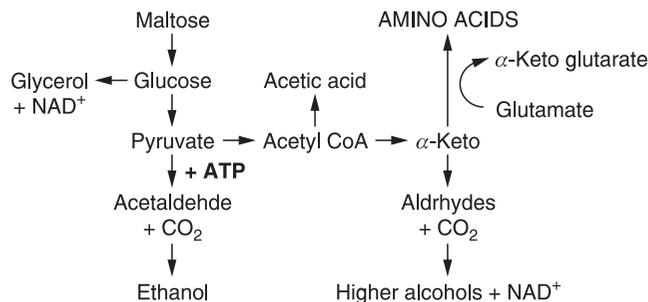
Table 4.3

Classification of amino acids by their rate of absorption from wort (Jones and Pierce, 1964; Pierce, 1987)

Group A	Group B	Group C	Group D
Rapidly absorbed from start of fermentation	Slowly absorbed from start of fermentation	Slowly absorbed, late in fermentation	Absorbed slowly over fermentation
Aspartic acid/amine	Histidine	Alanine	Proline
Glutamic acid/amine	Isoleucine	Glycine	Hydroxyproline
Lysine	Leucine	Phenylalanine	
Argine, serine, threonine	Methionine	Tryptophan	
	Valine	Tyrosine	

arginine, serine and threonine share a single permease, the rate and amount of uptake of each individual amino acid is approximately in proportion to their relative amounts in the wort, but all are absorbed from wort at a sufficiently rapid rate to satisfy the requirements of the growing cells for these amino acids. Proline and hydroxyproline are absorbed only slowly, but meet the requirements of the cell throughout fermentation. However, since all amino acids listed as 'group B' share a single permease, they are not absorbed in sufficient amount to meet the requirements of protein synthesis during active growth of the yeast. All amino acids can be transported by a general amino acid permease (GAP), but that is inactive while specific transport of the group A and B acids is in operation. Therefore the amino acids listed as group C, which are totally dependent on GAP for transport, could be absorbed only late in the fermentation, too late be incorporated directly into enzyme structure during the phase of active growth. For the development of the next generation bud the growing yeast cell must synthesize its own weight of cell material from the nutrients in the wort. That biosynthetic activity includes all of its requirements of group C amino acids and the necessary supplement of the limited uptake of group B. Since lysine cannot be used for that purpose by Saccharomyces spp., the amino groups of aspartic acid/asparagine and glutamic acid/glutamine provide the amino nitrogen for biosynthesis of other amino acids.
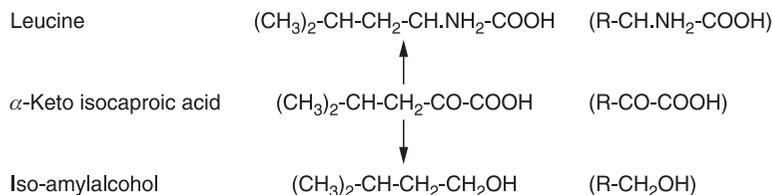
Figure 4.5 shows a basic summary of the formation of the various keto-acid intermediates required for synthesis of amino acids (Quain, 1988; Slaughter, 2002). Although enzyme-mediated reactions are normally reversible, some of the steps of keto-acid biosynthesis are not. If the supply of amino nitrogen is exhausted, obviously the keto acids cannot be converted to the intended amino acids. However, these oxidized compounds, keto acids, represent a valuable resource that cannot be permitted to accumulate under anaerobic conditions.

Maltose   AMINO ACIDS

Glycerol ◄— Glucose   Acetic acid α-Keto glutarate
+ NAD$^+$ Glutamate

Pyruvate —► Acetyl CoA —► α-Keto

+ ATP

Acetaldehde   Aldrhydes
+ CO$_2$ + CO$_2$

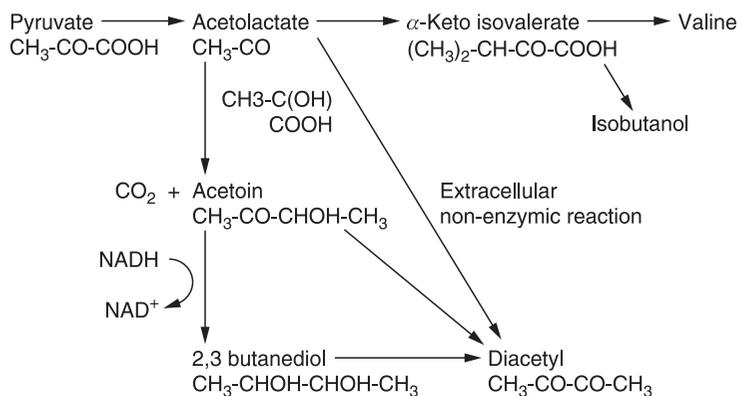Ethanol   Higher alcohols + NAD$^+$

Figure 4.5
Biosynthesis of amino acids or higher alcohols by *Saccharomyces cerevisiae*. Pathways generate an excess of ATP or oxidized NAD as shown.

Hence if they cannot be converted to amino acids, the keto acids are decarboxylated to the equivalent aldehyde and reduced to an alcohol by analogous reactions to the metabolism of pyruvate to ethanol. A general alcohol dehydrogenase reduces the aldehyde to its corresponding alcohol, although a specific decarboxylase is required for each keto acid. Therefore if nitrogen depletion prevents conversion of α-keto isocaproic acid to leucine, isoamyl alcohol is formed instead (Figure 4.6). This relationship between amino acids and higher alcohols was first noted by Ehrlich in 1911, but the reactions were not fully explained until the 1950s. It is also possible that even though amino-N is available, the yeast may convert part of its reserve of keto acids to higher alcohols in order to generate oxidized NAD (Quain, 1988; Slaughter, 2002). This response to a redox problem is an analogous reaction to the production of glycerol by the Embden–Meyerhof–Parnas pathway: whereas the pyruvate route is exactly balanced in NAD and generates a 'profit' of ATP, the glycerol branch is balanced with respect to ATP and provides oxidized NAD for use in biosynthetic reactions under anaerobic conditions.

Diacetyl is another by-product of nitrogen metabolism. Figure 4.7 shows that isobutanol is the higher alcohol associated with the biosynthesis of valine, but acetolactate, an intermediate in the biosynthetic pathway, is decarboxylated and reduced to acetoin and 2,3 butanediol by similar reactions to ethanol

Leucine $(CH_3)_2$-CH-CH$_2$-CH.NH$_2$-COOH   (R-CH.NH$_2$-COOH)

α-Keto isocaproic acid $(CH_3)_2$-CH-CH$_2$-CO-COOH   (R-CO-COOH)

Iso-amylalcohol $(CH_3)_2$-CH-CH$_2$-CH$_2$OH   (R-CH$_2$OH)

Figure 4.6
Structural relationship between amino acids and higher alcohols.

Figure 4.7
Relationship of valine biosynthesis to formation of isobutanol and diacetyl.

formation (Young, 1996; Slaughter, 2002). However, traces of these three compounds excreted into the wash can be oxidized by non-enzymic chemical reactions to diacetyl, for example by the atmospheric oxygen dissolved by the turbulence of filling the still charger vessel, or aerating the wash for continuous distillation. Since chemical reactions are accelerated by higher temperature, conversion to diacetyl is rapid in the early stages of distillation. With its low aroma threshold (approximately 1 ppm) and similar volatility to ethanol, diacetyl is the most important congener shown on Figure 4.7, and, incidentally is one of the few compounds that it is impossible to remove completely from neutral spirit.

Fatty acid and ester production

Esters represent another group of the flavour congeners formed during fermentation. Although non-enzymic esterification of acids and alcohols is an important part of flavour development during maturation of whisky, that reaction is too slow to account for the esters produced during fermentation. Production of esters is related to the recycling of the enzyme cofactor, coenzyme A (CoA) (Nordstrom, 1964; Peddie, 1990). Acetic acid and longer-chain fatty acids are important intermediates in biosynthetic activities, but a proportion of these compounds is lost to the culture medium, to appear as flavour congeners (Table 4.1). Acetyl CoA and its higher homologues, collectively indicated as R-CO~S.CoA in Figure 4.8, are important intermediates in the biosynthesis of enzyme proteins, nucleic acids and lipids. Note, incidentally, the unusual removal of two phosphate groups from ATP (to form adenosine monophosphate) in transferring the energy-rich bond from ATP to the CoA complex. If, however, the acetyl or higher acyl CoA is not required, it must be recycled to recover the energy conventionally represented by the ~ bond, and maintain the limited intracellular supply of CoA itself. On removal of the CoA
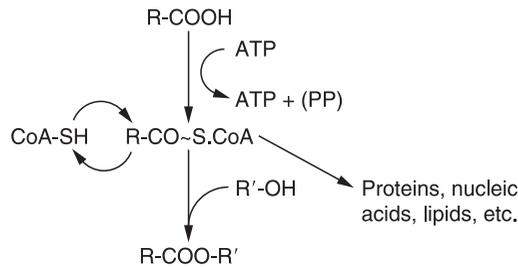
R-COOH

ATP

ATP + (PP)

CoA-SH   R-CO~S.CoA

R'-OH   Proteins, nucleic
                        acids, lipids, etc.

R-COO-R'

Figure 4.8
Formation of esters by recycling of coenzyme A.

the acid group is stabilized by esterification, with the involvement of an ester-
ase enzyme (esterases catalyse the reaction in either direction).

Since acetate and ethanol are the acid and alcohol formed in greatest
amounts during fermentation, naturally ethyl acetate is the principal ester in
quantity, although other esters with lower aroma threshold have more effect
on the wash, spirit and final whisky. The wider implications of recycling CoA
are shown in Figure 4.9, which indicates the sources of ethanol, higher alcohols
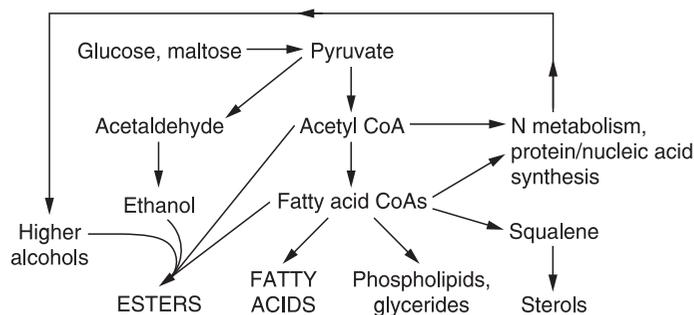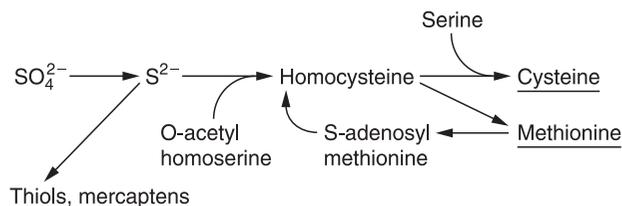and the various acid groups involved in ester production.

Glucose, maltose ⟶ Pyruvate

Acetaldehyde   Acetyl CoA ⟶ N metabolism,
                                        protein/nucleic acid
                                        synthesis

Ethanol   Fatty acid CoAs

Higher   Squalene
alcohols
                        FATTY   Phospholipids,
        ESTERS   ACIDS   glycerides   Sterols

Figure 4.9
Formation of esters and fatty acids as by-products of yeast growth.

Sulphur metabolism

As in brewing and wine-making, two types of reaction are particularly impor-
tant in the production of sulphur flavours in distillery fermentations: biosynth-
esis of sulphur-containing amino acids (Figure 4.10) and reduction of sulphate
salts in the wort. Distilling differs from these other processes in benefiting
from the remedial effects of the copper in the stills, but with the normal
ratio of copper surface area to pot still volume it is impossible (and undesir-
able) to remove sulphur compounds completely.

Serine

$SO_4^{2-} \longrightarrow S^{2-} \longrightarrow$ Homocysteine $\longrightarrow$ Cysteine

O-acetyl   S-adenosyl $\longleftarrow$ Methionine
homoserine   methionine

Thiols, mercaptens

Figure 4.10
Formation of sulphur compounds as by-products of synthesis of sulphur-containing amino acids (modified from Doyle and Slaughter, 1998).

So the biosynthesis of cysteine and methionine creates analogous flavour problems to the biosynthesis of the other amino acids (Doyle and Slaughter, 1998; Slaughter, 2002), but the flavour problems are more severe because of the lower flavour threshold of many of the sulphur-containing by-products. Another source of sulphury off-flavours is the reduction of SO_4^{2-} to S^{2-} in the course of fermentation. Much of the resulting hydrogen sulphide is stripped off by the evolution of carbon dioxide during fermentation, and is therefore mostly a problem in the collection of food-grade carbon dioxide, but sufficient hydrogen sulphide and organic sulphur compounds could possibly remain in the wash to affect the quality of the distilled spirit.

Oxygen metabolism

Louis Pasteur is credited with discovering the switch between microbial fermentation and respiration by withdrawal or restoration of the air supply, although there is now some doubt if this Pasteur effect was actually his discovery. However, it is certainly true that facultatively anaerobic *bacteria* rapidly adjust from oxidative to fermentative metabolism according to the presence or absence of atmospheric oxygen. The situation with yeasts is more complex.

Firstly, Saccharomyces spp. and other fermentative yeasts cannot grow indefinitely under anaerobic conditions without certain nutrient supplements that are unnecessary aerobically. Sterols and unsaturated fatty acids, the essential cell membrane components of yeasts (and indeed of all eukaryotic organisms), cannot be synthesized anaerobically, thus limiting anaerobic yeast growth to two or three generations unless these compounds are provided in the culture medium. Secondly, fermentative yeasts are not true facultative anaerobes, since the Crabtree effect (Fiechter *et al.*, 1981; Young, 1996) takes precedence. Above about 1 per cent fermentable sugar in the culture medium (the exact percentage varying between strains) the yeast ferments the sugar by anaerobic metabolism, no matter how well the culture medium may be aerated. Instead, the dissolved oxygen is used for the biosynthesis of membrane fatty acids and sterols, thereby permitting more extensive growth when aerobic conditions are no longer available. Therefore,

although deliberate addition of unsaturated fatty acids and sterols to wort to encourage yeast growth is certainly not permitted for Scotch whisky fermentations, the same effect can be achieved legally by aeration of the wort.

In a paper partly reporting their own experience but also extensively reviewing the literature on the Pasteur and Crabtree effects, O'Connor-Cox *et al.* (1996) stated that of the dissolved oxygen present in the wort at the start of the brewery fermentations, only about 30 per cent was used directly for synthesis of unsaturated fatty acids and sterols. It has been well established that none is used for the oxidative metabolism of sugars (see, for example, Lagunas, 1986). In fact the remainder was used in restoring the degenerate mitochondria of anaerobically-grown yeast cells to the fully functional state required for lipid synthesis, and perhaps also the synthesis of other essential growth factors. Within a few hours the dissolved oxygen content of the wort had fallen to zero, and the mitochondria returned to the degenerate state associated with anaerobic conditions. However, claimed O'Connor-Cox *et al.* (1996), without a period of full mitochondrial function there could be no subsequent fermentation in the brewery. Extrapolating to Scotch whisky from these observations, malt distillery fermentations pitched partly with brewing yeast certainly require the stimulus of aeration, but a pure culture of aerobically-grown distilling yeast, which already has fully developed mitochondria, should if necessary be able to carry out a satisfactory fermentation without aeration. Even so, it is likely that fermentations would proceed differently with and without aeration.

Summary of flavour production during fermentation

In breweries alteration of wort composition has an important influence on beer flavour profile (Hough *et al.*, 1982; Young, 1996; Slaughter, 2002), but that option is not legally available to Scotch whisky distillers. Other fermentation variables well known to influence beer flavour are relevant to Scotch whisky fermentations, although the possibilities for deliberate manipulation of flavour are more restricted. The following factors affect the production of flavour congeners:

- Genetic properties of the yeast strain
- The condition of the yeast at pitching (viability and vitality)
- The amount of yeast inoculum
- Initial aeration of the wort
- The temperature profile during fermentation
- Microbial contamination.

A summary of the effects of fermentation conditions on production of flavour congeners appears in Table 4.4. Fermentation temperature has an important influence, and was studied extensively in the context of brewery fermentations

Table 4.4
Factors affecting ester and higher alcohol production by yeast

Cultural condition	Effect on higher alcohol production	Effect on ester production
Increased growth of yeast (higher temperature, increased O_2)	Increase (less available – NH_2)	Decrease (less recycled CoA)
Decreased growth of yeast (lower temperature, decreased O_2)	Decrease (more available – NH_2)	Decrease (more recycled CoA)
Deficiency of amino N	Increase (less available – NH_2)	Usually decrease if less growth, less recycled CoA)
Redox effects:		
(a) Deficiency of NAD+	Increase (to generate NAD)	Neutral, or increase if less growth
(b) Sufficiency of NAD+	Neutral or decrease	Neutral, or decrease if more growth
Genetic properties of yeast	Increase or decrease, according to properties of yeast strain	

in the 1960s and early 1970s. As a general rule, increasing fermentation temperature increases the amount of yeast growth in proportion to the available α-amino nitrogen, resulting in increased production of higher alcohols (Enari *et al.*, 1970). Conversely, since increased growth means less recycling of CoA, ester production is reduced. Although it is not distillery practice to maintain a constant temperature as in breweries, consistent levels of flavour congeners in the wash require consistent temperature profiles throughout successive fermentations, achieved by judging the correct initial temperature of the wort for the ambient temperature.

The genetic properties of the yeast also affect production of flavour congeners. No two strains have identical responses to fermentation temperature and the dissolved oxygen and amino nitrogen content of the wort. Therefore changing to a different culture yeast may affect the flavour of the wash and ultimately the spirit and whisky. Since an 'estery' aroma has become a desirable quality, Hay *et al*. (1994) developed a higher-yielding distilling yeast by genetic manipulation. However, with the present public antagonism to genetically manipulated organisms, it is unlikely that such a yeast will be used commercially in the near future to generate higher ester levels in whisky, even though it complies with the Scotch whisky regulations.

Cultivation of distillery yeast

Most of the yeast used in distilleries is purchased from manufacturers of baking and distilling yeast. The final production stage culture is in fermentors of up to 5×10^5 litres capacity, but the inoculum is propagated from the laboratory stock culture through a succession of fermentation vessels of twenty-fold increases in size. The culture medium of molasses, from either the beet or cane sugar industries, is supplemented with ammonium salts and any trace nutrients shown to be necessary by analysis of the current batch of bulk molasses (Burrows, 1970). All cultures are grown with accurate temperature control at 30°C and vigorous aeration, and often with mechanical agitation, although for unicellular yeasts the aeration alone should suffice for efficient mixing.

For efficient cultivation of both baking and distilling strains of yeast, it is essential to maintain a low concentration of fermentable sugar in the culture medium. To avoid the Crabtree effect and maintain energy-efficient aerobic conversion of sugars to carbon dioxide and water, the fermentor is filled with a medium of NH_4 salts and other trace nutrients and the sugar is added as sterilized concentrated molasses – slowly at first, but increasingly rapidly as the yeast biomass becomes greater throughout the fermentation. The aim is to provide a consistent level of about 0.5 per cent sugar. Ultimately the yeast concentration exceeds the ability of the aeration system of the culture vessel to maintain the necessary aerobic conditions for efficient growth, so as the dissolved oxygen concentration approaches zero; after about 30 hours' growth the propagation is stopped.

The yeast culture is harvested by a rotary vacuum filter of about 2 m in diameter (Figure 4.11). The circumference of the filter consists of a continuous strip of fabric coated with food-grade starch. As the drum rotates slowly through the culture in the trough at about one revolution per minute the spent culture medium is drawn under vacuum through the hollow spokes (only a few are shown in the figure); the yeast is retained on the filter to be scraped off and dropped to the packaging department on the floor below.

Until recently, yeast for the distilling industry was sold only in 25-kg bags of compressed moist yeast (24–30 per cent dry weight) or in bulk as 'cream yeast' slurry of about 18 per cent dry weight (R. C. Jones, 1998). Both must be stored at 3–4°C and used within three weeks to avoid significant deterioration. Cream yeast, which is delivered, stored and pitched in bulk, is more convenient for automated large-scale operations. Bags of compressed yeast must first be slurried aseptically in a sterilized yeast-mixing vessel to provide the required consistency for pitching.

Now, an increasing proportion of distillery yeast production is dried at 45–55°C in an atmosphere of inert gas, usually nitrogen, under partial vacuum. Dried yeast (92–96 per cent dry weight) has a shelf-life of up to two years and does not require cold storage, although in some distilleries the stock is chilled for added security. The reactivation of dried yeast at pitching must be carried out carefully to prevent loss of viability. Two methods are recommended
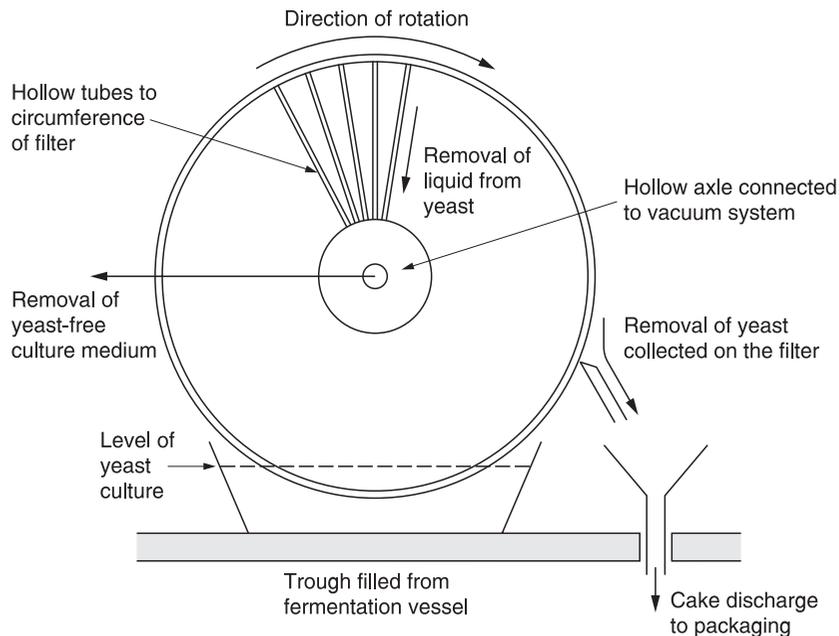
Figure 4.11
Rotary vacuum dryer for filtration of distillery yeast.

(MacDonald and Reid, 1998): adding dried yeast directly to the fermentation vessel, or pre-mixing to a slurry with water. For direct inoculation, the wort cooler is adjusted to produce wort at 38°C. The warm wort is collected to a depth of 10 cm in the washback and the required amount of dried yeast is sprinkled on to the wort, taking care to avoid clumping. After five minutes the heat exchanger is adjusted to the normal set temperature (about 20°C) and wort collection is completed. The disadvantage of this method is the requirement for two different but accurately controlled collection temperatures for the wort. For the pre-mixing method, sterile water or weak wort (ten times the volume of the dried yeast) is brought to 38°C in the rehydration tank and yeast is added with stirring to prevent clumping. After mixing for five minutes the yeast slurry is pumped into the washback at the normal set temperature. Therefore the pre-mixing method uses the same equipment as for the preparation of a slurry from bags of pressed yeast, but for dried yeast the higher temperature of mixing is critical.

Grain whisky distillers use only distilling yeast, but many prefer to mix cultures from different suppliers in a proportion that experience has shown to give the best results.

Many malt distillers believe that a mixture of brewer's and distiller's yeast gives a higher spirit yield than distiller's yeast alone, so they continue to use brewer's yeast at up to 50 per cent of the inoculum for the fermentation. Brewery yeast, recovered from an anaerobic alcoholic fermentation, cannot

be expected to meet the viability/vitality standards of aerobically grown distilling yeast, but it is reasonable to expect it consistently to meet a realistic specification of viability and vitality, and a low level of microbial contamination. Since the brewery must maintain yeast quality over successive fermentations for its own purposes (H. L. Jones, 1998), yeast from a reputable supplier should be in similarly good condition. Some breweries may not be quite so careful, so a strict quality specification is essential for purchase of brewer's yeast. There is a greater risk of bacterial contamination than in distilling yeast, so in some malt distilleries the brewing yeast is acid-washed before use to reduce or eliminate that possibility. Washing for up to one hour with chilled phosphoric acid (pH 2.8) is routine antibacterial practice in many breweries (Simpson and Hammond, 1989), but acid-washing is ineffective against 'wild' yeasts and many are more acid-tolerant than culture yeast. Certainly chilled storage is essential, partly to prevent further development of microbial contaminants, but also to prevent endogenous metabolism of the storage polysaccharides trehalose and glycogen and the resulting fall in viability and vitality (Maemura *et al.*, 1998).

Progress of fermentation

Most of the literature on brewery fermentations is equally applicable to Scotch whisky distilleries, provided allowance is made for certain process differences. The most important difference is the absence from the distillery operation of microbiological sterilization and inactivation of amylolytic enzymes resulting from brewery wort boiling. Therefore it is expected that more complete utilization of sugars and increased production of ethanol will reduce the specific gravity of wash below that of water, typically to 997–998°, whereas brewery fermentations are completed in the range 1005–1010°. However, some of the variables affecting flavour production in beer fermentations (e.g. manipulation of temperature, pressure and wort composition, including addition of yeast nutrients) are not permitted or may be technically impossible in Scotch whisky fermentations.

Design of fermentation vessels

Fermentation vessels (washbacks) in the distilling industry are now constructed of either wood (usually but not exclusively Oregon pine) or stainless steel. Wooden vessels with their rough internal surfaces are difficult to clean and impossible to sterilize, but are still common in malt whisky distilleries. Essentially the vessel is cylindrical, but it often tapers slightly inwards towards the top and it has a loose-fitting wooden cover. While traditional wooden vessels have a certain attraction for tourists, microbiological considerations have caused an increasing rate of replacement by stainless steel, which has become standard in grain whisky distilleries. In comparison

with fermentation vessels of similar size in the brewing and pharmaceutical industries the 250 000–500 000-l washbacks of grain distilleries are unsophisticated structures, but most allow for aeration at the start of fermentation, in-place cleaning, and perhaps steam sterilization and collection of carbon dioxide.

Yeast: quantity and quality

In order to follow the progress of fermentation it is necessary either to measure the quantity of yeast biomass or to count the number of cells. For pitching on a production scale, yeast is measured by mass – either directly, as the number of 25-kg bags of yeast required, or indirectly, as the volume of yeast slurry of standard concentration by weight. On a laboratory scale, counting the number of cells per millilitre is more convenient and is usually accomplished by microscope and an engraved counting chamber slide, also known as a haemocytometer after its originally designed use for counting blood cells.

The 'quality' of yeast is a more complex concept. For many years measurement of the percentage of viable cells in the culture by the simple methylene blue (MB) test was used without question (Baker, 1991). The method depends on the fact that MB is taken up only weakly, if at all, by living cells, and any taken up is rapidly converted to the colourless reduced form by the metabolic activity of the cells. On death cells become readily permeable to MB, and, since dead cells do not metabolize, remain blue. Other similar redox dyes have also been suggested from time to time, with claimed advantages over MB, but unfortunately all redox dyes are unreliable with yeasts of lower viability than about 90 per cent, which is often the case with yeast of brewery origin.

Fresh distiller's yeast should be close to 100 per cent viability, and should retain that value for several weeks of storage at 3–4°C. Storage at ambient temperature is unacceptable for two reasons: loss of viability of yeast, and the rapid growth of bacteria from the inevitable initial low level of contamination. The viability of brewer's yeast at the end of the brewery fermentation is unlikely to be over 95 per cent, and will fall even further during transport and storage. Even when chilled, yeast from an anaerobic fermentation does not have the storage stability of aerobically grown yeast (R. C. Jones, 1998). Dolan (1976) suggested 85 per cent viability as the lowest value for acceptance of brewer's yeast, but it is realistic to request 90 per cent on receipt. Yeast from beers brewed with traditional hop boiling is protected to some extent by the bactericidal effects of hop iso α-acids. However, yeast from breweries using pre-isomerized α-acids, which are added during post-fermentation processing, lacks that protection and therefore is potentially of poorer bacteriological quality (Hardy and Brown, 1989). Also, it is important to be aware of the lower viability of yeast from high-gravity brewing (Pratt-Marshall *et al.*, 2002).

If brewer's yeast is half of the culture, the average viability is unlikely to exceed 95 per cent; also, initial aeration of the wort will be required to ensure efficient growth. During growth the percentage viability approaches 100 per cent, since, obviously, only living cells grow, but the combination of alcohol

concentration, low pH and high temperature later in the fermentation causes a significant reduction in viability.

It is now recognized that measurement of percentage viability alone is insufficient to guarantee the quality of the pitching yeast. Hence the concept of yeast vitality, which concerns the ability of the yeast to ferment quickly and efficiently rather than simply being alive (similar to the concept of fitness in humans). Various methods have been suggested as indicators of vitality, but the most convenient method (i.e. rapid, and using simple equipment) measures the excretion of H^+ ions, by fall of pH, on transfer to a fermentable substrate (Kara *et al.*, 1988). A more accurate method on the same principle, measuring loss of Mg^{2+}, was described by Mochaba *et al.* (1997), but measurement of excreted Mg^{2+} is much more complex than measurement of H^+ by pH meter. Many other methods have been suggested for determination of vitality (Lentini, 1993), but these are even more labour-intensive. Heggart *et al.* (1999, 2000) reviewed factors affecting yeast vitality and viability, as well as methods of measuring them.

Kinetics of yeast growth

Wort, whether derived from malted or unmalted cereal, represents a rich source of carbon, nitrogen and mineral nutrients for the yeast. Its principal deficiency is in the specific lipid requirements for growth of cell membranes – the unsaturated fatty acids and sterols discussed previously. The growth of yeast in a distillery fermentation follows the graph shown in Figure 4.12. Note
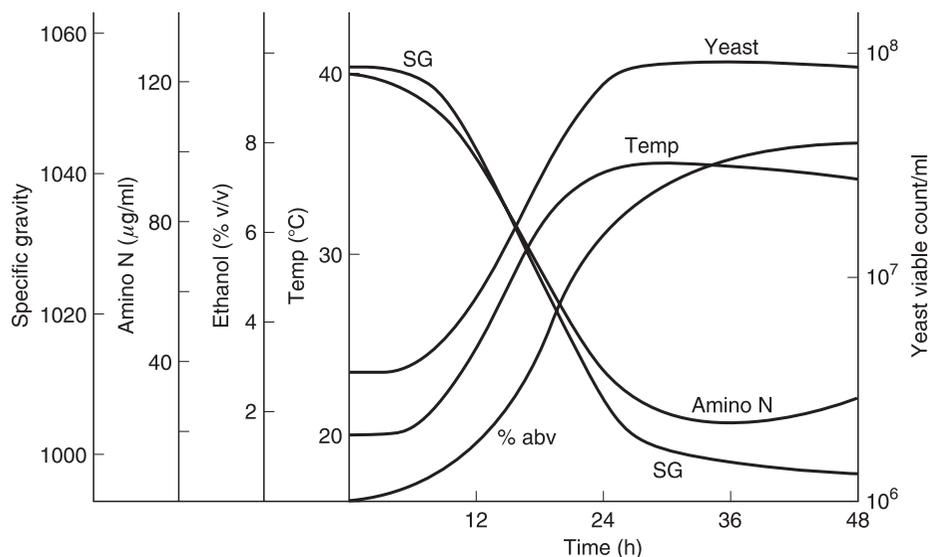

Figure 4.12
Progress of distillery fermentation.

that although active yeast growth ceases at about 24 hours, fermentation of sugars continues (although at a slower rate); therefore there is a continuing increase in alcohol content and a corresponding fall in specific gravity. However, with the end of active yeast growth there is no further uptake of α-amino nitrogen from the wort or increase in temperature. In fact, the amount of α-amino nitrogen normally increases in the wash in the later stages of fermentation due to autolysis of a proportion of the yeast, and the temperature may fall slightly without continuing yeast metabolism.

Metabolic activity generates heat. In a brewery, fermentation temperature is carefully controlled to within 0.5°C of the selected temperature by attemperated panels in the walls of the fermentation vessels. In most distilleries there is no attempt at temperature control other than adjusting the temperature of the wort entering the washback according to ambient temperature. In cold weather wort could be adjusted to, say, 22°C, but in warmer weather to 19°C, so that the natural increase in temperature would result in both fermentations reaching 33–34°C. Other reasons for reducing the setting temperature are a higher than normal original gravity of the wort (within a range that could be expected to accelerate the fermentation) or a lower ratio of brewing : distilling yeast. With stainless steel washbacks a simple temperature control may be used: in some distilleries by spraying the outside of the vessels with cold water, in other cases the fermenting wash may be circulated through an external cooler. In theory the latter system could also be used with wooden vessels, but it is not known whether this has been tried. Commercial distilling yeast strains ferment well at up to about 34–35°C, but at higher temperatures metabolic activity rapidly declines. Yeast purchased from ale breweries is also capable of growth at up to 33–34°C, but this is too high for lager yeast, which typically has a maximum growth temperature of 28–30°C (Walsh and Martin, 1978). Therefore if lager yeast is used as part of the inoculum it assists the distilling yeast only in the early part of the fermentation, but of course the structural components of the yeast are still available to contribute to congener development during distillation.

So the special conditions of a distillery fermentation result in an important difference from the textbook version of the microbial growth curve. While the growth of micro-organisms at constant temperature can be expressed as a straight line by plotting cell numbers logarithmically against actual time (hence the term log phase), that is not true in Scotch whisky fermentations. Heat generated by yeast metabolism causes the temperature to rise throughout most of the log phase, and growth rate increases accordingly.

Another important difference from the superficially similar brewery fermentation is the lack of heat treatment of the wort corresponding to the hop-boiling stage of brewing, which inactivates malt enzymes. Malt distillery mashing uses a succession of increasing temperatures (see Chapter 2), but the 'first water' is traditionally at about 64°C, the precise temperature varying according to distillery. At the end of mashing at that temperature Walker *et al*. (2001) measured (in that first batch of wort cooled to 20°C and collected in the washback)? α- and β-amylase levels at about 80 per cent of those in the original malt, and free limit-dextrinase was slightly increased (Figure 4.13).
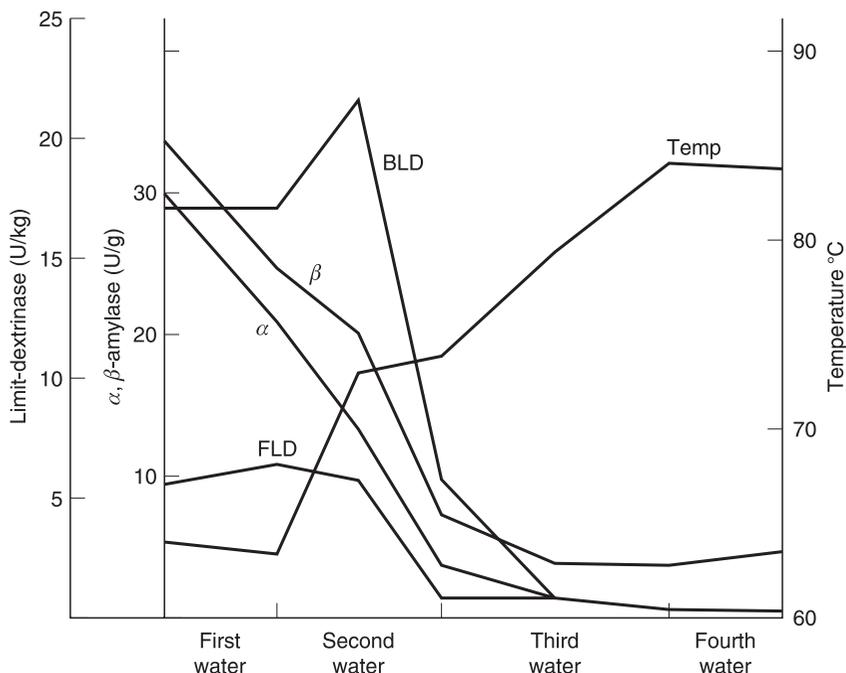
Figure 4.13
Activity of amylolytic enzymes during distillery mashing (Walker *et al.*, 2001). α, α-amylase, β, β-amylase, BLD, bound limit-dextrinase, FLD, free limit-dextrinase.

Appreciable activity of the amylases and bound limit-dextrinase remained even after mashing with the 'second water' of higher temperature. Since grain distillery mashing is at a fixed temperature of about 63–64°C, again appreciable hydrolytic activity persists to fermentation. Therefore in both grain and malt distilleries a high proportion of the cereal starch is converted to fermentable sugars and then to alcohol by continuing hydrolysis during fermentation.

Since the amount of pitching yeast for distillery fermentations is normally measured as cell dry weight, it is important to know the water content of the compressed, cream or dried yeast supplied. Experience has shown that a minimum of 18 kg dry weight of *distilling* yeast is required per tonne of malt in malt distilleries, with equivalent addition in grain distilleries. Lower pitching rates reduce spirit yield, probably because there is more yeast growth and therefore more sugar is converted to cell mass rather than ethanol. Also the pitching rate must be increased slightly for mixtures of brewing and distilling yeast, to at least 22 kg/t with 50 per cent of brewery yeast. In terms of cell count, 1.8 per cent of the weight of malt corresponds to $3–4 \times 10^7$ cells/ml, depending on the original gravity of the wort. Alternatively, the pitching rate can be expressed as 5 g pressed yeast per litre of wort (Bathgate, 1989).

For about six hours after inoculation the cell count remains constant, hence the term lag phase – a relic of the earlier belief that little activity took place. In reality the lag phase is a period of intense biochemical activity as the yeast cells adjust from their previous conditions of culture to the conditions in fresh wort. The change is particularly severe for brewing yeasts, transferred from an anaerobic alcoholic low-nutrient environment at the end of fermentation, via a period of transport and storage under moderately inhibitory conditions, to the nutrient-rich aerobic alcohol-free wort. It is possible that some yeast growth does take place during the lag phase, but is balanced by the death of a proportion of cells by the osmotic shock of transfer to the high sugar concentration of fresh wort so the cell count remains approximately constant.

By six hours after pitching, and perhaps earlier if only distilling yeast is used, growth begins in the sense of increasing cell numbers – the stage variously known as growth, logarithmic or log phase. In a brewery, only about eight- to ten-fold multiplication is possible, i.e. three cell generations, and possibly a fourth generation by a small proportion of cells. Further growth is impossible because of the lack of the essential membrane components unsaturated fatty acids and sterols, which cannot be synthesized under the anaerobic conditions of fermentation. Distillery yeast, unlike brewery yeast recovered from the previous fermentation, was originally grown under vigorous aerobic conditions, generating an excess of these essential lipids, and if added to well-aerated wort could grow up to twenty-fold (i.e. slightly over four generations). With the typical temperature profile of a distillery fermentation the log phase would be expected to last for 18–24 hours, but ultimately there is no further increase in yeast population and the culture enters the stationary phase (i.e. stationary with respect to the number of viable yeast cells). The lack of membrane lipids is an important factor ending yeast growth in a distillery fermentation, but metabolic activity continues for a further 12–24 hours, although at a slower rate. There may be some slight continuing growth of the yeast, but this is balanced by death and autolysis of a proportion of the cells so the viable population remains constant. It is reasonable to assume that the theoretical assessment by Werner-Washburne *et al*. (1993) is applicable to the stationary phase of Scotch whisky fermentations. Ultimately, yeast growth ceases and the increasing rates of cell death and autolysis initiate the decline phase, but this is happens only near the end of the 48-hour timescale of the majority of distillery fermentations.

A figure of 0.131 (or 0.1315) is in common use for estimating the alcohol yield of a distillery fermentation – for example, wort of original gravity 1057° fermented to 997° contains $(1057 - 997) \times 0.131 = 7.86$ per cent alcohol by volume. This conversion factor is higher than its equivalent value 0.129 in the brewing industry to take account of the more efficient fermentation of unboiled wort.

During fermentation hydrogen ions are excreted by the yeast, resulting in a fall in pH. Figure 4.14 shows the typical trend in fermentation of an all-malt wort by a pure culture of distiller's yeast, initially pH 5.4, falling to a lowest value of 4.0–4.1 (Dolan, 1976) and increasing slightly during the stationary phase by the release of amino acids from autolysing yeast cells. Perhaps auto-
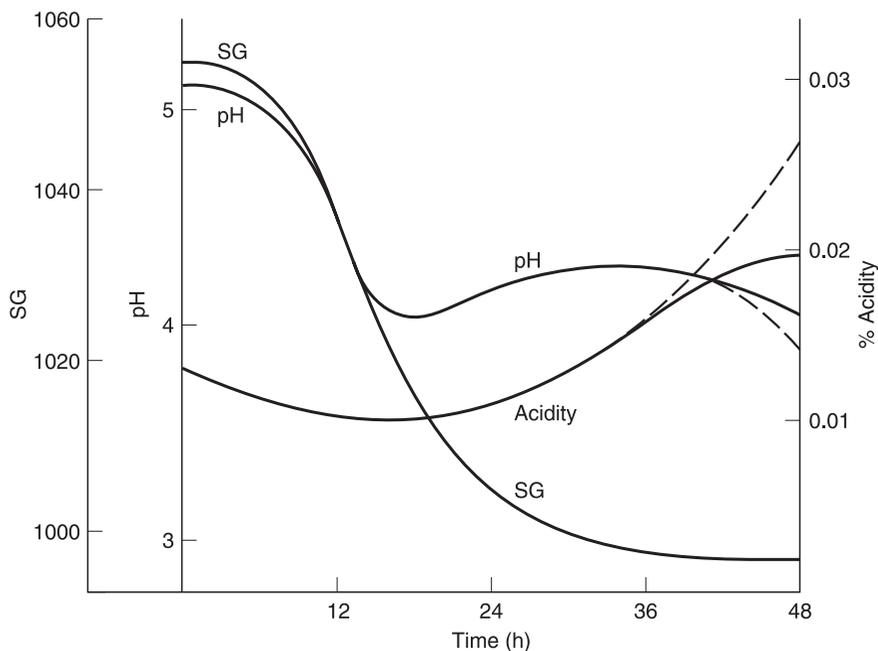
Figure 4.14
Variation in pH during a malt distillery fermentation (modified from Dolan, 1976).
Broken lines after 36 hours show the effect of contamination by lactic bacteria; solid
lines show the normal progress of fermentation. The initial pH in a grain distillery is
higher than shown here. Wheat or barley typically give pH 5.5–5.7 and maize pH
5.7–6.0 at the start of fermentation, but otherwise the graphs are equally applicable
to grain distillery fermentations.

lysis of part of the batch of brewer's yeast accounts for the slightly but
consistently higher pH values throughout mixed-yeast fermentations.
However, in the case of fermentations showing late growth of lactic bacteria,
which use yeast autolysate as nutrient, the pH can fall again to about 3.8.
Growth of lactic bacteria during the stationary phase, on residual dextrins
and pentoses that are non-fermentable by the yeast, and trehalose and nitro-
genous products of yeast autolysis, is generally considered to make a valu-
able contribution to the range of flavour congeners in the wash and
ultimately in the final whisky (Geddes, 1985; Barbour and Priest, 1988). On
the other hand, contamination early in the fermentation is undesirable,
reducing the spirit yield by loss of fermentable sugars to the metabolism
of lactic bacteria (Dolan, 1976).

   The changes in pH affect the activity of α- and β-amylases and limit-dex-
trinase, and therefore the hydrolysis of polysaccharide during fermentation.
Starch and dextrins remaining after mashing are hydrolysed most rapidly in
the lag phase, but as fermentation begins and pH falls the hydrolytic activity
of malt enzymes declines. Although Watson (1983) quoted inactivation of

α-amylase at about pH 4.9, many distillers believe that amylolytic activity continues (at a decreasing rate) to lower pH values, ultimately ceasing at about pH 4.4. Under-modified malts are especially dependent on continuing conversion during fermentation, so the higher pH (by approximately 0.2 units) throughout fermentations that include brewer's yeast is advantageous for amylolysis. However, in addition to the competition for carbohydrate referred to above, the lower pH associated with heavy early contamination by lactic bacteria may also reduce spirit yield.

Vigorous fermentation also causes foaming, which is normally controlled by a rotating 'switcher' blade in the head space of the washback. Some brewing yeast strains are more liable than others to cause froth or head during a brewery fermentation and, despite the moderating effect of the large proportion of distilling yeast, they inevitably maintain that characteristic in a washback. It has also been suspected that some barley varieties are associated with unusually frothy fermentations (and wash distillations). Although wheat has become the predominant cereal for grain distilleries in recent years, one of the advantages of maize is its high oil content, which has a valuable natural antifoam effect during fermentation. However, since additives are not permitted by the Scotch Whisky Regulations, the deliberate addition of antifoam to a distillery fermentation is not possible.

Brewers rely on flocculence of yeast to achieve a partial clarification of beer at the end of fermentation. Flocculation, the spontaneous aggregation of cells into clumps, which settle more readily by their increased mass (Stratford, 1992), is unacceptable in distilling yeast. Flocculated yeast cells settling on heating surfaces of the wash still will char, causing flavour defects in the spirit and restricting heat transfer, so it is essential to maintain a uniform yeast suspension throughout fermentation and wash (or continuous) distillation. Although lager yeast is either non- or poorly flocculent and therefore not a problem in this regard, distillers purchasing ale yeasts must beware of strongly flocculent strains.

In this section the duration of fermentation has been assumed to be 40–48 hours, which is certainly true of grain distilleries. However, even if a malt whisky fermentation appears complete by 40 hours, Geddes (1985) advised against distillation of the wash before 48 hours to allow development of flavour by lactic bacteria. Also, 48-hour fermentations are convenient for round-the-clock operation of a small traditional malt whisky distillery with mashing at eight-hour intervals. However, a few malt distilleries operate longer fermentations to encourage late lactic contamination of the fermentation, but therefore require more fermentation capacity to maintain energy-efficient rapid re-use of the wash still.

Production of carbon dioxide

Carbon dioxide and ethanol have similar molecular weights, and therefore are produced in approximately equal amounts during fermentation. Although carbon dioxide is not collected in malt distilleries on the same large scale as

in grain whisky distilleries, there may be some commercial value in collecting the gas as a co-product.

Although carbon dioxide evolution begins towards the end of the lag phase, the first of the gas is contaminated by the head-space air that it flushes from the washback. The most rapid evolution occurs during the log phase, subsequently subsiding during the stationary phase to a rate where it is uneconomical to collect it. Therefore although approximately half of the weight of sugar fermented is released as carbon dioxide, only about half of that amount is actually available for collection between about the tenth and thirtieth hours of fermentation. Figure 4.15 shows the general trend of production (measured in kg/h) during fermentation, but because of wide variation between distilleries actual values are not quoted.
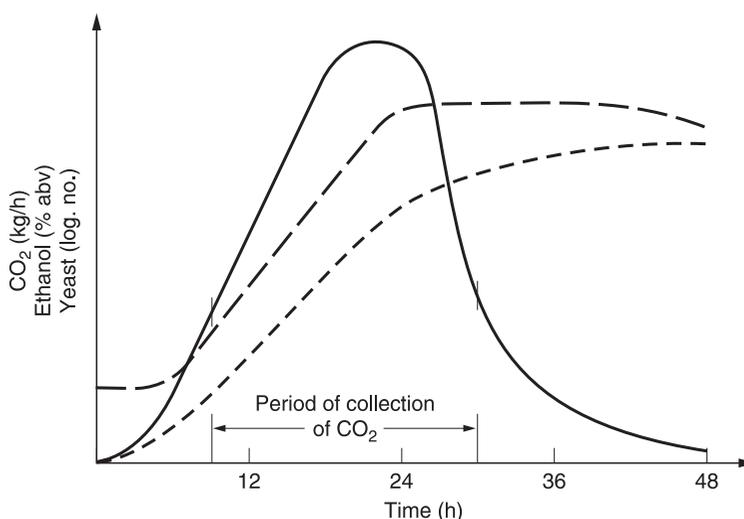

Figure 4.15
Evolution of CO_2 during fermentation. Solid line, CO_2; broken line, ethanol; circle/broken line, yeast log no.

Contamination

Possible contaminants of distillery fermentations

Microbial contamination of the fermentation is potentially another source of desirable flavours in the final whisky, although the effect could equally well be an off-flavour. The principal sources of the unavoidable contaminants of distillery fermentations (Table 4.5) are the microbial flora of the malt (acetic and lactic bacteria and aerobic yeasts), residual contamination of wooden washbacks (potentially by all organisms on the list) and the yeast itself (different in brewery or distillery yeast, see later).

Table 4.5

Possible microbial contaminants of distillery fermentations

	Aerobic yeasts	Fermentative yeasts	Lactic bacteria	Acetic bacteria	*Zymo-monas*	Entero-bacteria
Brewery yeast	+	+	+	+	+	+
Distillery yeast	+	−	−	+	−	+
Wort, early fermentation	+	+	+	+	−	+
Late fermentation, wash	−	+	+	−	+	−

Effect of contamination: approximate loss of spirit yield in a malt distillery due to growth of *Lactobacillus* from the start of fermentation (Dolan, 1976; Barbour and Priest, 1988):

Up to 10^6/ml   < 1% loss
1–10 10^6/ml   1–3% loss
1–10 × 10^7/ml   3–5% loss
Above 10^8/ml   > 5% loss

There is no loss of spirit yield when Lactillobacillus grow only at the end of fermentation, on nutrients from autolysed yeast.

Since the wort is not boiled, some microbial contamination of the wash is inevitable. The initial temperature of mashing in a malt whisky distillery is insufficient to sterilize the wort, although a temperature of 63–65°C will certainly cause a substantial reduction in the number of potential spoilage organisms (Geddes, 1985). In grain distilleries the cereal is sterilized by pressure cooking, but since the malt slurry is heated only to about 63°C, and then briefly, malt is a potential source of contamination, especially since with little kilning (or none at all) the microbial flora developing during germination is not destroyed. However, at approximately 10 per cent of the cereal raw material, the potentially contaminating effect of malt is proportionally less than in a malt distillery.

Wort itself has little anti-microbial effect other than the osmotic stress of the sugar concentration, but once fermentation is under way the wash is protected by the synergistic combination of various anti-microbial effects: the low pH, anaerobic conditions, dissolved carbon dioxide and the exhaustion of fermentable sugars by the end of fermentation. These factors were identified by Hammond *et al*. (1999) as protective effects in breweries, but are equally applicable to distillery fermentations. For example, Dolan (1976) encountered unidentified Gram-positive cocci in wort, which quickly died off at the start of fermentation. Only a limited range of spoilage microorganisms produce unacceptable flavour congeners that could distil into the new spirit, but it is also possible that a serious level of contamination, competing with the culture yeast for fermentable sugar, could reduce spirit yield (Dolan, 1979).

Aerobic yeasts grow well in the culture conditions of the yeast factory, where even a low level of accidental contamination can rise to significant

numbers in the final product. Fortunately such contaminants can grow only in the first few hours of the distillery fermentation (Campbell, 1996b), but some, especially Pichia spp., produce in that time sufficiently high levels of esters to affect the congener profile of the wash and, more probably in malt distilleries, of the spirit. Fermentative yeast contaminants are more likely to be associated with brewery yeast. Although they can be a serious problem in breweries, with their similar metabolism to *S. cerevisiae* they are unlikely to cause flavour problems in a distillery. The bacteria producing acetic and lactic acids may be present on malt, and Geddes (1985) reported introduction of both into the fermentation from that source. Since acetic bacteria are strict aerobes they are less of a problem than the lactic bacteria, especially lactobacillus, which can grow throughout the fermentation. Although written primarily for brewery microbiologists, the review by Priest (1996) also includes distillery lactobacilli. Enterobacteria are unlikely contaminants, but may be introduced if contaminated water is used for rinsing sterilized equipment. However, that group also includes Obesumbacterium, which is a possible contaminant of brewery yeast. Fortunately these bacteria are quickly inactivated by the falling pH and increasing alcohol content during fermentation. They are a more serious problem in breweries, since they increase over successive fermentations with re-use of contaminated yeast, but that is irrelevant to Scotch whisky distilleries: yeast is never re-pitched to a following fermentation. The Gram-negative bacteria that disappeared within ten hours of pitching the fermentations studied by Dolan (1976) could well have been either acetic bacteria or enterobacteria.

Manufacturers of distillery yeast do not guarantee a pure culture (R. C. Jones, 1998), but in practice the likely level of contamination is low. Brewery yeast, on the other hand, is a potential source of both fermentative yeasts and bacteria that are able to grow sufficiently during distillery fermentations to produce detectable amounts of flavour congeners. If obtained from an unreliable source, brewery yeast could be contaminated by lactobacilli, pediococci and *Zymomonas*, all of which produce congeners that distil with the spirit fraction and compete with the culture yeast for fermentable sugars to reduce the spirit yield (Dolan, 1976, 1979). Inhibition of yeast growth by some strains of acetic and lactic bacteria (Thomas *et al*., 2001) is also a possibility, although in practice the developing anaerobic conditions and excess of culture yeast suppress the antibiotic effect of acetic bacteria. In an interesting incidental observation Barbour and Priest (1988) noted that different strains of lactobacilli varied in their effects: even if the amount of their growth was approximately equal, some strains caused a substantial loss of spirit yield while in other contaminated fermentations different strains had no such effect.

Ineffective sterilization of washbacks and associated equipment, or contaminated rinse water, are other possible sources of contamination, but are largely avoidable with correct procedures. Stainless steel washbacks cleaned after each fermentation and sterilized either by steam or by the final stage of a CIP system are less likely to be a source of contamination than wooden washbacks, which are almost impossible to sterilize (Dolan, 1976). However, even though some microbial contaminants in the cracks will survive any practical

duration of steaming, cleaning and steaming between fermentations prevent the inevitable low level of contamination building up to dangerous levels.

Table 4.5 shows the possible types of contaminant. For different reasons, aerobic (oxidative) yeasts, acetic bacteria and enterobacteria are able to grow only in the early stages of fermentation. The development of anaerobic conditions inhibits the first two groups. Enterobacteria, although unaffected by anaerobiosis, are inhibited by the falling pH and increasing alcohol content. Although lactobacillus contaminants are the most likely bacteria from malt, Pediococcus spp. are also possible, and some strains produce an extracellular polysaccharide that causes ropiness in beer or wash (Priest, 1996) and can be genuinely troublesome. Fermentative yeast contaminants, like brewery and distillery culture yeasts, form ethanol and carbon dioxide and, usually, the same flavour metabolites, although these congeners are almost certainly be produced in different amounts. Although the resulting off-flavour is an important nuisance effect in breweries, distillation normally eliminates this problem. Even if a particular batch of low wines has a higher than normal level of flavour congeners, and some wild yeasts of the genus Pichia are notorious for high ester production, that effect can be diluted by mixing with feints derived from an earlier, uncontaminated fermentation. Provided there is efficient sterilization of fermentation equipment, distilleries are less likely to suffer the continuing contamination of successive fermentations caused by re-pitching of culture yeasts in breweries.

In summary, the most likely and potentially most troublesome microbial contaminants of malt distillery fermentations are lactic acid bacteria. It is reasonable to assume from the data of Dolan (1976), without the delay of a microbial culture, that if gravities are higher and pH values lower than normal, the cause is a dangerous level of lactic bacteria. Another possible off-flavour of microbial origin is butyric acid, but the most likely source is the growth of anaerobic Clostridium spp. in malt or grain residues in the mash tun. Therefore, although the flavour of an infected mash could persist through fermentation to the final spirit, Clostridia are not strictly contaminants of fermentations.

Cleaning and disinfection

It is true that various micro-organisms of malt can survive the brief exposure to mashing temperature, but it is good practice to reduce as much as possible the other, avoidable, sources of contamination. While mashing, cooking and distillation equipment should certainly be cleaned to prevent development of moulds or butyric acid bacteria between periods of use, the equipment listed in Table 4.6 requires both cleaning and sterilization before each use. Although written with the brewing industry in mind, the review by Singh and Fisher (1996) on cleaning and disinfection is equally applicable to the fermentation vessels and associated pipework of the distilling industry.

Table 4.6

Sterilization requirements in distilleries

Process	Equipment	Type of soil	Method of cleaning
Wort cooling	Heat exchanger	Protein, scale	CIP (high-velocity detergent/sterilant)
Fermentation	Washback	Yeast, protein, sugar, scale, 'beerstone'	CIP
Yeast preparation	Mixing vessel	Yeast, protein	Manual or CIP
Wort transfer	Fixed pipeline	Protein, sugar	CIP (high-velocity detergent/sterilant)

The rough surface of the wood of fermentation vessels, joints between the planks and right-angled corners at the base encourage accumulation of organic soil in general and microbial contamination. Although steam is a useful sterilant, a jet of steam has no cleaning effect. In fact, by forming an insulating skin on the surface of organic soiling, steam may actually protect underlying micro-organisms. A powerful jet of cold water or, even more effectively, hot water with added detergent, provides the necessary scouring effect to remove such deposits (Dolan, 1976; Singh and Fisher, 1996). Subsequently, steaming will kill most surviving micro-organisms. However, washbacks and other equipment need an appreciable time of steaming to heat up to an effective temperature, and micro-organisms are not killed instantaneously by heat – even by steam at 100°C. Therefore at least 30 minutes' steaming of the washback is required after cleaning, and preferably longer if possible.

Cleaning and sterilization of the smooth inner surfaces of stainless steel vessels is more reliable, and now in many distilleries such vessels are equipped with automatic cleaning-in-place systems, but the choice of sterilization by steam or lower-temperature chemical sterilants requires some consideration. Although naturally some details are different, and sterilization is unnecessary, a similar choice applies to cast iron versus stainless steel mash tuns. The rough surface of cast iron is difficult to clean, but residual grain debris encourages the growth of the anaerobic bacteria that cause butyric acid off-flavour. Table 4.7 lists important considerations for both steel and wooden washbacks, but ultimately the decision is based on specific local factors.

Finally, at this point of transition between fermentation and the distillation operations described in Chapter 5 and 6, it is important to prevent contamination of the wash charger vessel and associated pipework to the still. Although partially protected by the factors discussed above, microbial growth there is possible, and regular (if not necessarily daily) sterilization is advisable.

Table 4.7
Comparison of steam and chemical sterilants for washbacks

Method	Advantages	Disadvantages
Stainless steel:		
Steam	Already available (for stills, etc.), no residue	Radiated heat affects nearby washbacks
		Metal fatigue from repeated expansion and contraction
		Cannot remove soil from surfaces
Chemical	Suitable preparations remove organic and inorganic soil	Requires subsequent rinsing with sterile water
		Can be applied at ambient temperature (although 50–60°C is preferable)
Wood:		
Steam	Available, no residue, as above	Cannot remove soil from surfaces
		Probably unable to sterilize within crevices or between planks
		May eventually damage structure of wood
Chemical	Removal of soil at moderate temperature, as above	Complete removal of sterilant is impossible (the necessary rinsing would be impracticable)

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<div style="text-align: right">Chapter *5*</div>

Batch distillation

<div style="text-align: right">*Denis Arthur Nicol*</div>

Introduction

Unlike grain whisky production, malt whisky production is essentially a double batch-distillation process using pure malt, water and yeast as the raw materials. In size, the production capacity of a malt distillery is a tenth the capacity of a typical grain distillery. This chapter discusses the history of batch distilling; the design and construction of stills and ancillary equipment; the operation of wash and spirit stills; product quality; efficiency (production yield); and triple distillation. It goes on to consider how to deal with distillation problems, describes laboratory techniques employed in batch distillation, and finally looks to the future.

History

Distillation was carried out from earliest times using pot stills, initially made of ceramics or glass and eventually of copper (Nicol, 1997). These early stills were fired by open fires in a furnace or hearth or, if the charge to be distilled was heat sensitive, by placing the still in a water or sand bath to reduce the action of direct fire. The distillates were originally condensed using air condensers, which resembled tapering lyne arms, to deliver the product to a glass or clay vessel. The evolution of the worm tub and eventually the shell and tube condenser revolutionized the cooling of distillates.

Alcohol was not recovered by distillation in any quantity until the twelfth century, when stills of crude design caulked with clay and straw were improved upon by using a close-fitting pot, head and lye pipe so that the recovery of alcohol was facilitated by the distillation of inferior, unpalatable beers and wines without the alcohol escaping through ill-fitting connections.

The production of alcohol was the preserve of monks in monasteries, within whose cloisters alchemy was practised in the fruitless search for the Philosopher's Stone, which was thought to be central to the transmutation of base metals into gold. The Elixir of Life was also sought, to no avail; alcohol filled this niche and apparently was prescribed as a panacea for all ills.

The Reformation saw the dissolution of the monasteries in England and Scotland. With the dispersion of the monks their knowledge as brewers, distillers, barber-surgeons, alchemists and apothecaries was released into the secular, material world. The monks had become the founders of the early medical sciences, brewing and distilling industries. Thus whisky (*uisge baugh*), first witnessed by Henry II of England (whose army invaded Ireland in 1170) as being produced from cereals by the natives of that land, had mention in the Scottish Exchequer Rolls of 1494 as *aqua vitae* or 'water of life', alluding to that tantalizing and unattainable Elixir of Life.

Design of distillery

All malt whiskies derived from low-nitrogen malted barleys are produced in copper pot stills (Nicol, 1989) of time-honoured traditional design. (Figure 5.1) The following principal elements comprise a pot still, whether for distilling wash or low wines:

- Heating source – direct fire (coal or liquid petroleum gas), or indirect fire (oil, steam coils, kettles or pans)
- Pot – contains charge to be distilled
- Shoulder
- Swan neck
- Head
- Lyne arm, lye pipe or vapour pipe
- Worm tub or condenser
- Tail pipe
- Spirit safe.

Figure 5.2 shows the overall layout of a typical malt distillery.

Heating source

As noted above, there are several fuels available for the distiller to heat the stills (Watson, 1989). With direct-fired stills, the pot must be designed to withstand the rigours of the direct firing and the copper crown and flue plates must be made of a sufficient gauge of copper to withstand the intense local heating. Where the copper is not exposed to such intense heat, its gauge can be less. The base of the direct-fired still is convex, resembling an inverted saucer. The hearth or furnace upon which the still rests is of brick or steel
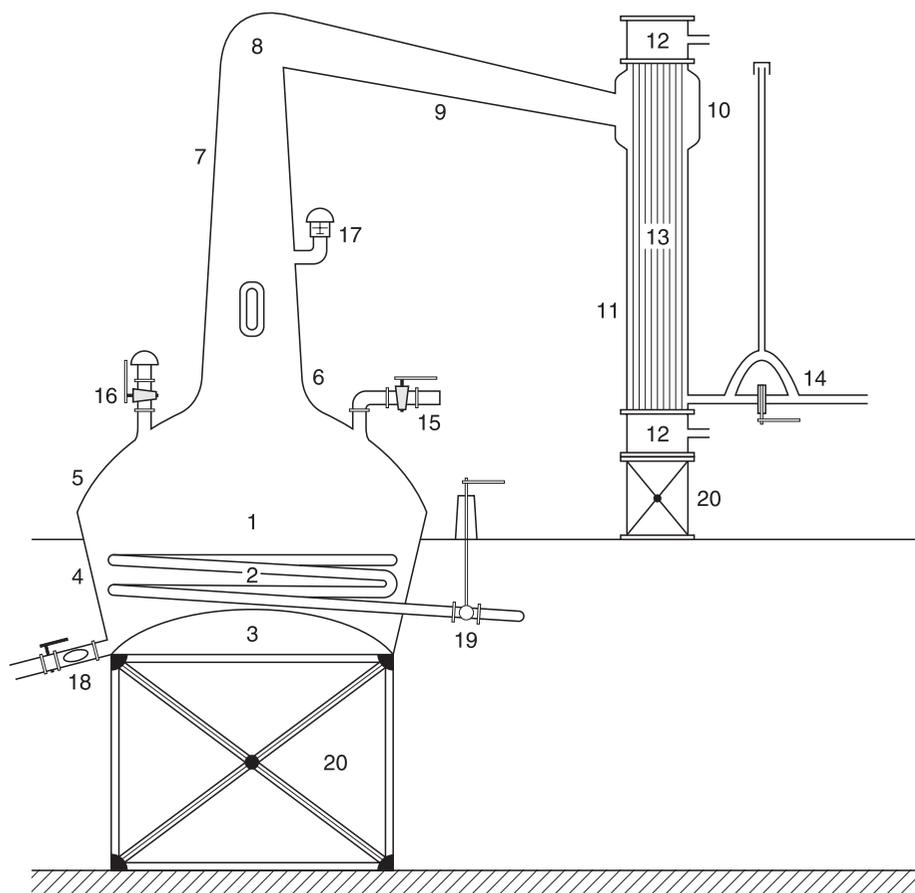
Legend

1. Pot	11. Shell and tube condenser
2. Steam heating coil	12. Water jacket
3. Crown	13. Tube bundle
4. Flue plate	14. Tail pipe with syphon
5. Shoulder	15. Charging line/valve
6. Ogee	16. Air valve
7. Swan neck	17. Anti – collapse valve
8. Head	18. Discharge line/valve/sight glass
9. Lyne arm/lye pipe	19. Steam line/valve
10. Vapour chamber	20. Cradle

Figure 5.1
Pot still design. 1, pot; 2, steam heating coil; 3, crown; 4, flue plate; 5, shoulder; 6, ogee; 7, swan neck; 8, head; 9, lyne arm/lye pipe; 10, vapour chamber; 11, shell and tube condenser; 12, water jacket; 13, tube bundle; 14, tail pipe with siphon; 15, charging line/valve; 16, air valve; 17, anti-collapse valve; 18, discharge line/valve/sight glass; 19, steam line/valve; 20, cradle.
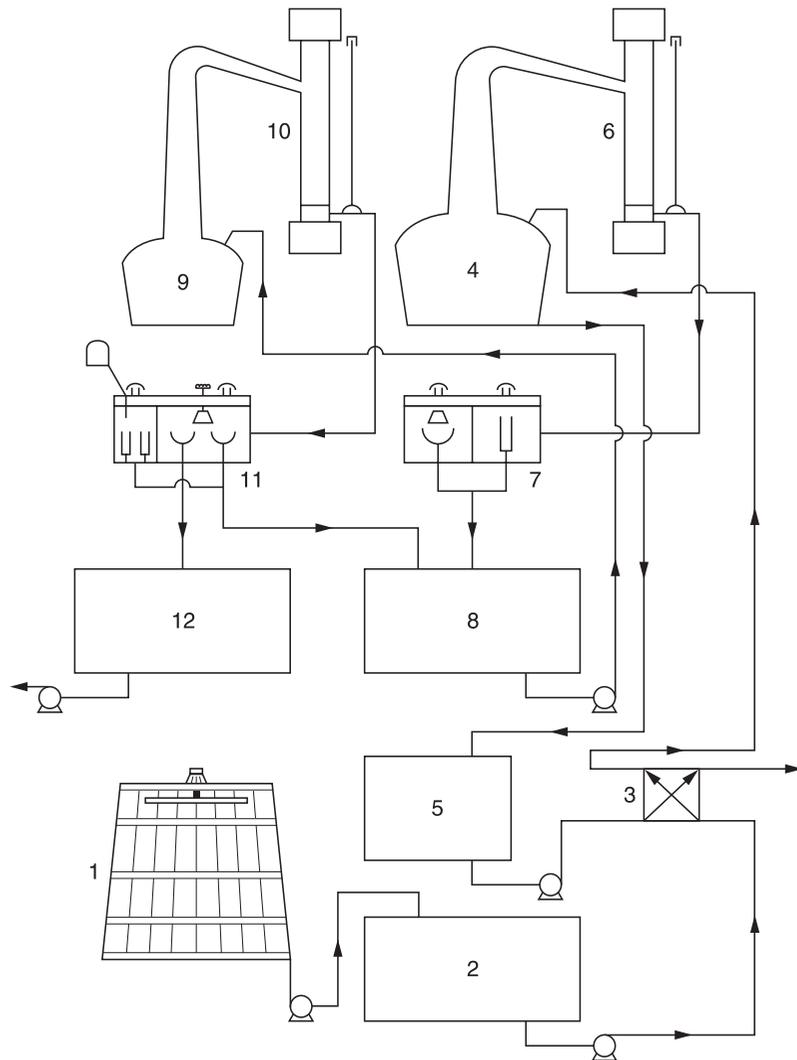
Legend

1. Washback	7. Wash still safe
2. Wash charger	8. Low wines receiver/charger
3. Wash preheater	9. Low wines/spirit still
4. Wash still	10. Low wines/spirit still condenser
5. Wash still condenser	11. Spirit still safe
6. Wash condenser	12. Intermediate spirit receiver (ISR)

Figure 5.2
Typical malt distillery layout. 1, washback; 2, wash charger; 3, wash preheater; 4, wash still; 5, wash still condenser; 6, wash condenser; 7, wash still safe; 8, low wines receiver/ charger; 9, low wines/spirit still; 10, low wines/spirit still condenser; 11, spirit still safe; 12, intermediate spirit receiver (ISR).

construction, lined with suitable firebricks to protect the supporting structure from the heat.

Whether gas- or coal-fired, the exhaust gases must be ducted to a flue stack or chimney made of brick or steel. With more than one still the flue gas can be led into a manifold, the flue gas flow being individually controlled by dampers. Where coal is used as the heating medium, each hearth is equipped with a chain grate stoker with automatic solid-fuel feed and ash removal. A damper is fitted to the flue to control the heat input. With gas firing, the burner can be modulated by controlling the gas flow.

For indirect heating by coil, pan or kettle, steam is supplied by an oil- or gas-fired boiler and is transferred by a steam main from the central boiler at the crown pressure; this pressure is reduced to the desired operating pressure for the internal heating elements. The heating elements must be designed to be totally immersed in the relevant charge to be distilled, at the beginning and end of the distillation cycle. A low wines and spirit still can be fitted with an extra coil that can be used distil the middle cut gently, ensuring good reflux. Steam traps are strategically positioned to remove any condensate that may waterlog the steam lines; condensate returns, post-heating, are fed to the boiler-feed water tank as an energy-saving step via a condensate manifold. The steam demand is calculated to provide sufficient energy to bring a still into the safe within an hour or less. The calculation should include the maximum demand for steam when heating hot liquor tanks and distilling several stills simultaneously. The boiler output is then designed on this demand and sized accordingly, with extra capacity included.

Direct firing of wash stills requires a rummager, a geared shaft from which a flail made of copper or brass is suspended. This rotates, scouring the flue plate and base of the wash still and preventing the build-up of charred material that would otherwise adversely affect the heat transfer.

To conserve energy, the external flue plates of an indirectly fired still can be insulated to prevent radiant heat losses through the sides of the pot. The shoulder and all other parts of the still should not be insulated as this would adversely affect the reflux, which is essential for spirit character – especially in the spirit still.

Wash preheating, when used, can considerably shorten distillation times and thus save energy. A treatise on energy-saving techniques can be found in *Whisky Distilling* (1985).**20

Pot

The pot (Figure 5.3) can assume many shapes (Whitby, 1992) – conical, onion, cylindro-conical, inverted cone, ball and lantern – provided that sufficient volume and surface area is maintained to ensure that the coils remain totally immersed at the end of the distillation. This is not a problem for directly fired stills.

The pot is equipped with close locking air, charging, discharging and safety valves. If manually operated, a special interlocking valve key that is shared
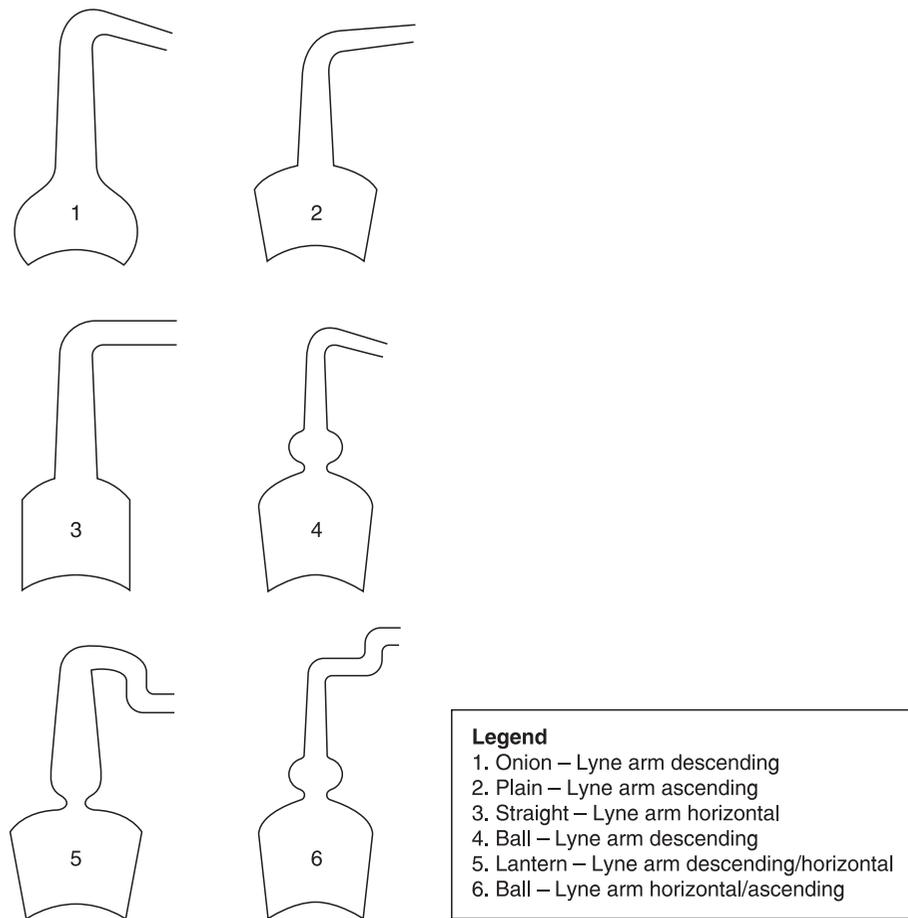
Figure 5.3
Still design configuration. 1, onion – lyne arm descending; 2, plain – lyne arm ascending; 3, straight – lyne arm horizontal; 4, ball – lyne arm descending; 5, lantern – lyne arm descending/horizontal; 6, ball – lyne arm horizontal/ascending.

between the air, discharge and charging valves is used. It can only be used in sequence, ensuring that the air valve followed by the discharge valve can be opened to prevent collapse or accidental discharge of the still. The reverse is applicable on charging, with sequential closing of the discharge valve, followed by the opening of the charging valve and finally closure of the air valve on the completion of charging and the application of heat.

Such requirements are redundant with today's use of automatic remote control through programmable logic controllers (PLC) with sequencing operations.

The pot connects to the swan neck via the ogee. Access to the pot is provided by a lockable brass man door.

Swan neck

The design of the neck can vary from short to long; it can be slightly tapered, straight-sided or severely swept in to the head. At the base of the neck it can be designed to resemble a lantern glass, assume a ball-shape or be connected directly to the pot with the still resembling an onion. The neck is usually provided with two sight glasses situated on opposite sides of the neck so that any foaming up the still, especially from boiling wash, can be observed. A light can be attached to the rear sight glass to illuminate the still internally.

A cold finger can also be installed at the top of the neck. This is supplied with cooling water, which is used to prevent any foam from boiling over into the receiver. To complete the attachments to the neck, a vacuum relief safety valve is fitted well above the reach of any foaming distillate in a wash still to avoid seizure of the valve seat by solids.

Head

The head is a curved extension of the neck, connecting it to the lyne arm or lye pipe. The head can be fitted with a thermometer to indicate the imminent arrival of condensate. The length or height of the head will dictate the degree of reflux within the still.

Lyne arm

The lyne arm, lye pipe or vapour pipe is of cylindrical construction and connects the head to the worm or shell and tube condenser.

The attitude of the lyne arm has an important bearing on the spirit characteristics. It can be designed to be horizontal, ascending, or descending to the condenser or worm tub, and may be short or long. Such permutations as there are will affect the nature of the new spirit.

The lyne arm can be interrupted by a purifier – a device fitted with baffles and cooled by an external water jacket or an internal coil. It is used to encourage heavy oils (higher fatty acid esters) to return to the still during distillation. The purifier returns the oils to the shoulder of the pot via a U-bend.

Worm tub or condenser

The worm tub is a vessel that contains a long coiled tube tapering to a nominal 76 mm at its outlet. The tub in which the worm is held is supplied with cooling water, which enters at the base of the tub and overflows at the top. At certain times of the year (in late spring or early summer) the supply of water may be insufficient to provide adequate cooling, and in such circumstances it may be necessary to incorporate a cooling tower or plate heat exchanger into the cooling circuit, the condenser water being air or water cooled. Seawater can be used for cooling heat exchangers in coastal distilleries.

A more advanced form of spirit condenser is the shell and tube condenser, or even a plate heat exchanger fitted with copper plates. If a condenser is used in a system that recovers sensible heat from the out flowing water at an elevated temperature (80°C), a sub-cooler, post-condenser, will be required so that spirit is recovered at less than 20°C.

Spirit safe

The spirit safe is used to monitor and control the cut point, strength and temperature of outflowing condensates prior to delivery to the relevant receivers. By means of spirit hydrometers, the strength of the low wines from a wash still, or foreshots, middle cut and feints from a spirit still, can be ascertained. The wash distillation is monitored using a hydrometer calibrated at 20°C in the full range 0–75 per cent alcohol by volume (ABV), or with a narrow range hydrometer reading 0–10 per cent ABV, to determine the completion of wash distillation.

On the other hand, a spirit still is controlled by two hydrometers, the tail pipe outflow being directed to one or other of two collecting bowls – one for foreshots and feints, and the other for potable spirit. By means of a swivelling spout the distillate flow can be directed to the foreshots and feints receiver or to the intermediate spirit receiver, as indicated by the hydrometer readings.

A small reservoir for cold water is provided for carrying out the demisting test. This test is used for distinguishing between foreshots and true potable spirit. By mixing foreshots with water to a strength of 46 per cent ABV, a time is reached when the normally milky/turbid mixture becomes clear at this strength. It is this test that determines the primary cut point; the second cut point is agreed according to the desired bouquet of the final spirit. The demisting test hydrometer jar is equipped with a three-way sampling valve. Distillate overflows from the hydrometer jars, draining via a small-bore pipe into the low wines and feints receiver.

Construction of stills and ancillary equipment

Any reputable coppersmith or distillery engineer should be capable of manufacturing the necessary equipment, from stills to receivers (Figure 5.2).

As previously mentioned, stills are totally constructed from copper, including the condensers or worm pipes. Man doors and valves are usually fashioned from brass, although stainless steel fittings can be used.

The pipework, 50–76 mm in diameter, can be made from suitable grade stainless steel, with flanges sealed using food-grade alcohol resistant gaskets.

The safe, as already described, is made of brass with plate-glass windows and a lockable lid for security. The safe is fitted with air vents capped with mushroom domes, and the vents can be extended to the external atmosphere through secure flame arresters.

Vessels for collecting charges – wash charger, low wines, foreshots and feints receivers, and intermediate spirit receivers – are now usually con-

structed of stainless steel. Coopered oak wood, epoxy-lined corten steel and glass-lined vessels have also been used for collecting distillates.

For excise purposes, vessels are gauged and fitted with dipsticks and striking pads for measuring wet dip. Volumes are calculated using the relevant gauging tables.

Valves are constructed of stainless steel and may be of ball, gate, butterfly or diaphragm design. When attached to gauged vessels, they must be lockable.

Vessels containing spirit are fitted with rousing devices – compressed air or mechanical screw agitation – for effecting good mixing prior to taking account.

Venting is also necessary in vessels, the vents leading to the external atmosphere and being fitted with flame arresters.

All electrical equipment must conform to current flame-proofing practice.

Pipe runs should be constructed so that dead legs are avoided, and should be angled down slightly, towards the collecting vessels, to aid drainage.

Wash still operation

A number of texts provide details regarding wash still operation and spirit distillation (see, for example, Lyons and Rose, 1977; Nicol, 1989, 1997; Whitby, 1992; Lyons, 1995; Piggott and Connor, 1995). Fermented wash with OG 1050–1060°**21 is pumped to the wash charger for wash preheating, such preheating being the norm in energy consuming processes.

The charge volume (two-thirds of the wash still capacity, usually lipping the bottom of the man door) is heated by either direct or indirect firing. Preheated wash, heat exchanged with hot discharging pot ale to within a few degrees of boiling, is gently brought in. Although the initial heat can be vigorously applied, as soon as the flap on the spout indicates the displacement of air and imminent arrival of distillate the heat should be reduced to prevent the still contents boiling over, which could result in a foul distillation.

Before charging the still the discharge valve is checked to see that it is closed, while the charging valve and air valves are open. The anti-collapse valve should be checked to see that it is moving freely. When the still has been charged to the correct volume, the air valve and charging valves are closed. The man door, if open, should also be closed, as the still contents expand with the heat and evolution of dissolved carbon dioxide. With the advent of the programmable logic controller, the sequential opening and closing of valves manually has given way to automation. As described above, with manual systems an interlocking valve key arrangement is used to prevent accidental opening of valves out of sequence.

The sight glass on the side of the still indicates the degree of frothing, which can occur during the initial distillation stages depending on the age of the fermented wash. Using this indicator, the amount of heat applied to the still contents can be controlled to prevent a foul distillation. When the frothing subsides, the heat can be increased to allow a steady, uniform flow of low wines to be collected.

The progress of the distillation is followed by hydrometry in the safe until the hydrometer reading indicates about 1 per cent ABV, when the distillation can be deemed complete. The end point of the distillation at 1 per cent ABV ensures that time and fuel are not wasted in recovering very weak spirit. A distillation cycle can last from five to eight hours, concomitant with the mashing and fermentation cycle.

When the distillation is completed and the low wines have been collected, the air valve is opened to equilibrate the internal still pressure with the atmospheric pressure. Failure to carry out this vital but very necessary procedure may result in a collapsing still, should the anti-collapse valve fail to respond automatically.

A record is kept of the original dip in the low wines receiver and of the final dip after distillation, and the temperature and low wines strength corrected to 20°C in the hydrometer jar is recorded every fifteen minutes over the period of the distillation cycle. Increasing distillation time is indicative of charring of wash on the internal still heating surfaces. This charring can be prevented by ensuring that excessive heat is not applied to cold wash – the temperature differential between hot surfaces should be kept to a minimum. Wash preheating minimizes this charring effect, which is equivalent to a cold protein break. Deteriorating heat transfer indicates the need for caustic cleaning, when caustic soda (1–2 per cent solution w/v) is boiled up within the still to strip the char from the heating surfaces. Should the charring be intractable and fail to respond to the caustic solution, it may be necessary to scrub the heating surfaces manually or to use an alternative sequestered caustic cleaning agent. The manufacturer's instructions should be followed and all safety precautions observed. On no account should pearl or powdered caustic soda be added directly to hot water as this will result in an exothermic blow back, when the solution boils violently. Work permits should also be issued both for cleaning with caustic soda and for entry into the enclosed body of the still.

Adequate protective clothing (including goggles and gauntlets) should be provided, and the washing residues must be neutralized with other acidic effluent, prior to further downstream processing.

Cleaning-in-place systems are now available and are preferred to manual cleaning as they greatly reduce the risk factors involved in caustic cleaning; an added benefit is the recycling of the cleaning agent.

The wash still is emptied via the discharge valve, the hot pot ale being heat exchanged with the incoming wash. Hot pot ale or hot preheated wash can be held prior to charging the still, which must be emptied before it can accept the preheated wash.

In some distilleries, the condenser water is regulated to outflow at around 80°C. This hot condenser water can be pumped through a mechanical vapour compressor or a steam ejector, the flash steam produced being used to drive the still. Such practices require the use of a sub-cooler to ensure that the low wines are collected at not more than 20°C.

Wash that is not fully fermented is at risk of causing foul distillations; this exacerbates any potential ethyl carbamate problem, and hence distillation of short fermentation washes should be avoided.

The volume of low wines collected approximates to one-third of the original charge volume.

Spirit still operation

Owing to the increased risk of loss of alcohol, low wines and feints are not normally preheated – although the discharge of spent lees may be treated similarly to those of pot ale as a vehicle for preheating.

As in a wash still, the spirit still is charged to a level not exceeding two-thirds of its working capacity. The precautions for charging are the same as in wash still distillation. The charge ingredients, a mixture of foreshots, low wines and feints, are of greater excise value and therefore require assiduous handling. Any loss of the charge or of the spirit will weigh heavily against the distiller.

The low wines and feints receiver is dipped before and after distillation. The spirit receiver into which the new potable will flow is also dipped both at the commencement of the new spirit run and at completion of the collection of the new spirit run when, as feints, the flow is directed towards the low wines and feints receiver.

A spirit distillation is divided into three fractions:

1. Foreshots
2. Middle cut
3. Feints.

The foreshots are the first runnings of the spirit distillation, and in most cases are deemed not worthy of collection as potable spirit, containing a high proportion of the highly volatile and flavoursome compounds like ethyl acetate. The time on foreshots is usually about fifteen to thirty minutes, when the incoming strength of the distillate (at around 85 per cent ABV) drops to 75 per cent ABV.

Normally a demisting test is carried out; this involves mixing foreshots with water in a hydrometer jar in the safe and reducing the strength of the mixture to 45.7 per cent ABV (Sykes Proof 80°). Initially the mixture is turbid, with a milky appearance not unlike the reaction between anis and water. This turbidity is caused by the displacement of water-insoluble residual long chain fatty acids and their esters, which have remained on the inner surfaces of the still and in the sub-pool at the bottom of the spirit condenser from the previous distillation. Being soluble in the high strength incoming foreshots, they are swept into the hydrometer jar. When the mixture of foreshots and water clears at the stated strength, the spirit is deemed potable. The flow of foreshots is redirected from the feints receiver to the spirit receiver, by means of the swivelling spout, as potable spirit.

Some blenders and distillers have abandoned this time-honoured technique, preferring to collect the foreshots after a timed run, with no resort to the

demisting test, regardless of the potability of the spirit. Such final distillates are high in higher fatty esters, making future chill-proofing more difficult.

Regardless of the way in which the spirit is deemed potable, collection of new spirit lasts for about two-and-a-half to three hours, during which time the strength drops from 72 to 60 per cent ABV, depending on the final cut point before the new spirit becomes feints.

The heat applied to the still as the foreshots distil and during the spirit distillation affects the spirit quality. Too harsh an application of heat will result in a fiery spirit that has not benefited from a gentle natural reflux on the sides of the swan neck. To avoid adverse flavour notes, both foreshots and middle-cut collections should be subjected to the delicate application of heat. On the other hand, feints can be treated like a wash distillation, following the initial collapse of the froth. The feints can be driven hard, reaching a distillation end point at 1 per cent ABV. The resulting residue (the spent lees) can then be discharged, observing the safety procedures adopted for discharging a wash still.

Chemical cleaning of the heating surfaces in a spirit still is rarely necessary and should be avoided to protect the internal patina, which is implicated in flavour reactions within the still.

Sulphur compounds present in the distillate vapour are (as with wash stills) highly volatile and odorous substances that take their toll on the copper, forming sulphides; the carbon dioxide in the wash encourages the formation of copper carbonate, which manifests itself as verdigris. It has been customary to suspend a muslin gauze over the spirit bowl to collect any such offending solids that might otherwise find their way into the new spirit.

Attack by carbon dioxide and sulphur also thins the copper so that eventually areas subject to this attack (above the boiling line, the shoulder, the swan neck, lyne arm, and condenser tubes exposed to vapour or the start of the worm) wear away, needing constant patching and eventually complete replacement. A still affected by erosion emulates the breathing of a dog, with the shoulders rising and falling in a rhythmic pattern; such a condition is known as panting, and indicates the need for replacement of the pot.

Like a wash distillation, a spirit distillation should last for five to eight hours in conjunction with the wash still distillation time.

Ethyl carbamate precursors, being more soluble in aqueous solution, exit via the spent lees (Riffkin *et al.*, 1989).

The alcoholic strength of the charge of combined foreshots, feints and low wines should not exceed 30 per cent ABV; strengths in excess of this lead to blank runs, when the demisting test fails to indicate potable spirit. In such circumstances the demisting test protects the previously collected potable charges from an influx of non-potable spirit, which, with its high concentrations of higher fatty acid esters and long chain saturated carboxylic acids, would impart a 'feinty' note to the spirit. The demisting test should always be available, even if foreshots are collected on a timed basis.

Low wines and feints receivers and chargers act as separating vessels. The last runnings of a spirit distillation contain the heavy oils or esters that are not readily soluble in water. Such oils have an affinity for alcohol, especially at

high strength. At a strength of less than 30 per cent ABV these compounds undergo a phase separation, where the esters float on top of the aqueous layer while a small proportion are dissolved in the aqueous layer. If the concentration of alcohol is allowed to exceed 30 per cent ABV, these floating surface oils migrate into the higher alcoholic strength aqueous layer, being completely dissolved. This effect eventually impacts not only on the demisting test, but also on the whole spirit distillation – potable spirit cannot be collected as the charge of low wines and feints contains a disproportionate concentration of heavy oils, making it impossible to have a turbidity-free demisting test result.

With low wines and feints charges at less than 30 per cent ABV, it is still possible to attract distillation problems. Presentation of the floating surface layer of heavy oils or higher fatty acid esters as a charge to the still (by completely emptying the contents of the charger into the still) will result in an episode when the collection of potable spirit (as determined by the demisting test) is unachievable. The whole spirit distillation system will have been contaminated by these esters, and it can take several distillations before satisfactory spirit is again obtained.

To avoid such scenarios, when the low wines and feints appear to be approaching higher strengths (or have even reached this situation) the charge can be diluted with water, aiming for a combined strength of less than 30 per cent ABV and thus stimulating hydroseparation. The surface phase must not be allowed to enter the spirit still on charging.

Adherence to these principles will ensure a consistent product, both on nose and analysis. The low wines and feints components reach a steady concentration state, maintaining equilibrium during subsequent distillations.

Product quality

A competent distiller ensures that the distillery staff are fully aware of the parameters that must be controlled to produce a high quality spirit.

To ensure consistency the plant and equipment must be designed in a balanced manner, and techniques borne of tradition strictly observed along with modern improvements allied to tradition.

First, the wash (whether traditionally derived from a worts recipe producing worts of OG 1050°, or lately at 1060° using high gravity brewing) should be fully attenuated, fermenting for at least 48 hours. It has been shown that short fermentations of less than 40 hours adversely affect the congener spectrum, producing an inferior spirit. Prolonged fermentations of more than 48 hours duration undergo a malo-lactic fermentation, the products of which, when distilled, produce a superior, mellower spirit. Even at 48 hours such a secondary fermentation is unlikely to have occurred, as it relies on the autolysis of the yeast cells and spilling of their contents to provide nutrients for lactic acid bacteria.

Fermentations should not be less than two days old to avoid an excessively gassy wash. A gassy, lively wash is difficult to distil, with much frothing,

giving the risk of a foul distillation and of producing a final spirit with an unacceptable ethyl carbamate concentration.

The wash still charge should ideally not exceed two-thirds of the working capacity of the still, thus reducing the risk of foul distillation and subsequent poor spirit quality.

Without wash preheating, the wash still contents should be heated up gently to prevent charring on the heating surfaces, which are more susceptible to burn-on of proteins and dextrins in the early stages of distillation if too much heat is applied (i.e. too great a temperature differential between the still contents and the heat within the pans or coils). With wash preheating, this temperature differential is much reduced and burn-on occurs far less, if at all.

An adequate supply of cooling water must be provided to avoid hot, uncondensed vapours reaching the spirit safe and causing serious damage to hydrometers, thermometers, jars and glass bowls, and hence detriment to the quality of the collected low wines and final spirit. Mercury-filled thermometers should not be used in contact with spirit; it is advisable to use only spirit-filled thermometers to avoid contamination.

The bouquet of the final spirit is influenced initially by the raw materials – the variety of barley malted, and the yeast. Water, too, influences the character. Processing parameters, including mashing temperatures, washback setting temperatures and length of fermentation, can also affect the flavour characteristics. Failure to observe agreed cut points will produce a spirit of highly fluctuating congener levels from batch to batch, and this can be ameliorated by collecting several batches of spirit distillations in a receiver that can hold three days' worth of distillations. The large capacity, with its ability to allow different batches to be blended, ensures that the peaks and troughs in congener concentrations are smoothed out.

With peated malts the spirit produced reflects a marked concentration of steam-volatile phenols, whose arrival in the spirit tends to concentrate towards the end of the distillation of the middle cut as the water to alcohol ratio alters with decreasing strength, favouring the entrainment of the phenols. To enhance the phenol concentration in the new spirit a lower strength cut point can be used, but not at the expense of producing a feinty spirit. A cut point of not less than 60 per cent ABV would be acceptable.

The rate of any spirit distillation is critical. Too rapid a distillation will result in an unpalatable spirit that is fiery in aroma and taste, lacking a proper congener balance. Foreshots and middle cut should be carefully and gently distilled to ensure adequate reflux, with foreshots completely purging the oily residues, which cling to the internal still surfaces, from the still. It is these residues that form the basis of the demisting test. Slow spirit collection ensures the production of a clean spirit, free from unwanted adverse aroma and flavour notes.

Throughout, it must be emphasized that an adequate supply of cold water to the condensers or worm tubs must be maintained. Inadequate cooling will result in a spirit being collected at temperatures greater than 20°C, which adversely affects the congener balance, allowing higher concentrations of compounds usually associated with the feints to distil over with the spirit. This is

also true of forced or rapid distillations. Warm weather means that to maintain spirit collection at less than 20°C the amount of heat applied to the still must be reduced to allow the spirit to be collected at a desirable temperature. This will result in prolonged distillation times, affecting the throughput of the distillery.

Collecting distillates at excessively high temperatures also increases spirit losses through evaporation. Malt whisky distillation is therefore facilitated by the low ambient air and water temperatures prevalent during the winter months. Distillation in previous centuries was, like curling, essentially a 'winter sport' – a time during which the floor malting of barley was easily controlled, providing fully modified malt unaffected by adverse summer temperatures and the resultant over-modified malt. Thus 'silent seasons' during the summer found malt distillery staff engaged in maintenance duties, harvesting barley and bringing peats home.

Contaminants that can bedevil the malt distiller are nitrosamines, ethyl carbamate, methanol, pesticide residues, haloforms, polycyclic aromatic hydrocarbons and herbicide residues, all of which are monitored according to laid down procedures governing frequency of sampling. Some of these compounds are derived from the raw materials, and others from the processing of malt or during distillation.

Genetically modified cereals and yeasts are now being scrutinized, as the definition of Scotch whisky demands the use of only pure water, yeast and cereals preferably derived from natural sources.

Copper has already been mentioned as a silent contributor to the quality of the spirit, as it removes the foul smelling, highly volatile sulphur compounds, and is allegedly implicated in the formation of esters. Copper catalyses the formation of ethyl carbamate from the cyanogenic glycosides derived from the original barley. Stainless steel is not recommended for the construction of the distillation apparatus to avoid compromising quality, but may be used for ancillary pipework and vessels.

The original gravity of wash impacts on spirit quality, and it has been determined that OGs in the range 1045–1050° encourage the formation of esters, thus imparting a fruity, sweet aroma to the finished product.

Efficiency – production yield

Having effectively designed and constructed a well balanced distillery, where milling, mashing, fermentation and distillation are in harmony, it is fairly easy to establish programmes of mashing and distillation in tandem. This is done by ensuring that the time cycles for mashing, fermentation and distillation are in step, with the week divided into set time periods reflecting the mashing cycle, so that if the mashing takes six hours to complete (with washbacks individually filled within this time slot), the maximum distillation time from still charging to discharging should not exceed six hours. This enables four mashes per day to be performed as if by clockwork. Better still,

the distillery may fully automated, removing the human element and its inevitable uncertainty.

One tonne of malted barley, fully modified and efficiently mashed, should ensure complete extraction of the available fermentable sugars, resulting in an overall distillery yield approaching 425 litres of alcohol. Without complete extraction, it will be impossible to achieve the potential spirit yield determined by laboratory analysis. Mashing efficiency is vital in achieving the maximum possible spirit yield.

On the distillation side, it is essential to ensure that the integrity of the distilling plant – pipework, vessels and stills – is maintained without leaks. Spirit can be lost through insidious vapour leaks that are not easily detected. The worm or the tube bundle, in a shell and tube condenser, are under constant attack by the sulphur compounds in the vapours, which eventually erode the copper. Condenser or worm leaks are noticeable from the entry of cooling water into the product side, reducing the strength of the distillate (as detected by the hydrometers in the safe) and most definitely producing a water flow into the safe when the still is off. Such a scenario requires that the still be run off and the offending tube spiled or blanked off before recommencing distillation. Other tubes are likely to be similarly affected, and the condenser should be pressure tested for further possible tube weaknesses. In the event of several tubes leaking, it will be necessary to replace the condenser and have the offending apparatus retubed.

Thinning of copper on the shoulder of the pot, swan neck or lyne arm can lead to the development of pinhole leaks as the copper becomes spongy. Such leaks are remedied by solder or the temporary use of molecular metal. Soldering requires the use of a blowtorch, and flammable vapours must be purged from the system with the blanking off of receivers to prevent explosion and fire. Such repairs require the complete cessation of all distilling operations. Leaks are therefore not to be tolerated and must be dealt with as quickly as possible.

Other losses occur via the pot ale or spent wash and spent lees, when the distillation end point is not accurately observed. Again these end points would indicate energy is being wasted if the distillation is continued beyond 1 per cent ABV as indicated by hydrometers in the safe. Early cessation of distillation at greater than 1 per cent ABV will result in significant residual ethanol being detected in the pot ale or spent lees. Permissible spirit losses (per cent ABV) are:

Pot ale	< 0.03
Spent lees	< 0.03
Condensate	< 0.0001
Condenser water	< 0.0001

Where pot ale is required for evaporation, ethanol interferes with the evaporation efficiency.

The distillery yield is calculated from the weekly production figures, and takes account of the weight of malted barley used, and the amount of spirit remaining in the low wines and feints receiver, intermediate spirit receiver

and final spirit receiver warehouse vat. Depending feints carried forward from the previous week are deducted from the total sum of spirit produced, expressed in litres of absolute alcohol.

Calculation of distillery yield

(a) Depending feints   15 500 l abs. alc., carried forward from previous week
(b) Spirit produced in ISR 7200 l abs. alc.
(c) Spirit receiver W/H vat 30 300 l abs. alc.
(d) Feints remaining   13 500 l abs. alc.
(e) Spirit produced $(b) + (c) + (d) - (a) = 7200 + 30\,300 + 13\,500 - 15\,500$ $= 35\,500$ l abs. alc.
(f) Tonnes of malt mashed   85.54
(g) Distillery yield $\dfrac{\text{Spirit produced (l abs. alc.)}}{\text{malt used (t)}}$
$= \dfrac{35\,500}{85.54}$
$= 415$ l abs. alc. per tonne

Customs & Excise is able to calculate the projected amount of spirit produced from a given amount of malt through the attenuation charge.

Calculation of attenuation charge and percentage over-attenuation

Say, for example, the attenuation across several fermentations comprising a week's production is 57° gravity from an OG of 1055° and an FG of 998°, or 02 under. For ten fermentations of total volume 485 000 litres of wash and an average attenuation of 57°, the attenuation charge is calculated as follows:

$$\text{Attenuation charge} = \frac{\text{litres of wash} \times \text{average attenuation}}{8 \times 100}$$

$$= \frac{485\,000 \times 57}{800}$$

$$= 34\,556 \text{ l abs. alc.}$$

Customs & Excise calculates the percentage over-attenuation from the following formula:

$$\frac{\text{Spirit produced (l abs. alc.)} \times 100}{\text{Attenuation charge}} - 100 \times 100$$

$$= \frac{35\,500 \times 100}{34\,556} - 100 \times 100 = 2.73 \text{ per cent}$$

Hence an acceptable percentage over-attenuation has been achieved of 2.73 per cent. A 3.0 per cent over attenuation is deemed acceptable; any departure from this figure by more than 1–2 per cent either way demands an investigation.

Under-declaring the OG of worts collected will inflate the figure, while over-declaration will reduce the figure. Resort to laboratory analysis for OG determinations is necessary. The law demands that at least six declared washbacks per week should be examined to find out the true declarations, with the distiller adjusting the saccharometer readings to allow for the amount of work done relating to the temperature and gravity at the time of declaration.

Triple distillation

Within the Scotch malt whisky industry, there are at least two distilleries that practise triple distillation. This technique ensures a lighter spirit at higher natural strength than double-distilled whiskies, and it is primarily carried out in lowland distilleries. It is much akin to the distilling practice in Ireland.

In principle, there is a wash still from which two fractions are derived – strong low wines and weak low wines – and are separately collected. A second still, the low wines still, is charged with the weak low wines. From this low wines still, two fractions are similarly collected – strong feints and weak feints (tails). The strong feints are presented to the third still, the spirit still, while the weak feints are redistilled in the low wines still.

The distillates from the spirit still are divided into three collected fractions – the foreshots or heads, the new spirit, and the tails (which, with the heads, are collected and returned for redistillation in the spirit still). This recycling of the various fractions derived from the low wines still and the spirit still impacts on the final bouquet and strength of the new spirit, which is collected at a strength in excess of that normal double-distilled products, which are usually in the region of 68–72 per cent ABV. The triple-distilled product can approach a strength of 90 per cent ABV.

The Irish distilleries boast very large pot stills in comparison with those used in the double-distilling technique of their Scottish cousins. It is not within the remit of this chapter to discuss the triple-distillation technique in fine detail.

Dealing with distillation problems

As with all manufacturing processes, problems can occur that impact on the quality of the finished product if not addressed quickly. Such problems can occur at the mashing, fermentation and distillation stages.

Atypical percentages over-attenuation can be attributed to false declarations when determining the original gravities in a set washback. This is overcome by laboratory determinations of OG over at least six declared washbacks, and calculating the allowance for loss in gravity due to fermentation and to tem-

perature. The average OG is calculated, and the difference between what was observed at the time of declaration and the true laboratory OG is the figure that must be added to the tun room declaration.

Saccharometers and thermometers must be regularly checked for accuracy against standard solutions and thermometers. The addition for work and temperature is then not guesswork.

All worts and final washes should be attempered to 20°C prior to taking readings.

Product losses, with low percentages over-attenuation accompanied by low yields when compared with the potential spirit yields obtained by analysis, are invariably caused by one or a number of problems – poor mash tun extraction efficiency, infection (Geddes, 1985), high wash fermentation temperatures, or physical losses of worts, wash, low wines and feints or even spirit. To improve mash tun extraction efficiency it is necessary to examine sparge to malt ratios and mashing temperatures, especially the first water. Overloading a mash tun with excess goods most definitely results in poor extraction, with loss of potential fementable sugars with the draff.

Infection, which competes for the fermentable sugars with the yeast, is overcome by paying close attention to hygiene, especially throughout the mashing and fermentation plant (dead legs are especially vulnerable).

Setting temperatures within the washback can be adjusted downwards to avoid overheating – temperatures in excess of 33°C not only result in evaporation losses but also encourage lactobacillus development, which impacts adversely on the final flavour and can even result in the production of acrolein.

Physical losses of process materials can be due to accidental plant failure or mistakes by personnel, demanding management investigation.

Wash distillation can be beset with foul distillations, resulting in the wash still boiling over and overwhelming the safe, with damage to the safe and its instruments. The low wines and feints are contaminated with wash, with an increased risk of encouraging ethyl carbamate formation. An overloaded still, excessive application of heat and even blocked condenser tubes can cause this problem.

Fresh, lively wash can also cause wash distillation control to be difficult, and should be avoided.

Organoleptic problems, manifested in 'feinty' spirit, are caused by the overrun of the middle cut. Spirit hydrometers and high condensate temperatures should be suspected, with the necessary corrections being made.

It is possible to be plagued with blank spirit runs, when no spirit is collected, with resultant adverse effect on fuel usage. This is caused by weak charges or an imbalance in the low wines and feints, which may have increased in strength allowing a high concentration of fusel oil to dissolve in the aqueous alcohol layer. This is best avoided by distilling low wines and feints charges at a strength of less than 30 per cent ABV. It can be overcome by adding water to the charge to reduce the strength to below 30 per cent ABV.

Discharging the total contents of a low wines and feints charger into a still is a recipe for blank runs, as the fusel oil layer that collects on the surface of the

low wines and feints in the receiver/charger can contaminate the still, making it impossible to have a successful demisting test when applied to the foreshots. A balanced distillation regime can overcome these problems.

The collection of weak spirit, resulting in an altered congener balance, can be attributable to poor reflux, poor cooling or a leaking condenser. It will be necessary to slow the distillation rate to improve the reflux and cooling. A leaking tube in a condenser manifests itself by presenting a water flow to the safe when the distillation is completed. The condenser tubes should be checked and pressure tested. Any offending tube(s) should be temporarily blanked off by spiling, and a constant check maintained for future leaks. One leaking tube indicates potential multiple tube failures at a later date.

Wash stills are prone to the fouling of direct or indirectly fired heating surfaces, which causes increased distillation times with corresponding energy wastage. The obvious cure for this problem is to caustic (1–2 per cent) clean the offending surfaces.

High ambient water and air temperatures, especially in summer and when using surface waters, will result in higher temperatures of low wines and spirit entering the respective safes. To counteract this it is necessary to cut back on the heating input to the stills to reduce the distillate temperature. It is recommended that distillates should be collected at a temperature not exceeding 20°C to avoid evaporation losses and adverse organoleptic effects.

Reasons for distillation losses can be obvious, and such losses can be dealt with expeditiously. However, many can be insidious and difficult to pinpoint.

Water in the safe, as already mentioned, indicates leaking condenser tubes.

An almost inaudible high-pitched hissing emanating from a still equipped with steam heating during distillation indicates a steam leak, which not only dilutes the charge but can also result in product gaining access to the condensate system, with resultant losses and condensate contamination. Product access to the steam side of the coils or pans occurs during charging, before the steam is applied.

All flanges associated with the steam heating system should be checked for leaks. Coils or pans, especially those made of copper, may also have developed cracks or pinholes.

Vapour losses from the body of the still above the charge line result from pinholes, with invisible spirit vapour exhausting to atmosphere. Such leaks usually manifest themselves by staining the external copper surfaces. Losses can also occur behind insulation, and such areas should not be ignored. These unsightly leaks can seriously affect the yield if not dealt with quickly. Passing valves – such as the air and anti-collapse valves – will also cause losses. This defect is recognized by stains on the body of the still. Flanges on pipe runs can also be a source of leaks.

It is therefore in the management's interest regularly to inspect the integrity of the whole system to prevent waste through unnecessary losses of product, which can adversely impact on the quality and quantity of the spirit recovered from each mashing and distillation period.

Laboratory techniques

Distillers aim to satisfy quality, cost and customer requirements to the best of their ability, within the confines of the law. To ensure that these objectives are met, a team of chemists, chemical engineers, microbiologists and accountants help to guide the distiller through the maze of fiscal and environmental regulations. From the purchase of the raw materials through to the production of the final distilled product, the distiller must ensure that each stage of the process is optimized to provide maximum return. Customs & Excise is also concerned with the maximum recovery of spirit. **23The Institute of Brewing's *Recommended Methods of Analysis* (1997) gives details of how to predict the amount of spirit from a known weight of malt – the potential spirit yield (PSY). This potential yield can be compared with the actual distillery yield, and a distillery efficiency can be calculated as a percentage. The actual alcohol content of individual wash backs can be determined and cumulatively compared with the actual overall distillery yield. A large difference between expected volumes of spirit by laboratory analyses would highlight physical losses either between the tun room or during distillation, with accompanying poor percentage over-attenuation.

Spirit losses can occur in several areas, as previously mentioned, so that routine checks for spirit contamination should be carried out on condensate from steam-heated stills and on condenser outflow cooling water, as well as pot ale and spent lees.

With reference to potential condensate and condenser water spirit losses, it is prudent to concentrate on lines coming from individual stills, rather than on manifolds.

The Institute of Brewing's *Recommended Methods of Analysis* (1997) concentrates on process controls to prevent potential spirit losses and the optimization of yield.**24 Other analyses are required to satisfy Customs & Excise, food safety and environmental regulations. The distiller, by definition of The Scotch Whisky Order (1990), is required by law to use only pure water and yeast in a cereal mash saccharified by the enzymes of malt in the production of grain or malt whiskies. The strength at which the whisky is collected must not exceed 98.4 per cent ABV, thus encompassing grain whisky.

Food safety regulations impact on the malt whisky process, from water through to the final product. The water used must be wholesome, and free from chemical and potential pathogenic contaminants. Water is thus checked for its microbiological integrity. Well water is less susceptible than surface waters.

Chemical analyses of water and spirit cover pesticides, herbicides, fungicides, polycyclic aromatic hydrocarbons, nitrosamines, and ethyl carbamate analyses. Heavy metal analyses, particularly for lead, should also be included in the 'duty of care' and 'due diligence' analyses.

In conjunction with organoleptic assessments, full-scale gas liquid chromatography of the congeners in each new spirit charge should be carried out, using glass or capillary columns (Nykanen and Suomalainen, 1983).

Flavour compounds that are peculiar to an individual spirit in their degrees of concentration are acetaldehyde, diacetyl, ethyl acetate, fusel oils, furfural, and ethyl esters of midrange fatty acids and higher fatty acid esters. Phenol analysis is carried out on peated malt spirits. Variations from the standard spirit detected by nose may be confirmed by congener analysis (see Table 5.1). Furfural and ethyl lactate concentrations, if high, reflect acidic wash conditions due to lactobacillus infections – ideal for the production of such compounds during distillation and also for the release of steam volatile phenol from the wash.

Environmental pressures now demand that distillery emissions, both liquid and gaseous, should fall within agreed environmental constraints. Biological oxygen demand (BOD), pH, suspended solids and volumes must be monitored and controlled within the agreed limits. The temperature of cooling

Table 5.1
Typical malt distillery analyses
(g/100l abs. alc.)

	Speyside	Islay
Acetaldehyde	5.4	7.0
Ethyl acetate	26.3	33.2
Diacetyl	2.1	2.8
Methanol	4.8	9.0
Propanol	40.3	37.6
Isobutanol	80.1	85.6
o.a. Amyl alcohol	44.2	53.1
Iso-amyl alcohol	138.9	170.8
Total higher alcohols	303.5	347.1
Ethyl acetate	3.7	5.3
Ethyl octanoate	1.8	2.7
Furfural	3.6	4.8
Ethyl decanoate	6.0	8.9
β-Phenethyl alcohol	7.2	8.7
Ethyl myristate	0.9	1.2
Ethyl palmitate	2.8	3.3
Ethyl palmitoleate	1.6	2.1
Phenols	1.0	4.5

water discharges must equate to the temperature of the receiving waters within a narrow range. Most of the effluent volumes in rural distilleries are associated with pot ale, spent lees, washing waters and boiler blow down.

Pot ale, with draff, is the main component of distillers' dark grains. Excess residual ethanol and high levels of suspended solids in pot ale adversely affect evaporation efficiencies, with ethanol loss being detrimental to the distillery yield.

From the humble retort, saccharometer, Syke's spirit hydrometer and monocular brass microscope used for early simple analyses, the distillery chemist now has an extended range of sophisticated analytical instruments and tests, including the following:

- The atomic absorption spectrometer – provides metal analyses (lead, mercury, copper, iron, calcium and magnesium)
- Ion chromatography – tests for chloride, sulphate, nitrate and carbonate anions
- Gas chromatography/mass spectrometry – tests for ethyl carbamate
- The thermal electron analyser – tests for N, N-dimethylnitrosamine
- Gas chromatography (capillary or glass) – tests for congeners/aroma compounds
- The densitometer – measures the specific gravity/density/percentage alcohol in worts, wash and spirit
- High performance liquid chromatography (HPLC) – measures carbohydrates and wood maturation compounds in spirit
- Original gravity analysers
- The BOD analyser – an automatic barometric apparatus
- The near infrared analyser (NIR) – tests for cereals/co-products.

Most of the above instruments are now equipped with automatic samplers, allowing several analyses to be carried out sequentially – preferably overnight – with the results calculated by computer.

The future

Environmental, energy, fiscal, health and safety pressures will continue to impact on distillers. Already reports have indicated that distillers with private water supplies may be forced to pay for licences to abstract water from sources that they have used freely for more than a century.

Press reports have indicated that once thriving communities attached to distilleries are evaporating as tied houses are abandoned for more secure rented or purchased properties, leaving distilleries surrounded by ghost premises.

Companies are now considering the use of itinerant teams of production workers, concentrating on a few weeks' production at one distillery to satisfy

customer requirements before moving on to the next distillery in a peripatetic production campaign. This can only be done where the knowledge of plant and processes is very similar.

Barley, malted barley, yeast and water, as raw materials, will continue to be researched for the perfect combination that enhances spirit yield, without detracting from the uniqueness of an individual distillery's product.

Other countries will continue to produce their own peculiar form of whisky, while many products matured in wood (including cognac and rum) will compete as batch distillates with that universal spirit, whisky.

A complete glossary of terminology relating to whisky has been compiled by Smith (1977), and is a useful reference source.

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Chapter *6*

Grain whisky distillation

Iain Campbell

Introduction

Early in the history of legal whisky distillation in Scotland, it was realized that the operation of traditional pot stills was expensive in terms of raw materials, labour and energy. By 1830 more efficient continuous distillation had been developed, principally by Stein and Coffey (Whitby, 1992). Although the spirit from continuous distillation, even from an all-malt wash, contained lower levels of flavour congeners than pot-distilled spirit, in the 1830s that may have been an improvement. However, continuous distillation stimulated two important developments: it was realized that it was unnecessary to use expensive malt to produce the milder flavour of continuously distilled spirit, since a mash prepared largely from unmalted cereal provided a satisfactory product, and also that the flavour of the whisky could be controlled by the process of blending grain and malt whiskies. So both grain whiskies and the modern range of blended whiskies are derived from the introduction of continuous distillation. There was also the export value of continuously distilled spirit as feedstock for the production of gin in England.

Theory of continuous distillation

The separation of the components of a liquid by batch or continuous distillation is based on differences in volatility. Data on boiling points are easily available and provide a useful guide, but in certain circumstances can be misleading. For the simple case of a solution of ethyl alcohol in water, the relationship between temperature and composition at constant pressure (usually atmospheric) is represented in Figure 6.1. The units should actually be expressed in percentages of alcohol by weight, but since Customs & Excise in Britain requires units of alcohol by volume, distilleries must use volume
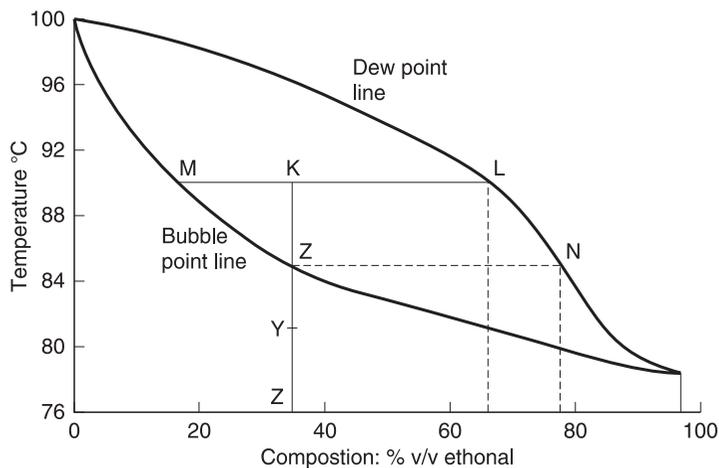
Figure 6.1
Distillation temperature versus composition of a pure ethanol/water mixture at constant pressure of 1 bar (101.3 kPa). Values of alcohol by volume were calculated from the data of Seader and Kurytka (1984).

units for all production records. Note that the graph is valid from 0 to 97.2 per cent ethanol by volume (equivalent to 95.6 per cent by weight), the azeotropic point. At this point the percentage of alcohol in the vapour at atmospheric pressure is equal to that in the liquid.

In engineering terms, the mole fraction of ethanol in vapour and liquid is equal (89.43 per cent) at 78.15°C, the boiling point of the azeotropic mixture at atmospheric pressure (Table 6.1). With decreasing mole fraction of ethanol the boiling point increases, ultimately to 100°C with pure water. For a binary mixture temperature and pressure fix the equilibrium vapour/liquid composition, but in industrial practice for alcohol/water distillations the pressure is fixed at 1 bar (101.3 kPa) and the mole fraction of ethanol in the vapour over the range 78.15°C to 100°C is as shown in Table 6.1 (Seader and Kurtyka, 1984). It is impossible to achieve a higher alcohol concentration than the azeotropic mixture by conventional distillation, although stronger ethanol than 97.2 per cent by volume is possible under vacuum, ultimately reaching 100 per cent at 9.3 kPa (Cosaric *et al.*, 1983). However, for some other two-component mixtures, such as benzene–toluene, distillation to 100 per cent of the more volatile component can be achieved.

Figure 6.1 shows that at temperatures below the bubble point (at which the liquid evaporates) the two-component mixture exists only as liquid, and at temperatures above the dew point (at which the vapour condenses) the mixture exists only as vapour. The distilling process depends on the region between the graphs of bubble point and dew point, a two-phase state where mixtures of liquid and vapour alcohol and water co-exist. For a particular strength of ethanol X, arbitrarily chosen at 35 per cent, at point Y the mixture is still entirely in the liquid phase, but as the temperature increases above

Table 6.1
Vapour/liquid equilibrium data for ethanol/water mixtures at a constant pressure 1 bar (101.3 kPa or 101.3 kN/m^2) (calculated from Seader and Kurtyka, 1984)

Temperature (°C)	Mole fraction of ethanol in:		% w/w:		% v/v:	
	Liquid	Vapour	Liquid	Vapour	Liquid	Vapour
78.15	0.8943	0.8943	95.6	95.6	97.2	97.2
78.41	0.7472	0.7815	88.4	89.3	92.1	92.8
78.74	0.6783	0.7385	84.2	87.8	88.9	91.7
79.3	0.5732	0.6841	77.4	84.7	83.3	89.2
80.7	0.3965	0.6122	62.7	80.1	70.3	85.6
82.3	0.2608	0.5580	47.4	76.3	55.2	82.4
84.1	0.1661	0.5089	33.7	72.6	40.5	79.2
86.7	0.0966	0.4375	21.5	66.5	27.5	73.8
89.0	0.0721	0.3891	16.6	61.9	20.4	69.6
95.5	0.0190	0.1700	4.7	34.4	5.9	41.2
100.0	0.0000	0.0000	0.0	0.0	0.0	0.0

point Z an increasing quantity of vapour is formed. Therefore at the temperature corresponding to point K there is a two-phase equilibrium mixture: vapour of composition L and liquid of composition M. The relative amounts of liquid and vapour in the mixture are in proportion to the lengths of KL (liquid) and KM (vapour), and the concentration of ethanol in the condensed vapour is shown by the position of L on the composition scale. In the non-equilibrium state of a batch distillation, a mixture of composition X would be distilled at a temperature just slightly above Z and the vapour removed to the condenser, yielding an alcohol concentration of approximately N per cent in the condensate. In batch distillation, however, ethanol is distilled off the ethanol concentration in the liquid falls, with a corresponding decrease in the strength of the distillate. An important advantage of the steady-state conditions of continuous distillation is the constant composition of the distillate, since vaporized alcohol is continuously replenished from the wash.

The basic design of a continuous still for any two-component mixture is shown in Figure 6.2, although the fourteen perforated plates shown are insufficient for separation of most mixtures of two compounds of different volatility. The feed, preferably already at bubble-point temperature for its concentration of the volatile component, will form the equilibrium vapour/liquid mixture appropriate to the temperature at the level of entry (which is
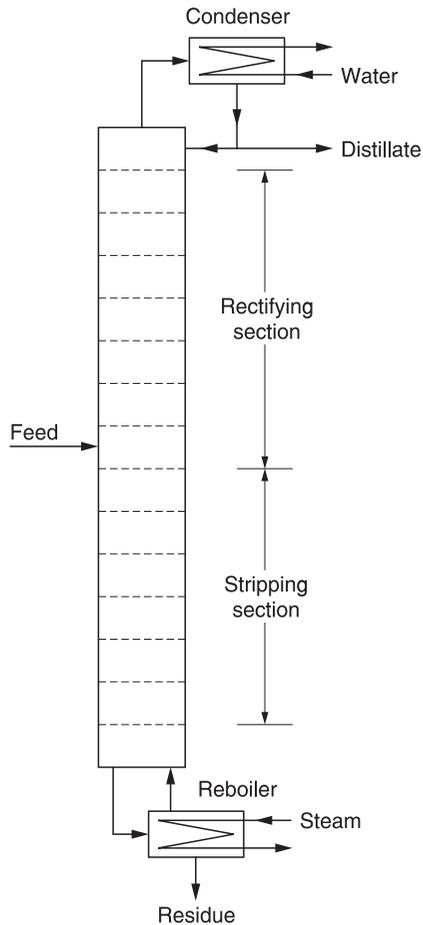
Figure 6.2
Simple distillation column.

not necessarily the middle of the column as in Figure 6.2). Part of the vapour condenses as it bubbles through the slightly cooler layer of liquid on the plate immediately above, releasing its latent heat of condensation (or evaporation), which is then available to vaporize a proportion of the liquid level at that level. In this state of dynamic equilibrium the vapour phase, increasingly enriched in the more volatile component, rises in the direction of decreasing temperature. The liquid stream flows down the column in the direction of higher temperature, increasingly losing the more volatile component at each lower level of the cascade.

Liquid leaving the bottom section of the still is re-heated in an external calandria, or reboiler, to generate the vapour for operation of the still, incidentally evaporating any residual ethanol. Technically it is a partial reboiler, since only part of the bottom product is evaporated and the residue is discharged,

either as a purified product (as in the petrochemical industry) or as effluent (as in the Scotch grain whisky industry). For an aqueous mixture like alcohol and water the heat source could be steam injected into the base of the column, but an important advantage of a reboiler is that the primary steam supply is a closed loop returning to the main boiler. With direct injection, steam is lost into the still system and has to be replaced continuously by expensively treated boiler-feed water. Also, the bottom product is diluted by the condensed steam.

The vapour leaving the top of the column is condensed and a proportion of the resulting liquid is returned to the top plate to maintain the liquid level there, and to sustain the downflow of reflux to lower sections. It is a total condenser in the sense that all vapour is condensed, but only part of the distillate can be drawn off as top product; the remainder must be returned to the top plate as reflux.

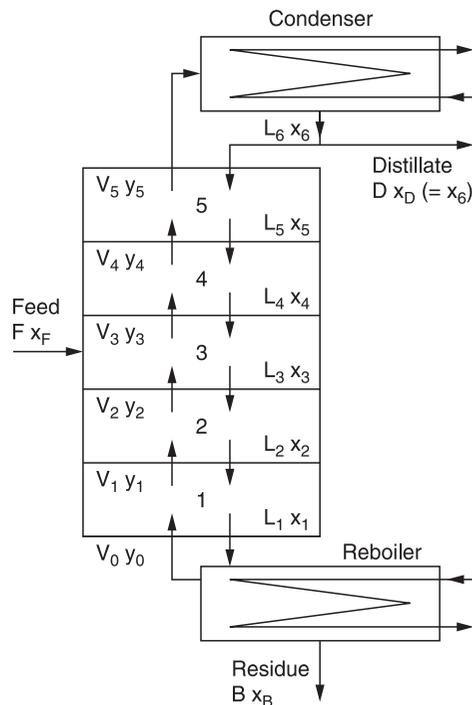Distillation in a continuous still can conveniently be represented as the cascade shown in Figure 6.3.

Figure 6.3
Equilibrium stages of a simple distillation column. B, flow rate of still bottom product (residue from reboiler); D, distillate flow rate; F, feed flow rate; L, liquid flow rate; V, vapour flow rate; x, mole fraction of more volatile component in liquid; y, mole fraction of more volatile component in vapour (Seader and Kurtyka, 1984).

For simplicity only five equilibrium stages of the column are shown – the feed plate itself and two plates above and two below – although the reboiler and top condenser, conventionally not shown as part of the column, also function as equilibrium stages 0 and 6 respectively. So in total Figure 6.3 shows seven equilibrium stages. Continuous distillation is a multi-stage counter-current process in which liquid and vapour phases of the mixture are in contact at each of the equilibrium stages, and are separated before rising (vapour) or falling (liquid) to the adjacent stage. The composition x and y of liquid and vapour respectively will be different at the individual levels of the cascade. Since the system operates under steady-state conditions there is no sudden change in composition around plate 3, the feed plate. The continuous flow of saturated liquid feed to stage 3 will adjust to the temperature (and pressure, if relevant) at that point to form an equilibrium vapour–liquid mixture. The vapour, V_3, of composition y_3 rises from stage 3 to bubble through, and partially condense in, the layer of liquid on plate 4, which, having a higher concentration of the more volatile component, is at a lower temperature than plate 3. Liquid descending from plate 5 (L_5, of composition x_5) to flood plate 4 and rising vapour V_3 mix on plate 4 and reach a new liquid-vapour equilibrium, and vapour V_4 of composition y_4 rises to plate 5. At each higher level the process is repeated, at a lower temperature, and y_3, y_4 and y_5 represent increasing percentages of the more volatile component. Referring to Figure 6.1, vapour rising from a plate equivalent to the line MKL contains the equivalent of L per cent of the more volatile component, which creates a new temperature and composition equilibrium on the plate above.

The liquid component flows down the column in the direction of increasing temperature, becoming increasingly enriched in the less volatile component. Therefore y_0 to y_5 represent an increasing concentration of the volatile component in the vapour, and x_6 to x_1 represent a decreasing proportion of the volatile component in the liquid.

The number of stages to achieve the required separation can be calculated from material balances at each plate, and determining the equilibrium compositions of vapour and liquid at each plate. For a discussion of these aspects of distillation, refer to specialist chemical engineering sources (e.g. Seader and Kurtyka, 1984; Coulson *et al.*, 1991). However, such texts concentrate on the mathematical treatment of two-component systems and, perhaps with good reason, do not consider the more complex composition of distillery wash.

The theoretical model of Figures 6.2 and 6.3 is inappropriate to the distillation of grain whisky spirit for several reasons:

1. The still is required to separate several hundred flavour congeners from fermented wash (Nykanen and Suomalainen, 1983; Korhola *et al.*, 1989), not just ethanol from water
2. The concept of theoretical plate is not entirely relevant to distillation in practice, since more than one actual plate is required for each theoretical plate of the calculation

3. The top condenser cannot be a total condenser since a proportion of the most volatile congeners must be vented off to prevent an unacceptable increase in the spirit over the duration of the distillation run

4. Even so, the highest plates have an unacceptable concentration of these more volatile congeners and the spirit is drawn from the column at a point several plates down from the top.

5. Also, at the highest plates the ethanol concentration may reach the legal maximum of 94.7 per cent ABV**27 for Scotch whisky spirit, but a concentration no more than 94.0 per cent in the collected spirit is preferred in practice, for increased content of flavour congeners.

6. A reboiler cannot be used, since flavours produced by boiling the spent wash would be unacceptable and direct injection of steam to the column is necessary; also, grain and yeast material in the wash would bake on to the steam coil of a reboiler, creating heat transfer problems as well as off-flavours

7. Finally, distillation of wash, which is unlikely to exceed 0.03 mole per cent ethanol (9.14 per cent by volume), requires so many plates that a single column would be too tall, and the stripping and rectifying sections must be constructed as two adjacent columns.

Therefore the stripping section of the still in Figure 6.2 is represented by the separate analyser column in Figure 6.4, with the heated feed entering at its top plate. Since the rectifying section is a separate structure, vapour equivalent to V_3 of Figure 6.3 is piped from the top of the analyser to the bottom of the rectifier column. Although only a single pipe is shown for hot spirit vapour in Figure 6.4, normally there are two, of sufficient diameter to permit a free flow of vapour between the columns. The wash feed is heated as it flows within a copper pipe (the wash coil) through the rectifier column, so it is at bubble point temperature as it is discharged on to feed plate, the top plate of the analyser. The wash coil itself also provides additional reflux as the vapour within the rectifier column condenses on its external surface.

Feedstock for distillation

The physical composition of wash varies between different grain whisky distilleries (see Chapter 3). In some, the complete mixture of ground malt and cooked cereal is cooled to the initial temperature of fermentation, usually about 20°C, and inoculated for an 'all grains in' fermentation. In others, some form of separation of solids is practised between mashing and fermentation: removing the coarser particles of cereal between mash tun and washback minimizes damage to pipework, washback and still by abrasion. There is the additional possibility of chemical and physical variation according whether maize, wheat or other cereal is used – for example, the higher oil content of maize has an valuable antifoam effect during fermentation and in the analyser column.
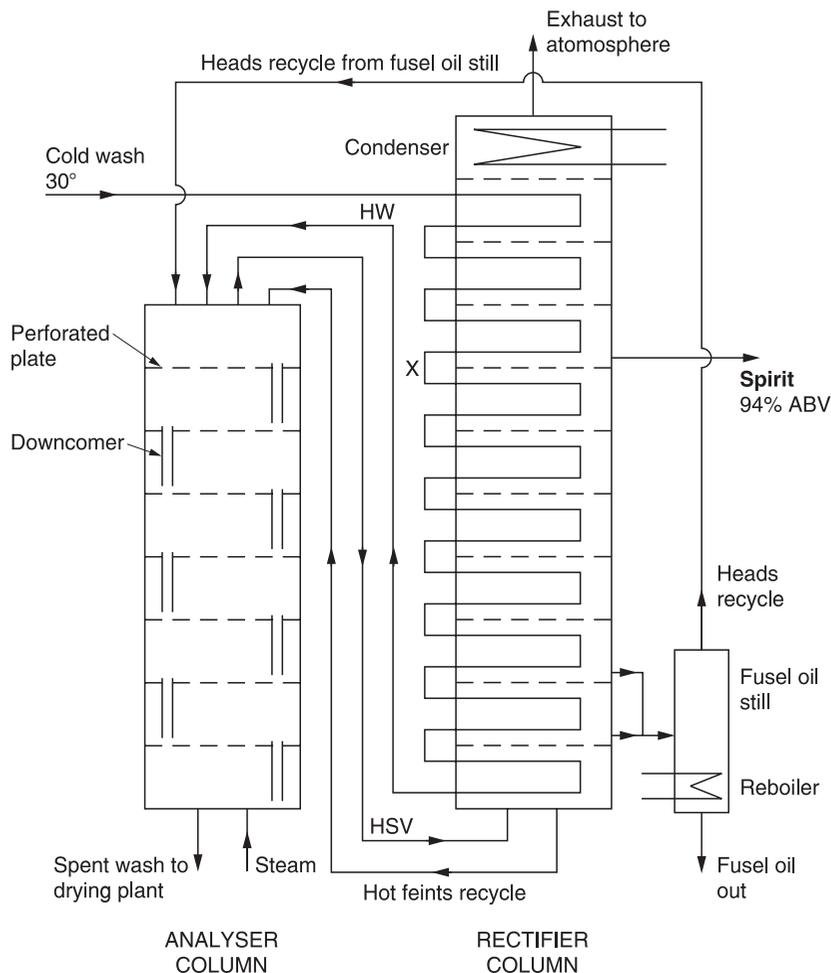
Figure 6.4
Essential features of the Coffey still. HSV, hot spirit vapour; HW, hot wash. X is the wash coil bend at the spirit plate; the temperature here controls the flow rate of wash.

The temperature rises to 30–34°C during fermentation, and as an energy-saving procedure the wash should be distilled before any significant cooling occurs. It is common practice to discharge the washbacks into a still charger vessel of capacity approximately that of two washbacks, and equipped with a mixer to ensure homogeneous composition (and incidentally to remove much of the CO_2). Wash is pumped continuously to the stills, and as the level of wash in the charger falls below half full the contents of the next washback can be added. However, there is a potential microbiological hazard in the continued use of the charger vessel over a period of time without regular cleaning and sterilization.

To maintain stable operating conditions in the stills it is useful to maintain a constant alcohol composition of the wash. Mixing in the still charger vessel helps to balance slight variations in alcohol content between fermentations, but for many years dilution with warm water (30°C) to a constant percentage of alcohol was considered to be necessary. This was normally the lowest concentration typically achieved in fermentation.

Although weaker wash may be preferable for more stable operation and optimum separation of flavour congeners, the potential energy savings from distillation of the strongest possible wash are now more important. However, in continuous stills for grain whisky production it becomes increasingly difficult to maintain the necessary steady-state conditions with increasing alcohol content of the wash, and above about 8.5 per cent alcohol by volume they need particularly careful attention.

Only to a limited extent can the still adjust itself to a change in the alcohol content of the wash during the run. A weaker wash produces a smaller amount of spirit and therefore an increase in reflux, since the proportion of the less volatile compounds has increased. Conversely, introduction of stronger wash generates more ethanol and a decrease in reflux. Therefore less fractionation occurs of the various flavour congeners. Ultimately with increasing alcohol content the system requires greater fractionation than the still is capable of providing by its fixed number of plates, so, whatever the energy implications, it would be necessary to dilute the wash if it exceeds the maximum strength the still is capable of distilling to the desired quality standard. In theory, increased alcohol content could be compensated by reducing the steam supply (as in pot stills, slower distillation means more reflux), but in practice that would probably not be an option, since the wash would then tend to fall through the holes in the plates. The still is designed for a specific flow rate of steam, within narrow limits.

Design and operation of continuous grain whisky stills

Various designs of continuous still are used in the Scotch grain whisky industry. Figure 6.4 shows a simplified drawing of a Coffey still, which until recently was the commonest type in Scotland. Coffey's original design (Figure 6.5) differs in several respects from the modern still bearing his name, but the essential features are recognizable. In other designs for Scotch grain whisky production (Walker, 1988; Panek and Boucher, 1989) the basic principle is the same except in one respect. In the Coffey still, the incoming wash is heated to at least 90°C by passage through the rectifier section in a copper coil, before discharge onto the feed plate of the analyser. In other designs of still, the wash is pre-heated to 90–93° in a separate heat exchanger before discharge onto the top plate of the analyser.

The newer designs show the influence of North American whiskey distillation procedures. However, not only do continuous distilleries in Canada and Kentucky have different nomenclature of the equipment and distillates, and of
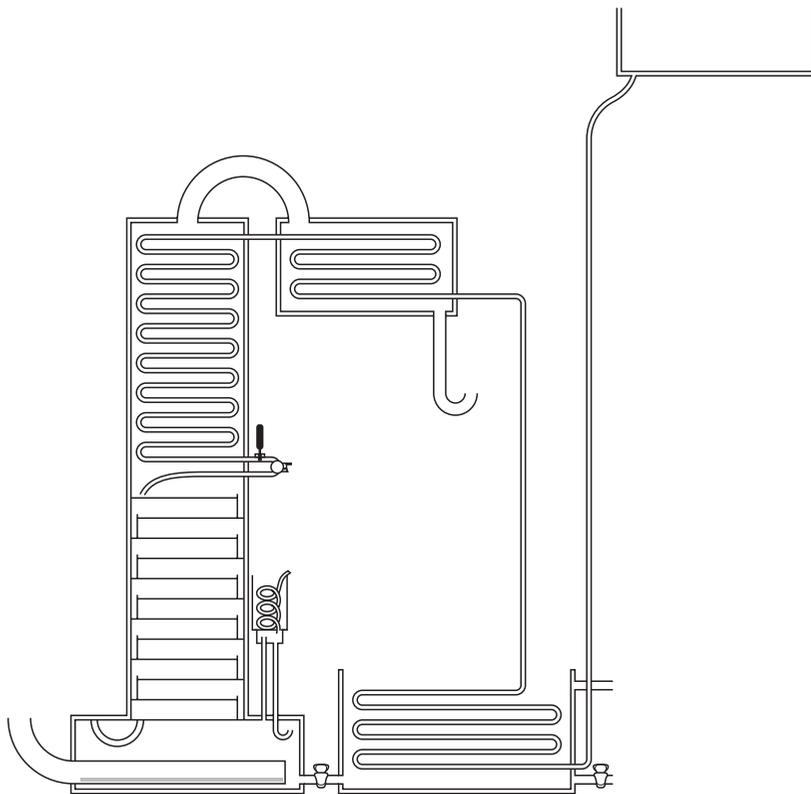
Figure 6.5
Coffey's original design of still (Whitby, 1992).

course different raw materials; their maximum permitted spirit strength of 80 per cent ABV (160° US proof), and normally in the range 60–70 per cent ABV, also gives a higher concentration of flavour congeners in the distillate than would be the case in Scotland. The production of American bourbon and Canadian whiskies has been reviewed recently by Travis (1998) and Wright (1998) respectively, as well as featuring prominently in the review by Panek and Boucher (1989).

In Figure 6.4 only seven plates are shown in the analyser and nine in the rectifier (ten, counting the top condenser), far short of the number actually required. Usually 35–40 are fitted to each column of a Coffey still, but up to 60 are required in a rectifier lacking the reflux effect of the wash coil of a Coffey still. Another simplification is the diagrammatic layout of the wash coils, which are usually in one horizontal plane above each perforated plate as shown in Figure 6.6, but in a very large still could be two superimposed horizontal coils in the space above each plate. Also, in most distilleries the bottom product from the rectifier does not return directly to the top of the analyser but is collected in a hot feints tank from where it is drawn as required
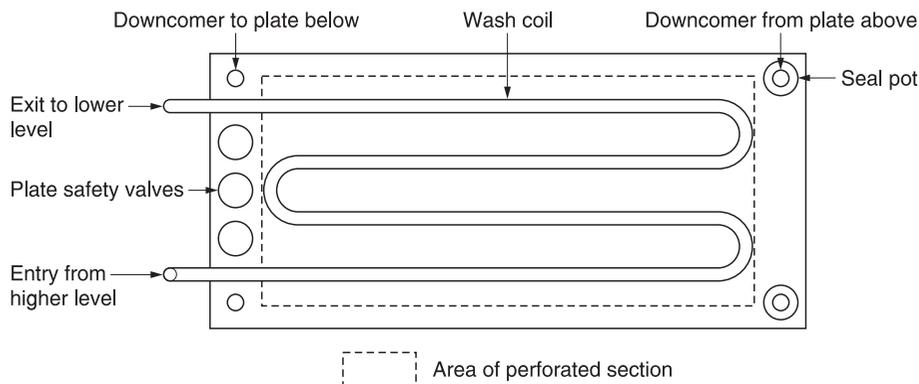
Figure 6.6
Plan view of a rectifier plate and its wash coil in a Coffey still.

to the top of the analyser. This is useful to even out the flow of hot feints, which must be recycled at constant temperature and flow rate. The actual layout is explained in a later part of this chapter dealing with the recycling of all feints streams of the still system. For a similar reason, the top product of the feints still is collected as cold feints before recycling.

Different sized perforations are required in the plates of the two columns: larger holes are necessary in the analyser to prevent blockage by grain solids and yeast (Figure 6.7). For obvious reasons, in a distillery operating with 'grains-in' fermentations the holes in the analyser plates must be larger than for distilleries using partially clarified wort, so a particular distillery is effectively committed to one system. Even so, in addition to the pressure and vacuum relief valves fitted to the tops of the two still columns, the individual plates are fitted with a row of safety valves to protect against the effects of blockage.

Although perforated copper plates have always been used in Coffey stills, originally the frame was constructed of baulks of wood and was therefore rectangular. When it became the custom to construct entirely of copper the rectangular shape was retained, but the more modern designs of stainless steel stills are normally circular in cross-section. Stainless steel stills have a longer working life, since corrosion of the structure is negligible in comparison with copper, but sacrificial copper, usually as copper turnings, must be inserted to react with and remove sulphur compounds at sensitive points. Copper is certainly required in the vicinity of the spirit plate of the rectifier.

Also, it is convenient to fit copper mesh to the vapour pipes at the top of a stainless steel analyser to remove volatile sulphur compounds from the spirit vapour, and to remove entrained wash droplets, as well as acting as a flame arrester. The distance between the still plates, usually 0.4–0.5 m, should be sufficient to avoid problems from normal frothing of wash on the analyser plates. The feed plate at the top of the analyser is the most likely place for frothing, and a particularly frothy wash could contaminate the lower levels of
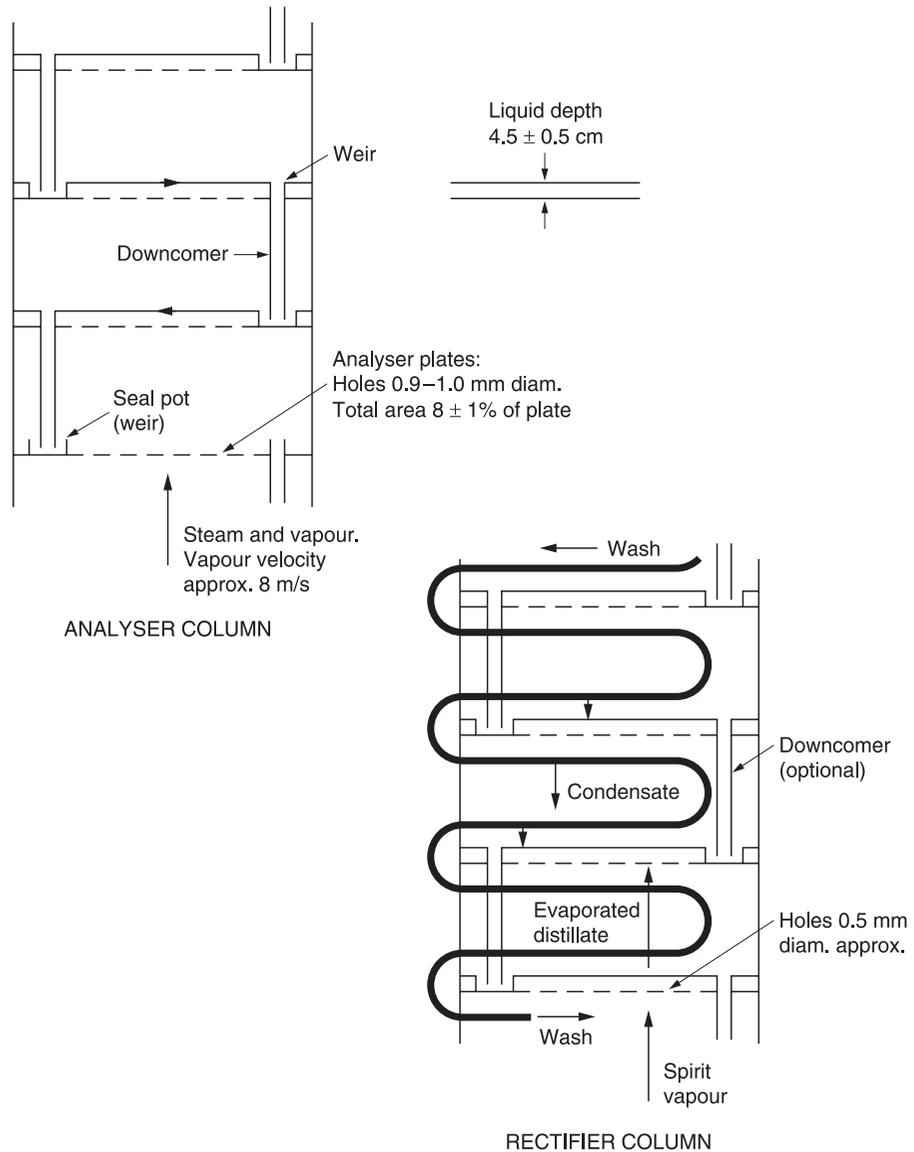
Figure 6.7
Detail of analyser and rectifier plates of a Coffey still.

the rectifier with wash and cause off-odours in the spirit. Such transfer can be prevented by a cyclone in the hot spirit vapour line to remove droplets of wash. Although surface-active components of the malt, cereal or yeast are also possible causes of frothing, entrained CO_2 is certainly important, but can be largely eliminated by sufficiently vigorous agitation in the wash charger.

The amounts of ethanol at the various stages of the still system are shown in Table 6.2. Wash, usually 7.5–8.5 per cent ABV and 30–34°C, is pumped through the coil at the designed rate, with fine adjustment to maintain constant temperature in the wash coil at the level of the spirit plate. A temperature sensor at this point (X in Figure 6.4) controls the rate of the wash pump automatically. Constant temperature at this stage is believed to be more important for spirit quality than constant flow rate, but ideally, with a stable temperature of incoming wash the flow rate is also constant. Alternatively,

Table 6.2
Mass balance of continuous grain whisky distillation

Analyser column					
	UNITS IN			UNITS OUT	
	Mass	Ethanol		Mass	Ethanol
Wash	100	7.5	Spent wash	103	0
Steam	12	0	Hot spirit vapour	20	9.4
Cold feints recycle	1	0.9	TOTAL	123	9.4
Hot feints recycle	10	1.0			
TOTAL	123	9.4			
Rectifier column					
	UNITS IN			UNITS OUT	
	Mass	Ethanol		Mass	Ethanol
Hot spirit vapour	20	9.4	Spirit	8	7.5
			Feints	2	0.9
			(Condenser vent)	<0.1	<0.1
			Hot feints recycle	10	1.0
			TOTAL	20	9.4
Fusel oil still					
	UNITS IN			UNITS OUT	
	Mass	Ethanol		Mass	Ethanol
From rectifier	2	0.9	Cold feints	1	0.9
			Fusel oil product	1	0

Strictly, this is not a *mass* balance since the units refer to alcohol by volume, the measurements normally used in operating the process. However, all units of ethanol are in the correct proportions.

and certainly in continuous stills without a wash coil, the feed to the top plate of the analyser is kept to a constant temperature within the range 90–92°C to control the system.

Wash is heated to 90–92°C as it passes through the length of the wash coil in the rectifier column, and is discharged onto the top of the analyser – usually into a tray or trough that overflows to flood the top plate. Wash passes to the next lower level through a downcomer pipe, projecting approximately 5 cm above the level of the upper plate to maintain that depth of liquid over the entire surface of the plate, to create the necessary liquid–vapour contact at each level (Figure 6.7). It is essential that each plate is absolutely flat and level to prevent shallow areas, which would dry out, causing at least a scorched flavour and perhaps further distortion of the plate. The downcomer reaches almost to the next lower plate and is protected by a circular 5-cm high weir (seal pot), maintaining sufficient depth of liquid to prevent escape of steam or vapour up the downcomer pipes.

Owing to the two-column design of the still, when the descending liquid phase in the stripping section (rectifier column) reaches the bottom plate as 'hot feints', 10–15 per cent ABV it must be pumped up, usually via the hot feints tank, to the feed plate of the analyser. Its flow rate must be synchronized with the flow of wash in order to maintain a constant alcohol concentration at the feed plate. This combined charge to the top of the analyser cascades downwards, providing sufficient liquid to cover all plates of the column, and volatile components are evaporated by the rising vapour from steam supplied at the designed rate for the still. Water and non-volatile liquid and solid components of the wash are removed at the base of the analyser as spent wash.

Figures 6.4 and 6.6 show that at each level in the column the downcomer pipes are fitted at alternate ends, so that descending wash must flow over the length of each plate and volatiles are stripped from the wash by the steam or hot vapour bubbling upwards through the holes. With the constant rate of steam supply of the designed operating conditions, the rate of upward flow of vapour is sufficient to prevent all but a slight leak of liquid through the holes. In fact, some 'weeping' of liquid through the holes is said to be useful to wash away any accumulation of solids and prevent blockage of the holes.

Heating of the analyser column of a grain whisky distillery is by direct steam injection. In theory, a reboiler would be a more economical use of steam and boiler-feed water, and would reduce the volume of water to be removed from spent wash in the animal feed plant. Unfortunately there are several disadvantages, especially the fouling of the heat exchange surfaces in a reboiler by the wash solids and the adverse effect on flavour by reheating the spent wash. However, modern stills use a thermocompressor as an energy-saving device to provide the steam supply (Watson, 1989). Three sources of heat energy are available in a grain distillery for upgrading: steam flashed off at the end of cooking, hot water from the top condenser, and hot spent wash. The disadvantage of cooker exhaust is that it is only intermittently available, whereas condenser water and spent wash are continuously matched to current operating conditions in the still. Figure 6.8
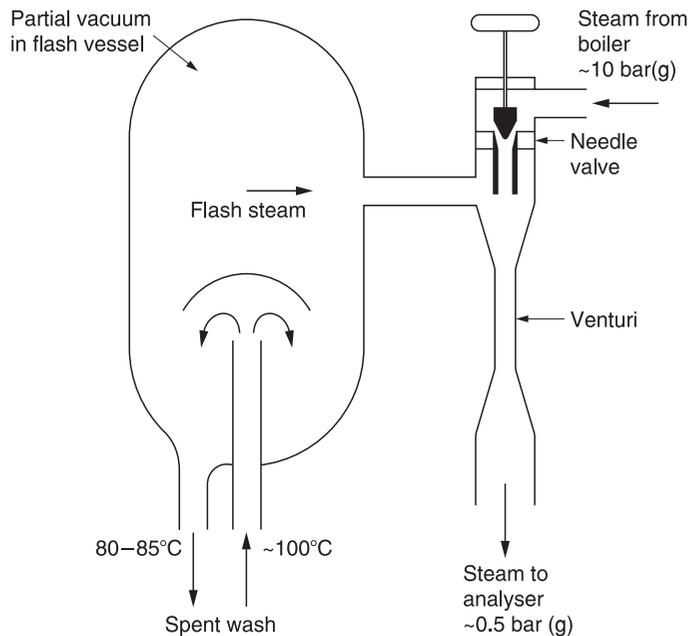
Figure 6.8
Thermocompressor for steam recovery.

shows steam continuously flashed off the hot (approximately 100°C) spent wash in the partial vacuum generated by the venturi being reactivated for recycling to the base of the analyser column. Since a thermocompressor does not heat the spent wash (on the contrary, it is cooled by at least 10°C), there is no risk of a deleterious flavour effect.

The hot vapour rising to the top of the analyser, containing all of the volatile components stripped from the wash, flows through the vapour pipes to the base of the rectifier. The spirit vapour contains all of the congeners from the wash that are sufficiently volatile to be evaporated from the top of the analyser. Rising up the rectifier, the different components separate according to their volatility and boiling points. In the temperature gradient of the rectifier column, created partly by the normal kinetics of distillation and partly by the wash in the coil entering the system about 30°C at the top and increasing to at least 90°C as the wash pipe leaves the bottom, the least volatile components condense in the lower section and highly volatile compounds remain in the vapour phase throughout its height.

Since, unlike in pot stills, the external surface of the still columns is well insulated by lagging, there is negligible condensation and reflux on the wall. Reflux is generated by a combination of:

● Condensation of rising hot spirit vapour on the wash coil to provide the liquid flooding each plate of the rectifier

- Condensation of the vapour as it passes through the liquid on each plate
- Reflux from the top condenser.

In a Coffey still, with its reflux from the wash coil, approximately 5 cm of liquid depth would collect naturally on each plate but in most stills that depth is ensured by downcomer pipes projecting 5 cm above each plate in the column.

Unlike the simple still illustrated in Figure 6.2, in a grain whisky still the top condenser does not yield the spirit product; that is drawn off lower down the column. The condensate from the top condenser, although at least 94 per cent ABV, is termed feints, since it does not meet the quality specifications of spirit. Most is recycled to the rectifier as reflux, and only a small amount is drawn off as 'cold feints', or 'top feints' (cold, since it has been cooled in the top condenser). In some distilleries the top section (top plate, in a sense) of the rectifier column functions as the condenser, as in Figure 6.4, in which case all of the condensate is returned to the column. Figure 6.9 shows the alternative system of a separate top condenser. When the top section of the still itself contains the condenser, the wash coil enters at the next lower level. In neither case is the condensing system operated under total reflux. A proportion of the congeners that are more volatile than ethanol is allowed to escape to atmosphere, to prevent their accumulation to unacceptably high levels in the spirit over the duration of the run.

The best quality spirit accumulates several plates down from the top of the rectifier (Figure 6.10), and at the designed steady-state operational conditions of the still is drawn off at that 'spirit plate'. The definition of Scotch whisky limits distillate strength to less than 94.8 per cent by volume, so theoretically the maximum permitted alcohol concentration for spirit used for grain whisky is 94.7 per cent. However, no Scotch grain whisky still is operated to produce spirit above 94.0 per cent ABV, allowing sufficient contribution by congeners to its flavour.

Aeration

It has been known for many years that although beer suffers flavour defects as a result of post-fermentation aeration, distillery wash and spirit can be aerated without adverse flavour effects. Indeed, with the recent concern about levels of ethyl carbamate in distilled spirits it has been suggested that aeration could be beneficial, by eliminating the cyanide precursors of ethyl carbamate. Also, aerated wash is more reactive with the copper surfaces of the stills, thus reducing the amount of sulphury off-flavour.

So in most distilleries air is added to the wash, usually at the pump feeding the wash coil. Throughout the still system, the dissolved oxygen encourages reactions between wash and the copper of the coil, producing soluble copper compounds or complexes that react with and remove sulphur compounds. The solution of copper salts is particularly important at plates 1–3 at the base of the rectifier column, where the hot, aerobic (by air carried in the hot
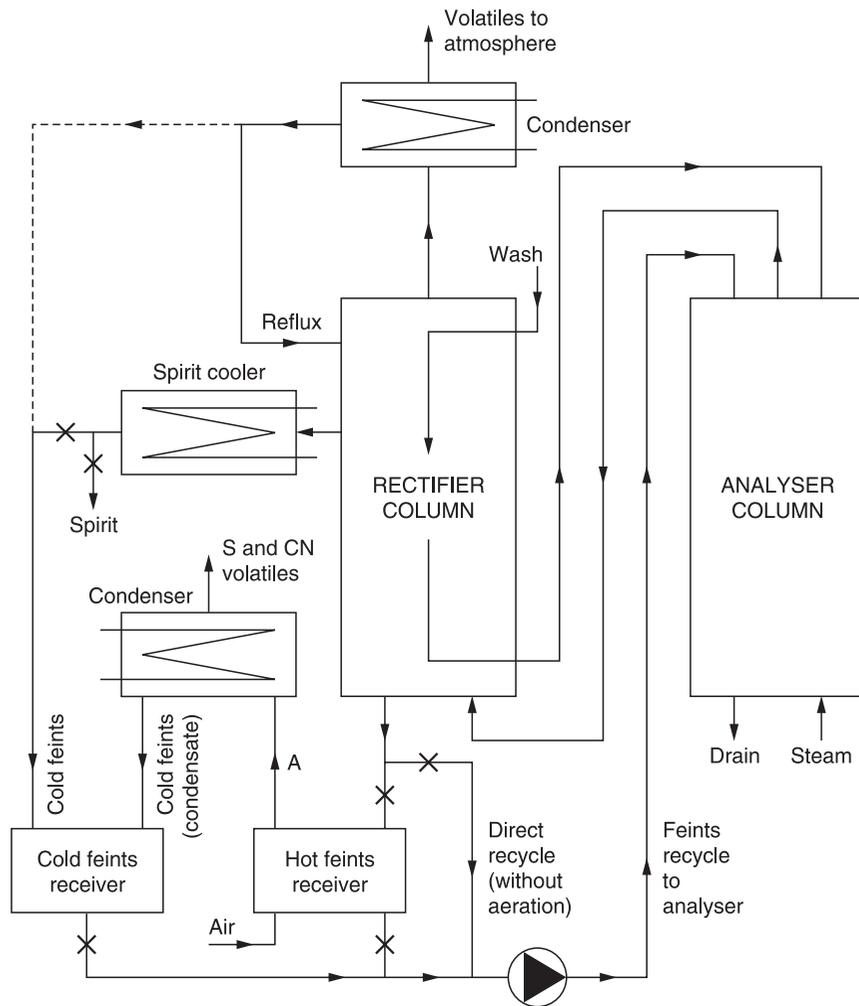
Figure 6.9

Treatment of feints from the rectifier column. Stream A from the hot feints receiver to the hot feints condenser is the alcoholic vapour evaporated by aeration of the hot feints tank. The unidentified streams between the analyser and rectifier columns are the hot wash to the top of the analyser and the hot spirit vapour to the base of the rectifier.

spirit vapour from the feed plate of the analyser), acid (volatile fatty acids) conditions and relatively low concentrations of ethanol and fusel alcohols encourage removal of cyanides associated with ethyl carbamate production. Therefore unwanted congeners such as sulphur compounds and cyanides are removed with the hot feints drawn from the base of the column. Unfortunately, aeration of the wash increases the rate of corrosion of the wash coil and plates of the still and increases the copper content of animal
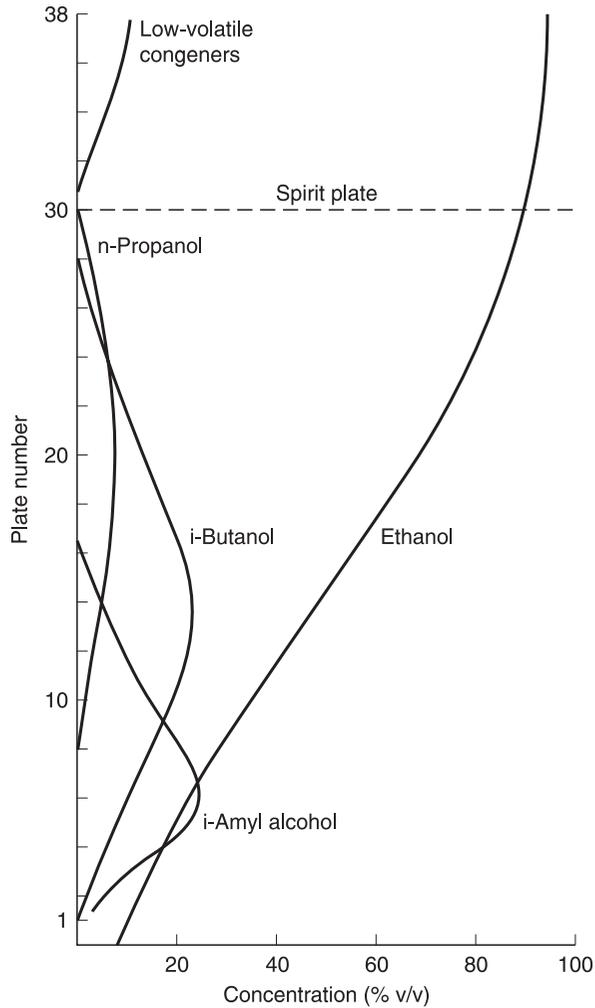
Figure 6.10
Congener profile in the rectifier column (from Whitby, 1992).

feed from spent wash, but these difficulties are tolerated in the quest for quality spirit.

Although it is possible to recirculate the hot feints from the base of the rectifier directly to the top of the analyser column, it may be preferable to aerate first, as shown in Figure 6.9, to evaporate the more volatile copper complexes and break down the precursors of ethyl carbamate. Since the hot feints are at a temperature of at least 90°C a significant amount of ethanol is stripped out by the airflow, but that is condensed, cooled and recycled to the analyser with cold feints. During aeration at 90°C, however, there is no possibility of further solution of oxygen.

Removal of feints

Butanols, n-propanol and iso-amyl alcohol are withdrawn continuously, at a rate equal to their distillation from the wash, from several different levels in the lower section of the rectifier (the region of the maximum concentrations of the principal higher alcohols) to maintain constant conditions in the still. A typical profile of the principal higher alcohols in grain whisky distilling is shown in Figure 6.10. The collected fusel oil is distilled to recover ethanol (Figure 6.11). Although UK Customs & Excise permits up to 8.6 per cent ethanol in fusel oil for sale, that amount represents a wasteful loss from the system and it is usually possible to reduce the ethanol content of fusel oil to 1 per cent or less.
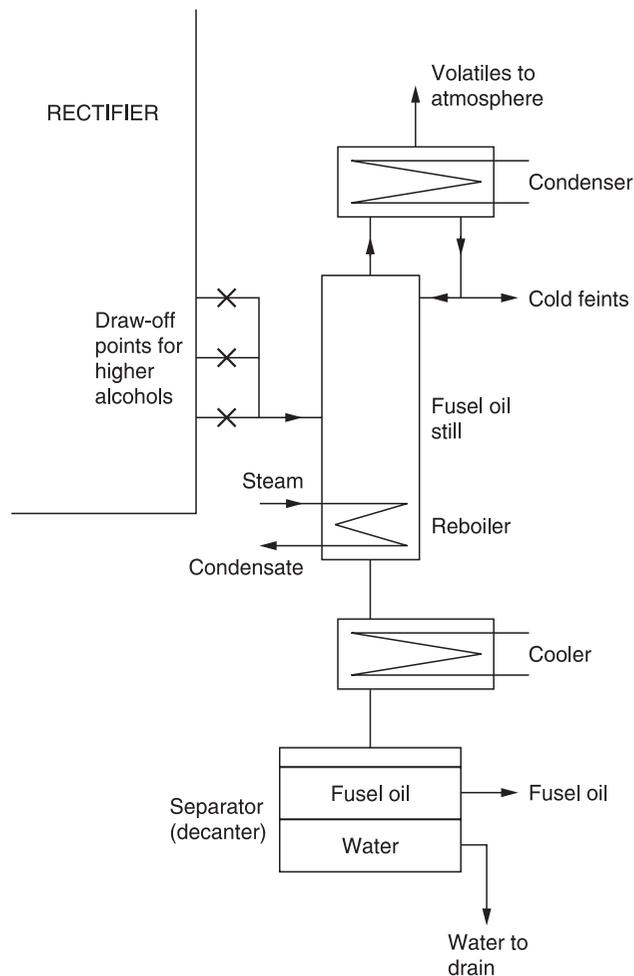
Figure 6.11
Fusel oil still and decanter.

The fusel oil still is a continuous still in its own right, and it too must operate under stable conditions as far as possible – i.e. constant flow rate, temperature and composition of feed. Since the still is of narrow cross-section (it is unlikely to be more than 0.5 m in diameter), perforated plates alone would have insufficient area and reflux capability so efficient separation is achieved by packing with stainless steel or ceramic rings. Since flavour is irrelevant at this stage there is no need for copper in the system, and the whole structure would now be constructed of stainless steel. The fusel oil still has an entirely liquid feed and is therefore operated with a (partial) reboiler, so the fusel oil still is very similar to the simple single-column continuous still shown in Figure 6.2. Non-vaporized bottom product proceeds to the cooling and separation stages. Fusel oil, i-amyl alcohol in particular, is only poorly soluble in water, and can be collected as the surface layer in the separator (decanter) tank. The lower water layer is of sufficiently low organic content to be discharged to drain. As with any continuous still, part of the condensed top product must be recycled as reflux, but the remainder is returned to the main stills via the cold feints tank to recover the ethanol. Incidentally, in a neutral spirit distillery the amount of fusel oil to be removed from the system is greater than in grain spirit production, so a larger multi-plate column would probably be justified.

Although not a common situation, it is possible to operate a grain whisky still without the complication of a fusel oil still. In that situation, a proportion of the liquid on the plate at the level of maximum i-amyl alcohol concentration is drawn off and cooled by addition of water. The water also causes separation of the water and fusel oil phases; fusel oil is decanted off and the ethanol-rich water phase is returned to a lower plate of the column.

Fusel oil contains up to 95 per cent v/v iso-amyl alcohol, and small amounts of ethanol, propanol and butanols. Various other metabolic products of fermentation are present in trace amounts (Walker, 1988). Although its removal obviously has a direct influence on flavour, there is also the indirect benefit that high levels of iso-amyl alcohol inhibit the solution of copper, and therefore restrict the copper-catalysed breakdown of ethyl carbamate precursors and other unwanted compounds. Also, high levels of iso-amyl alcohol in the lower section of the still inhibit the rectification of ethanol. Figure 6.12 shows the effect of accumulation of iso-amyl alcohol in the base of the still: the amount of ethanol condensing in the part below plate 10 is substantially reduced. Figure 6.12A is copied from the lower section of Figure 6.10; Figure 6.12B shows that an increase in the concentration of iso-amyl alcohol over the lower plates of the rectifier suppresses the condensation of ethanol. Although the profiles of both ethanol and iso-amyl alcohol appear to be normal above plate 11, the overall operation of the still is less efficient because plates 1–10 are not involved to the same extent in rectification of the spirit. Therefore, in two ways removal of iso-amyl alcohol contributes to a better quality of spirit.
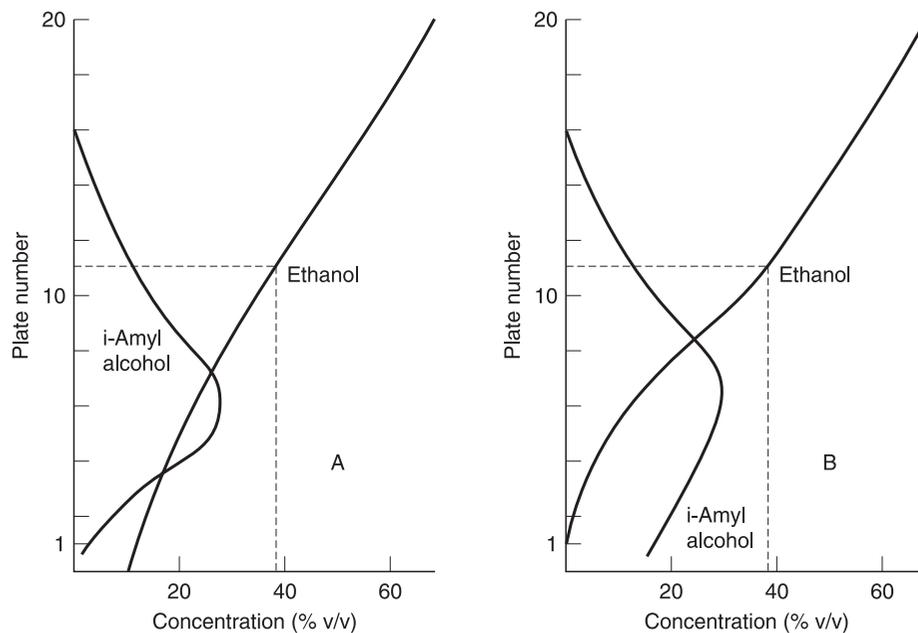
Figure 6.12
Effect of i-amyl alcohol concentration on fractionation of ethanol: A, normal operation in the lower section of the still; B, restricted fractionation of ethanol by unusually high i-amyl alcohol concentration.

Stop and start procedures

Continuous stills should be operated for as long as possible, but accumulation of wash solids on the analyser plates requires eventual stoppage for cleaning. Oxidation and reactivation of internal copper surfaces also occur while the still is open to the air. Since a substantial amount of alcohol is in circulation in the system at any one time it is important to prevent any loss during the close-down procedure. This is done in the following way:

1. The wash coil supply is changed from wash to water at the same temperature.
2. From then on, the quality of spirit must be checked at frequent intervals. It will be acceptable for some time and then begin to deteriorate. At that stage the flow is transferred to the cold feints tank, but at the same time the composition of the bottom product of the analyser must be checked as the amount of wash material decreases. When sufficiently free of solids, it is run to drain rather than to the spent grains plant.
3. When no more ethanol can be collected, as cold feints, the steam supply is turned off.
4. The contents of the rectifier column are then drained into the hot feints tank, and stored to start the next run.
5. Finally, the water supply to the condensers and coolers is turned off.

Similarly, the start-up routine should avoid any loss of the previously recovered alcohol. Different distilleries have their individual methods, but the following is a possible start-up system:

1. The still is heated to its working temperature with a water flow through the wash coil, running to drain from a valve at the bottom of the rectifier.
2. Simultaneously, feints (originally hot feints) stored from the previous distillation is pumped at the normal flow rate for hot wash to the top of the analyser. From there it provides feed for the analyser column and, evaporated by the steam supply, hot spirit vapour to heat the rectifier wash coil. Although the cold feints tank will also be fairly full at this stage, because of its high strength only the normal small proportion can be bled into the hot feints to supplement its alcohol content.
3. When the temperature in the wash coil is steady the water flow in the coil can be replaced by wash, and when the first wash appears at the bottom drain it is closed to direct the flow to the top of the analyser column.
4. The hot feints feed onto the analyser column is adjusted to the same rate as collection; this should be constant during the run.
5. Spirit is collected as cold feints until of acceptable quality for maturation.
6. Owing to its high alcohol content, any variation in addition of cold feints into the still creates unstable conditions. So, stored cold feints from the stop and start procedures is run onto the analyser column at a constant rate calculated to empty the cold feints tank over the planned duration of the run. Only a relatively small quantity of new cold feints is collected during the run. Most (in some distilleries all) of the condensate from the top condenser is recycled as reflux to the rectifier plates, but the top product of the fusel oil still must be recycled as cold feints. However, if an excess of cold feints does accumulate it can be recycled in the same way as unacceptable spirit.

In an alternative start-up procedure the water is not drained from the bottom of the wash coil at stage (1) as above, but continues to the feed plate of the analyser, and the stills are heated up with steam against the downflow of that water. When normal working temperature is reached, the wash flow is turned on to replace water in the coil and the feed of supplementary hot and cold feints to the analyser is started. When spent wash rather than water appears at the base of the analyser, the flow is diverted to the feed plant. The distillate at the spirit plate is collected as cold feints until it meets quality specifications, or for a standard time that is known from previous experience to ensure collection of the desired quality of spirit.

Spirit that fails to the meet chemical or nosing tests for quality must be redistilled. Although an unlikely event, on the large-scale operation of a continuous distillation process the quantity would probably be too large for blending with normal feed as cold feints. In that case the still would have to be run only on failed spirit, diluted with warm water to the normal temperature and alcoholic strength of wash. Since spent wash is no longer produced the bottom product of the analyser is run to drain, but otherwise the still operates normally until all the faulty batch has been re-distilled.

Development of flavour

The first section of this chapter described the theory of continuous distillation of a simple solution of ethanol in water. In reality, the feed to the still is a solution in aqueous alcohol of several hundred compounds derived from the cereal, the structure of the yeast and its metabolic products during fermentation (Nykanen and Suomalainen, 1983; Chapter 4). Irrespective of their contribution as congeners to the overall flavour and aroma of the distilled spirit, it is common knowledge in the distilling industry that these compounds can be classified into three types: (A) those more volatile than ethanol; (B) those with approximately the same volatility as ethanol; and (C) those less volatile than ethanol.

Athough volatility is related to the boiling point of the compound, it is also influenced by the concentration of ethanol. The type B congeners are less volatile than ethanol itself at high ethanol concentrations, but become more volatile than ethanol as its concentration falls. In batch distillation the alcoholic strength falls throughout the run, and therefore the relative volatility of type B compounds increases with time (Figure 6.13). In continuous distillation, how-
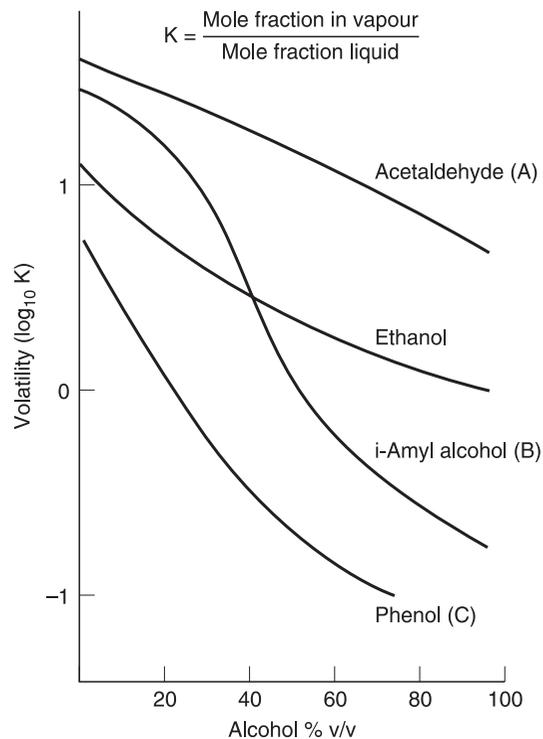

Figure 6.13
Relative volatility of flavour congeners (from Panek and Boucher, 1989).

ever, the ethanol concentration at any specific point in the system should be constant over time, but varies with the position in the still, rising from about 10 per cent at the base of the rectifier to at least 94 per cent at the top.

Figure 6.14 shows the distillation of typical compounds of types A, B and C in the batch process. Line A shows collection of type A compounds principally in the foreshots, and line B (i.e. type B) shows collection throughout the entire distillation process in a similar profile to ethanol, but of course in much smaller amount. Line C1 of Figure 6.14 shows a type C compound distilling at maximum amount about the end of spirit collection, but a different compound (also of type C) could appear late in the spirit cut, increasing throughout the distillation of feints (line C2). These effects can be explained in terms of their relative volatility to ethanol as shown in Figure 6.13. In the continuous process, however, compounds of type C are unlikely to make any contribution to the rectifier column at all. A small amount may be vaporized from the top plate of the analyser and carried over to the rectifier, only to be condensed in the lower
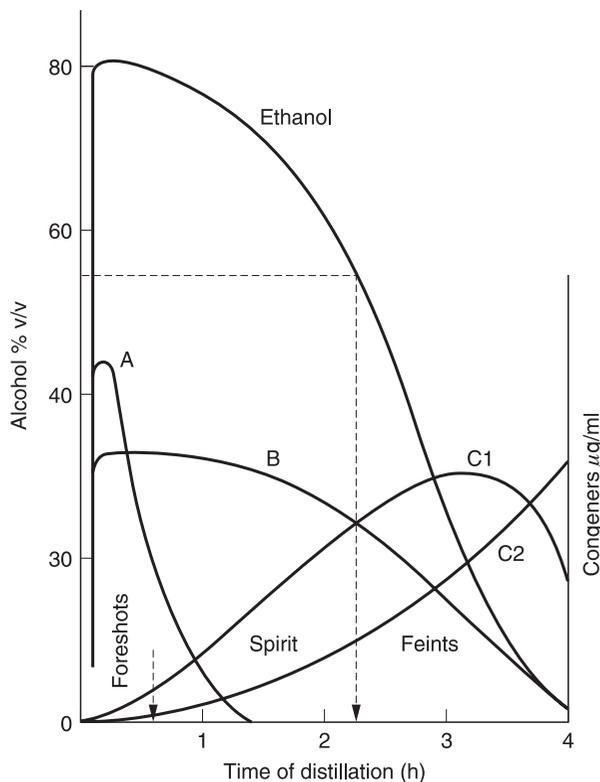

Figure 6.14

Separation of flavour congeners according to volatility during batch distillation (from data provided by United Distillers Ltd). A, congeners that are more volatile than ethanol; B, congeners of similar volatility to ethanol; C, volatiles (C1 and C2) that are less volatile than ethanol.

levels and returned to the analyser in the hot feints. A similar effect occurs throughout the analyser, and the lower volatile material (type C) will gradually cascade down the column to leave eventually in the spent wash.

So the hot spirit vapour flowing into the base of the rectifier column will be rich in type A and B compounds but contain only a small amount of type C, which will soon be returned to the analyser. Type A congeners, being more volatile at all concentrations of ethanol, migrate to the top of the column. According to the operating conditions of the top condenser, individual components of that mixture will be vented to atmosphere, recycled in the reflux to the top plate of the rectifier, or drawn off in the cold feints for recycling to the top of the analyser. The situation with type B congeners is more complex, since the ethanol concentration varies throughout the height of the column. These compounds will stabilize at the level in the column where their volatility and that of ethanol are equal.

Figure 6.10 shows the distribution of the principal alcohols, ethanol, n-propanol, butanols and iso-amyl alcohol, within the rectifier. Note that the spirit plate is not necessarily the level with the highest concentration of ethanol, but higher plates with a higher ethanol concentration are seen (Figure 6.10) to have an unacceptable level of volatile type A congeners – mainly acetaldehyde and any sulphur compounds that have escaped reaction with the copper in the system. The flavour of the spirit is more important than its strength, and eventually the ethanol condensed at higher levels will find its way back to the spirit plate take-off point. Concentrations of all compounds are shown as percentages by volume in Figure 6.10, but congeners are normally measured in mg/l and affect quality at well below 1 per cent. Therefore all of the compounds shown in Figure 6.10 have an important effect on flavour for several plates above and below their disappearance from the graph. Table 6.3 shows typical concentrations of flavour congeners in new grain spirit: all classes of congener are present in much lower concentration than in malt spirit.

Table 6.3
Comparison of flavour congeners of new-make grain and malt spirit, diluted for cask filling (data provided by United Distillers Ltd)

	Grain spirit	Malt spirit
Ethanol (% by volume)	68	63
Total higher alcohols (ppm)	800	2500
Total esters (ppm)	50	650
Total acids (ppm)	0	100
Total aldehydes (ppm)	0	50

Although iso-amyl alcohol is by far the most abundant of the higher alcohols, all three shown in Figure 6.10 must be continuously drawn off at constant rate, otherwise an increasing amount would collect in the spirit over the duration of the run. Inspection of Figure 6.10 suggests that at some point between plates 10 and 15, where all three overlap, they could be drawn off together, but in practice it has been found preferable to operate several take-off points as feed for the fusel oil still. For steady-state conditions, these congeners must be drawn off at the same rate as their introduction in the wash, and this is most easily achieved as separate side streams. They can be adjusted independently, but cautiously, to control the flavour of the spirit if necessary. For example, to increase specifically the butanol content of the spirit, it is obvious that less butanol should be drawn off as side stream. In practice, the situation is more complex. If the original removal of propanol is maintained, as the amount of butanol increases the propanol 'bulge' in Figure 6.10 will move upwards, increasing the amount of propanol at the level of the spirit plate. Therefore the rate of removal of propanol to the fusel oil still should be increased to maintain the original propanol content in the spirit. This effect will be enhanced if iso-amyl alcohol is also removed more slowly. As that less volatile compound accumulates at lower levels, it will further encourage the movement of butanol (and propanol) up the column. However, there are practical limits to this type of manipulation, and an earlier discussion of Figure 6.12 drew attention to the destabilizing effect of unusually high levels of i-amyl alcohol.

The earlier section on feedstock for distillation mentioned the importance of a constant alcohol concentration in the wash for stable operation of the still. Variation in alcohol content also affects flavour, since the changes in alcohol strength over the height of the rectifier affect the separation of the congeners of type B volatility (Figure 6.13).

Energy and utilities

A continuous process is inherently more energy-efficient than batch operation (Whitby, 1995), but nevertheless the production of grain spirit is an energy-intensive process. The principal energy requirements are steam for cooking and distillation, and heat for concentration and drying of spent wash as animal feed. There is also a substantial use of electric power in a grain whisky distillery, although only a relatively small proportion of the overall demand is specifically required for the distillation. At least three pumps are required for operation of the stills: the wash pump, hot feints pump and feed pump to the fusel oil still. Depending on the layout of pipework, other process flows may operate by gravity or additional pumps may be required – for example, for transfer of the top product of the fusel oil still or for removal of spent wash. Centrifugal pumps have suitable characteristics for all of these duties, but it is important to be aware of the significance of a net positive suction head in pumping hot alcoholic solutions (Genereaux *et al*., 1984). Recycling of hot

feints in particular (high temperature and high ethanol concentration) needs careful design of the pump and pipework system to prevent cavitation in the pump.

Stable continuous distillation requires a constant rate of supply of steam. Cereal cooking requires a large amount of steam intermittently for short periods, and drawing steam for the cookers directly from the boiler that supplies the analyser column would cause substantial fluctuation in steam pressure and destabilize the still. The use of separate boilers for cookers and stills is an expensive solution, and it is more economical to use a constant flow of steam from the main boiler to superheat water in an accumulator pressure vessel, which in turn provides cooker steam as necessary. This ensures a constant flow of the remaining steam to the stills, or to the thermocompressor that recovers heat energy for the stills (Figure 6.8). Two separate heating systems are required for the complete distillation system (analyser and fusel oil still), but the reboiler of the fusel oil still is a light duty in comparison with the steam requirements of the analyser.

A simple system of one analyser, one rectifier and one fusel-oil still requires five separate cooling units in total:

1. A spirit cooler, for the hot alcohol from the spirit plate
2. A top condenser (cold feints condenser) above the rectifier
3. A hot feints condenser, to recover ethanol from aeration of the hot feints
4. The heads condenser of the fusel oil still
5. The fusel oil cooler.

However, a larger installation does not require multiplication of all of these facilities.

Unless a mains supply of drinking water quality is used for all purposes, the water supply for a grain distillery requires careful consideration. Three different grades of water quality may be used. The criteria for condenser cooling water are a low supply temperature and low levels of the hardness salts, which could cause problems by scale formation in heat exchangers. Provided routine maintenance is adequate to insure against leaks, other chemical, microbiological and physical properties are irrelevant. A proportion of the water used for mashing, dilution of wash and start-up and close-down procedures ultimately becomes incorporated in the spirit and must be free from flavour taints, but since it is sterilized in the course of the cooking and distillation processes it does not require the microbiological quality of drinking water; only the flavour quality is mportant. Water for steam raising is also included in this category, and therefore boiler water-treatment compounds must be carefully chosen to avoid flavour problems from the steam injected into the cooker vessel and analyser column. Only the dilution water used at cask filling will persist untreated to be consumed with the final whisky, and therefore has an absolute requirement for a drinking-water standard of microbiological purity. However, a drinking-water supply could legally contain levels of calcium and iron salts that are unacceptable in whisky spirit, and these ions must be removed – usually by ion exchange.

Acknowledgements

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Chapter 7

Maturation and blending

John Conner, Ken Reid and Frances Jack

Introduction

In the production of Scotch whisky many factors are known to influence the final quality of the product, including the water, barley type, extent of peating, still type and distillation conditions. The most important contributor, however, is undoubtedly the oak container in which maturation takes place. During the time that the spirit spends in this container, major changes occur in its chemical composition. The pungent, feinty aromas of the new distillate transform into the typical mellow characteristics of a mature whisky. The colour of the spirit also changes, from virtually clear to golden brown.

Historically the origins of maturation are obscure, with the majority of whisky distilled in the eighteenth and nineteenth centuries probably drunk unmatured. However, the making of whisky was traditionally a seasonal process and therefore involved a period of storage, and this storage would inevitably have used wooden casks, the nature of which depended on the availability of suitable timber. In the USA, where there were large reserves of white oak, storage in new casks was the norm. In Scotland suitable supplies were limited, and second-hand containers such as old sherry casks or brandy barrels were used. With time the benefits of this storage were realized, and a period of maturation became an accepted part of whisky production and was incorporated into the legal definitions of whisky that exist throughout the world.

Outwardly the maturation process appears simple. Casks are filled with spirit and set aside in a warehouse to mature. However, a wide range of variables can influence the quality of the final product. The type of cask used, its method of manufacture and even the climatic conditions during storage all affect the maturation process, and are described in detail in this

chapter. When selecting and controlling maturation variables, the traditions and product expectations for the particular whisky have to be considered. American bourbon and rye whiskies are matured in new charred-oak casks, while whiskies produced in Scotland, Ireland and Canada are matured in oak casks previously used for the maturation of bourbon, or for the fermentation and shipment of sherry (Booth *et al.*, 1989). Consequently, while maturation of a Scotch malt whisky in a new charred-oak cask may produce a well-matured whisky, it may not be readily identifiable as Scotch (Clyne *et al.*, 1993).

This chapter aims to give a detailed overview of the current technical understanding of the maturation process and indicate the main variables that influence product quality. Current control of the maturation process is achieved by the careful selection and sourcing of casks and their re-use. This 'wood policy' varies from company to company, and even within a company for individual products, and is used to ensure a continued supply of quality and diverse whiskies to the blender, who can then assemble the finished products.

Most bottled whiskies are blended from different production batches and cask types. However, blended whisky generally refers to a combination of mature whiskies from several different distilleries. The reasons for blending whisky, the major considerations in formulating a blend and the practicalities of blend manufacture are also covered in this chapter.

Finally, the authors' expertise has been gained from working within the Scotch whisky industry and for this reason the chapter focuses mostly on the maturation and blending of Scotch whisky. The production of other whiskies around the world uses a similar range of cask types to that used in Scotland. The exception is American straight whiskies, most notably bourbon, which are matured in new charred-oak casks. Again the process of blending is similar throughout the world, with 'light bodied' whiskies distilled at high ethanol concentration in column stills being combined with 'heavier bodied' whiskies produced in pot stills or distilled to a lower ethanol concentrations in column stills. Consequently, the basic principles described can be widely applied to all whiskies.

Cooperage oak wood

A considerable proportion of the production costs of Scotch whisky is due to investment in cooperage oak. The majority of this is in the form of ex-bourbon barrels imported from the USA and, to a lesser extent, sherry butts imported from Spain. These wooden containers are constructed using the heartwood of several species of white oak that are grown in the forests of North America and Western Europe.

Today the majority of ex-bourbon barrels are imported from the USA as standing barrels (180 l). The practice of disassembling barrels for transportation is in decline. These stave 'shooks' were commonly re-built to form a hogshead, using a greater number of staves and new plain oak ends to give a larger capacity (250 l). The other traditional source of cooperage timber is

Spain, in the form of sherry butts (500 l). These are much sought after due to their excellent reputation for maturing whisky and for their extended lifespan compared to that of a typical American oak container. The Spanish sherry industry, however, can only supply a fraction of the cooperage oak wood required by Scotland, and therefore the Scotch whisky industry will continue to look to the USA for its main supply of cooperage wood.

In the past, many types of wood have been used for the production of tight cooperage containers. However, most wood types either contribute undesirable flavours to the maturing alcoholic beverage or have porosity problems that render them unsuitable for the prolonged storage of liquid. Only a limited number of oak species are completely suited to the production of casks for wine and spirit maturation.

Tight cooperage production from European oak uses two wood species preferentially, namely *Quercus robur* and *Quercus sessilis*, both of which are widely grown throughout the continent (Singleton, 1974). There is a large degree of similarity between the two species, and indeed *Q. sessilis* has been considered as a variety of *Q. robur*. For the production of Spanish sherry casks the European oak, predominantly *Q. robur*, is harvested mainly from the regions of Galicia, Asturias, Cantabria and Pais Vasco in Northern Spain. White oak wood imported from the USA is also used to produce sherry casks, and is generally the preferred wood of the sherry maker.

The list of American oak species used for tight cooperage is, however, considerably larger than that used in Europe, and includes *Q. alba*, *Q. bicolor*, *Q. muehlenbergii*, *Q. stellata*, *Q. macrocarpa*, *Q. lyrata* and *Q. durandii*. *Q. alba* accounts for approximately 45 per cent of the timber used. The main areas of bourbon barrel production in the USA are Kentucky and Missouri. Barrels produced in these areas would probably contain *Q. alba*, *Q. bicolor* and *Q. macrocarpa*, with small amounts of *Q. lyrata* (Singleton, 1974).

Structure of wood

The structural features of certain oaks that make them ideal for tight cooperage are their medullary rays and tyloses. Medullary rays are thin, flat structures that lie along the radius of the tree trunk from its centre to the bark. They are much harder than the structure of the rest of the tree, and are considered to be impervious. In most trees the medullary rays are only one cell wide, but in oaks and some other hardwoods they can be several cells wide (multiseriate). In oak species, these unusually large rays represent between 19 and 32 per cent of the wood volume. In *Quercus alba*, the most common species of oak wood used in American tight cooperage, these rays represent 28 per cent of the wood volume and contribute significantly towards the strength and flexibility of the wood (Schahinger and Rankin, 1992).

White oak also contains conductive vessel elements or tubes that can be as large as 0.3 mm in diameter in the spring portion of each annual ring. The growth of tyloses, which are formed by the ballooning of the cell walls during

the conversion of sapwood into heartwood, seals the channels of the vessels and consequently stops the cask from leaking at the ends. Tylose formation only occurs in approximately twenty species of oak (Schahinger and Rankin, 1992).

Wood tissue is composed of cellular and intercellular material. The cell wall structure is constructed from the macromolecular components cellulose, hemicellulose and lignin, whereas the intercellular region consists mainly of lignin. In addition to the main polymeric fraction of the wood, low molecular weight components (extractives) are present in relatively small amounts. However these are not an integral part of the cell wall structure.

Cellulose is the most abundant component of wood, accounting for approximately one-half of the total content. It can be described as a linear polymer with a uniform chain structure that is composed of anhydroglucopyranose units. These units are bound by β(1-4)-glycosidic linkages formed by the elimination of one molecule of water between hydroxyl groups at C1 and C4 of two glucose units. Hydrogen bonding between hydroxyl groups on adjacent cellulose molecules results in the formation of fibrils, which go together to make the cell wall layers and consequently produce a superstructure for the remaining wood components to build upon (Fengel and Wegener, 1983).

In addition to cellulose, the cell walls contain other polysaccharides known as hemicelluloses that act as a matrix for the cellulose superstructure (Parham and Gray, 1984). These compounds are branched heteropolymers that are much shorter than the cellulose molecules, having a degree of polymerization of approximately 200.**28 Hemicellulose consists mainly of sugar components that can be divided into groups such as pentoses, hexoses, hexuronic acids and deoxy-hexoses. The main chain (backbone) of a hemicellulose molecule can consist of one or more sugar units, and attached to this chain are other sugar components (side groups) such as 4-0-methylglucuronic acid and galactose (Fengel and Wegener, 1984). Oak wood hemicellulose is mainly xylose based (xylans), and accounts for 15–30 per cent of the dry weight. Its backbone consists of β-D-xylopyranose units, which are linked by β(1-4)-glycosidic bonds. Approximately seven xylose units in every ten are substituted at the C2 or C3 positions with O-acetyl groups. In addition to the acetyl side groups, 4-O-methylglucuronic acid units are attached by α(1-2)-glycosidic bonds at irregular intervals along the chain (Sjostrom, 1981).

The third major component of wood is lignin, which has a highly branched three-dimensional structure of high molecular weight. Lignin is located in both the cell wall and intercellular regions (middle lamella), and serves as a binding agent for the wood cells. Oak wood contains between 15 and 30 per cent lignin, of which 70 per cent or more is located in the cell walls themselves (Parham and Gray, 1984). The complex structure of lignin is built from phenylpropane units that are substituted with hydroxyl and methoxyl groups. In hardwoods such as oak, lignin is formed by the polymerization of the two precursors, coniferyl alcohol and sinapyl alcohol, producing 'guaiacyl-lignin' and 'syringyl-lignin' respectively. However no natural lignin is exclusively composed of these groups. The mixed polymers are formed by a variety of ether and carbon–carbon bonds involving both aromatic rings and side chains

of the sinapyl and coniferyl monomers (Monties, 1992). Chemical bonds have also been demonstrated between lignin and almost all the hemicellulosic constituents, and are thought to exist between lignin and cellulose. It is not known what roles these bonds and the physical associations between constituents play in maintaining the structural integrity of oak wood (Haluk and Irmouli, 1998).

In addition to the main macromolecular cell wall components of wood tissue, oak can also contain up to 12 per cent low molecular weight components. These do not contribute to the superstructure of the wood, but are readily extracted by the maturing spirit and may have a significant influence on flavour development during maturation. The composition of the extract is complex, and varies depending on the species and origin of the tree. The main components of interest for cooperage oak are the hydrolysable tannins and volatile compounds (Masson and Puech, 2000). Hydrolysable tannins are partly responsible for the bitterness and astringency of oak wood, and may play a role as catalysts for oxidation during maturation. A variety of structures based on gallic and ellagic acids have been identified, though these degrade during heat treatment, with the major constituents extracted by maturing spirits being the free acids (Mosedale, 1995).

Oak contains numerous volatile constituents, with around 100 peaks detected by gas chromatography-mass spectrometry (Masson and Puech, 2000). Of interest in the maturation of whisky are organic acids, lactones, norisoprenoids and volatile phenols. The major organic acids are acetic and linolenic acids. Linolenic acid is one of the haze constituents in mature whiskies, and its degradation produces odorous aldehydes and alcohols. Oak lactone is one of the main volatile compounds in oak wood and plays an important role in the aroma of mature whiskies, particularly the *cis* isomer, which has a much lower perception threshold than the *trans* isomer. Norisoprenoid compounds such as 3-oxo-ionol are also detected. Although 3-oxo-ionol has little odour, it may be the precursor for highly aromatic megastigmatrienone isomers. Volatile phenols such as eugenol and vanillin are also present in small quantities, though these may be increased by the thermal degradation of oak.

Cask manufacture

Timber processing

Oak wood logs destined for cask production are initially cut into either stave or heading (for cask ends) lengths, depending upon any defects that may be present on the log. These shorter lengths are then quarter sawn with the cuts being made through the heart of the log, running along its radius. From these quarters flat stave and heading timber can be cut to the appropriate thickness, resulting in a recovery of approximately 50 per cent useable timber from the log. Figure 7.1 illustrates the stave cuts.
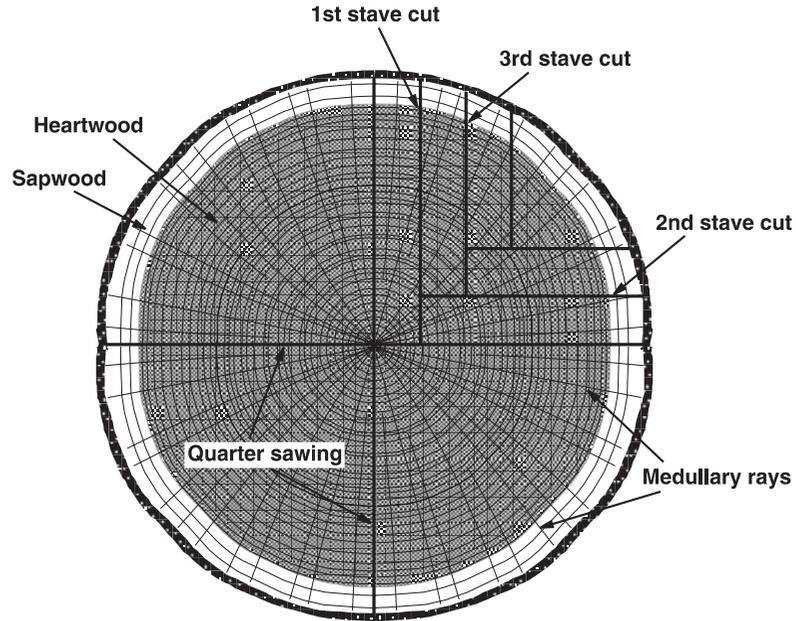
Figure 7.1
Stave cuts in timber processing.

From a log quarter, the first saw cut is removed from one of the flat surfaces parallel to the radius of the tree. The quarter is then turned 90° and the second cut is removed from the other flat surface. This is repeated until the amount of timber left in the quarter is too small to be of any use. The sapwood and any dead heartwood are then removed from the sawn timber to produce the finished stave and heading blanks.

The next stage of the process is the drying or seasoning of the timber. In the USA, all of the oak for the production of bourbon barrels is dried in a drying kiln over a period of approximately one month. This reduces the moisture content of the wood to a workable level of approximately 12 per cent. Throughout the drying process the timber is held under specific temperature and humidity conditions to ensure that the drying procedure is efficient and that wood damage, such as the appearance of splits or cracks in the stave ends, is minimized.

In Spain the seasoning of timber for the production of sherry casks is quite different. The stave and heading blanks are initially air dried in the growing regions of northern Spain for a period of approximately nine months, which reduces the moisture content of the wood to approximately 20 per cent. The timber is then shipped to the warmer sherry-producing regions in the south of Spain, where it is further air seasoned for a period of approximately six to nine months, or until its moisture content is reduced to a workable level of approximately 14–16 per cent.

Bourbon cask construction

In the manufacture of bourbon barrels, the dried, rough-cut stave blanks are initially jointed to produce staves that have smooth angled edges and are slightly wider in the middle compared to the ends. This is essential for forming the desired barrel shape. The assembly of the barrel begins by arranging the straight staves into a circular structure that is closed at one end. This is then steamed for a period of between ten and twenty minutes at a temperature of approximately 95°C to soften the fibres of the wood and enable the staves to be bent. The staves are then drawn into the conventional barrel shape using a windlass, and a temporary iron hoop is used to hold them in place.

The next part of the process is to heat treat the inside of the cask shell. In terms of whisky maturation this is probably the most important stage of barrel production, as it defines the cask's ability to mature spirit. The cask shell, which is still wet from the steaming process, is initially heated to between 230 and 260°C for a period of approximately fifteen minutes. This drives off the surface water and sets the staves in the shape of a barrel (Hankerson, 1947). The shell is then charred, which involves setting the inside on fire and allowing it to burn until the required degree of char has been obtained. The role of the formed char layer during maturation, and the toasted layer that lies beneath, is described in detail in the following section.

Barrel ends are produced by pinning together pieces of oak wood of appropriate thickness using wooden dowels. This is then cut into the circular head and is given a bevelled edge. The ends, which are also charred, are then inserted into the cask shell. Their bevelled edges locate into grooves, known as the croze, cut into each end of the shell. Finally, the hoops are driven on to produce the finished bourbon barrel.

Sherry cask construction

The staves for a 500-l sherry butt are tapered in the same way as in the production of a bourbon barrel, but are longer and thicker. Where the 180-l barrel requires around 30 staves for its construction, the larger butt will contain approximately 50 staves. These are initially raised into a circular structure, as in bourbon barrel production, which is held in shape using temporary hoops. This is then placed over an open fire at approximately 200°C, which both heat treats the wood, turning it brown in colour, and makes it more pliable, allowing it to be slowly pulled into the conventional butt shape using a windlass. No steaming is involved in the construction of a sherry cask, but water is applied to the outside of the staves during heating to prevent cracking. As in the production of a bourbon barrel, the ends are initially prepared by pinning together heading timber using wire dowels. These are then cut into the circular head and inserted into the cask shell to produce the finished sherry butt.

Heat treatment chemistry

Heat treatment has always played an important role in the manufacture of casks for maturing distilled spirits. Two distinct methods of heating are used: toasting is a milder but more prolonged form of heat treatment, while charring is more rapid and involves heating the inner face of the cask with a gas burner until the inner face catches fire and becomes carbonized. Despite these differences, the objectives of the treatments are the same:

- Degradation of wood polymers to yield flavour compounds
- Destruction of resinous or unpleasant aroma compounds present in the wood
- Production of a layer of 'active' carbon on the inner surface of casks (charring only).

Degradation of wood polymers to yield flavour compounds

The main effect of heat treatment is the degradation of wood polymers to form colour and flavour compounds. Most studies in the past have focused on the degradation of lignin to produce aromatic aldehydes and acids, such as vanillin and vanillic acid. However, recent research has highlighted the importance of polysaccharide breakdown in the formation of a number of flavour compounds. Thermal degradation of polysaccharides (cellulose and hemicellulose) produces large quantities of furaldehydes, of which furfural predominates because of the preferential deterioration of the hemicellulose. Furaldehydes themselves have little sensory impact, but their formation is accompanied by that of many other molecules with sweet, caramel and toasted aromas. Maltol and 2-hydroxy-3-methyl-2-cyclopentenone were identified in toasted oak after heating (Nishimura *et al.*, 1983), but comparison of the amounts present with their odour thresholds suggests that their sensory impact may be limited (Cutzach *et al.*, 1997). More recently, furylhydroxymethyl ketone and 2,5-furandicarbaldehyde have been identified in extracts of toasted oak, being formed by the direct pyrolysis of sugars (Cutzach *et al.*, 1999). Extracts also contained 2,3-dihydromaltol, furaneol, and 2,3-dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one, which are the products of the Maillard reaction between amino acids and sugars (Cutzach *et al.*, 1997).

The products of lignin degradation, such as vanillin, syringaldehyde, coniferaldehyde and sinapaldehyde (Nishimura *et al.*, 1983; Reazin, 1983) undergo further oxidation, either in the wood or in the maturing spirit, to give vanillic and syringic acids. Of these, vanillin is of greatest sensory importance on account of its low odour threshold. Analysis of whisky matured in new toasted casks shows that vanillin exceeds its odour threshold within the first six months of maturation (Conner *et al.*, 2001). Synergism has been reported to decrease the odour threshold of mixtures of lignin breakdown products (Maga, 1985). The intensity of heat treatment can affect the levels of aromatic aldehydes and acids generated. Studies using oak chips have shown that toasting temperatures up to approximately 200°C increase levels of aromatic aldehydes and acids (Nishimura *et al.*, 1983). Higher temperatures and char-

ring decrease levels, due to the formation of volatile phenols such as guaiacol and syringol, or carbonization of the aromatics. In casks however, charring increases the levels of lignin breakdown products extracted by the spirit. Although the char layer contains few aromatics, heat penetration to sub-surface layers promotes thermal degradation reactions, increasing aromatic aldehydes and acids, over a depth of 6 mm (Perry *et al.*, 1990). Consequently, with increasing charring time there is increased thermal degradation of wood constituents behind the char layer. Although deeper in the stave, behind a thicker char layer, this does not hinder the extraction of these components because the disruption of the wood structure during charring increases the penetration of the maturing spirit.

Other constituents of wood are affected by heat treatment. Complex ellagitannins, such as vescalagin and castalagin, are greatly reduced by heating and charring. Levels are generally low in distilled spirits (Mosedale, 1995), possibly as a result of conversion to ellagic acid (Hale *et al.*, 1999). Two aroma compounds present in untreated oak are eugenol (clove-like aroma) and oak lactone (coconut). While some studies show that toasting and charring increase levels, others suggest there is little effect (Mosedale, 1995). Although heat treatment may increase the formation of these compounds, their low boiling points result in volatilization from the wood surface.

Heat treatment also increases the level of coloured compounds extracted from the wood by the spirit. However, the exact chemical nature of cask colour has not been elucidated. Heat treatment of model compounds suggests that colour formation is mostly the result of hemicellulose and lignin degradation, with little or no contribution from cellulose. The level of extractable colour generally increases with the intensity of toasting and charring. However, as for the aromatic aldehydes and acids, the char layer contributes little colour.

Destruction of resinous or unpleasant aroma compounds present in the wood

Maturation of wines and spirits in new barrels can result in a rancid, sawdust aroma. The major compound responsible for this aroma has been identified as *trans*-2-nonenal, though other unsaturated aldehydes and ketones such as *trans*-2-octenal and 1-octen-3-one may enhance this character (Chatonnet and Dubourdieu, 1998). The levels present varied greatly between samples of wood and their most likely route of formation is by chemical auto-oxidation of linoleic acid during seasoning. The amount of *trans*-2-nonenal could be markedly decreased, and the off-flavour completely eliminated, by increasing toasting intensity during cask manufacture.

Production of a layer of 'active' carbon on the inner surface of casks

The formation of the 'active' carbon layer on the inner surface of the cask is the result of carbonization of the polymeric constituents. This layer contributes little in the way of colour or extractives to the maturing whisky. It does, however, play an important role in the removal of immature character.

Experiments have shown that it promotes the oxidation of dimethyl sulphide (Fujii *et al.*, 1992) and may reduce the concentration of other sulphur compounds by a combination of adsorption and oxidation (Philp, 1986). Also, the break up of the wood structure near the surface may allow easier penetration by the spirit and increase the extraction of degradation components from sub-surface layers (Mosedale, 1995).

Control of heat treatment

The control of toasting and charring of casks has a major effect on the sensory properties of the matured spirits. The intensity of toasting is generally controlled by time, with light, medium and heavy toasts the result of five to ten, ten to fifteen, and fifteen to twenty minutes' heating respectively (Mosedale and Puech, 1998). Toasting normally uses oak-chip fires maintained by individual coopers, and consequently there are large variations between different cooperages and from one cooper to another. New methods are being investigated to provide a better classification of toasted casks, and these are based on either chromatographic analysis of the volatiles produced during toasting or their assessment using metal oxide-based odour sensors (Chatonnet, 1999). The degree of charring is also controlled by the burn time, with 15 seconds used for a light char, 30 seconds for medium char and 45 seconds for a heavy char (Mosedale and Puech, 1998).

Cask regeneration

Casks that fail to produce a satisfactory maturation, because continued re-use has depleted the level of available extractives, can undergo regeneration. These casks are first de-charred, using a rotating brush or flail system, and the de-charred casks are then re-charred using a gas burner. When casks are re-charred thermal degradation of lignin and polysaccharide occurs, yielding similar flavour compounds to those produced in a new charred cask. However, other constituents of oak are not regenerated, such as oak lactones and hydrolysable tannins. Consequently the balance of wood extractives in regenerated casks is very different from that in a new charred cask.

Re-charring is controlled either manually or by a timer. Reliance solely on time can produce variable results, because casks from different sources have variable moisture and spirit contents. This affects the drying time of the wood before ignition, and as a result the degree of charring and the levels of colour and extractives produced are not controlled. This problem can be overcome by control measures based on the surface temperature of the cask wood, or by the colour of the flame. Initially the cask burns with a blue flame partly fuelled by the spirit volatilizing from the cask wood; when dry the wood ignites and burns with a strong yellow flame (Perry *et al.*, 1990). Charring times of 30 to 40 seconds are generally employed, although longer burn times can generate higher levels of cask extractives and colour.

Scotch whisky maturation

Maturation can be viewed as the specific combination of one type of distillate with any one type of cask leading to the development of a flavour profile relative to time (Philp, 1986). Modern analytical techniques have been used to identify an increasing number of reactions that take place during whisky maturation. Most of these reactions are identified by chemical changes, and their influence on the sensory properties of a mature spirit has not been clarified. This lack of sensory understanding is due to the complex nature of whisky flavour. Overall flavour is the result of an interaction of a large number of different aromas, some of which have yet to be identified. Frequently wood or spirit treatments change more than one compound (or group of compounds), and this can compromise the modelling, and therefore the prediction, of the sensory impact of a single aroma compound.

Another drawback is that the research has been fragmented, with different researchers looking at different products in different parts of the world. As the number of reactions increases, maturation can no longer be thought of as a homogeneous process with the same reactions occurring irrespective of the cask type. Most probably maturation is a mixture of different reactions, and the nature and extent of each is determined by the type of cask used. Consequently, this description of maturation is in two sections; the first part outlines the different reactions that have been identified, and the second relates these reactions to different cask types.

Maturation reactions

The reactions that occur during maturation can be separated into additive and subtractive activities:

- Additive activity includes reactions that introduce or form new aroma compounds
- Subtractive activity includes reactions that remove or alter constituents of new-make spirit.

The main example of additive activity during maturation is extraction of cask-derived congeners. These can originate from the unprocessed heartwood, the thermal degradation of wood polymers during cask manufacture, and carry-over from previous use of the cask. These extractives can also be supplemented by the hydrolysis of wood constituents during maturation, and interactions between wood and distillate components that form new aroma compounds. Subtractive reactions may involve the removal of constituents by physical processes such as evaporation, adsorption/degradation by the charred surface of the cask, and chemical degradation reactions such as oxidation and the masking of distillate aromas either directly or through changes in the whisky matrix.

Additive activity

Cask charring generates high levels of colour and extractives in the cask wood. A large proportion of these are extracted during the first use of the casks, though the actual amount will depend on variables such as the length of the maturation period and the warehousing conditions. In subsequent use further extraction of these thermal degradation products occurs, and this is supplemented by the breakdown of lignin and hemicellulose polymers in the wood by the combined actions of oxidation and hydrolysis. Two mechanisms have been proposed for the degradation of lignin during maturation (Reazin, 1981; Puech, 1984). The first involves extraction of an ethanol–lignin complex by the spirit, which breaks down to form coniferyl and sinapyl alcohols. These alcohols are then oxidized into conifer- and sinapaldehydes, with further oxidation to vanillin and syringaldehyde respectively. The second mechanism involves similar reactions, which take place in the wood to produce aromatic aldehydes that are later extracted by the spirit.

Degradation of hemicellulose under these conditions has not been fully investigated, but may give rise to sugars such as xylose and glucose rather than the furan compounds produced during toasting and charring. The level of sugars obtained, however, does not reach the level required to impart sweetness to the maturing spirit.

There are also a number of aroma compounds present in oak wood that are not formed by the degradation of structural polymers. Of these, both oak lactone (coconut aroma) and eugenol (spicy, clove aroma) have been identified in mature spirits. The sensory importance of oak lactone is complicated by the fact that two diastereoisomers are present in oak, each with a different sensory impact. The relative amounts of these depend on oak origin and its pre-treatment. The highest concentrations are present in American oak, in which the *cis* isomer predominates. Grain whisky matured for four years in new, toasted American oak casks has been found to contain *cis* oak lactone at a concentration twenty times greater than its odour threshold (Conner *et al.*, 2001). On its own, this level of *cis* oak lactone has a pronounced coconut aroma, which was not detected in the grain whisky matured in the new charred cask. Although it is undoubtedly an important aroma compound, other aroma compounds present in the cask wood must modify its sensory impact. With cask re-use the level of this compound extracted into the spirit decreases, with a corresponding reduction in its sensory impact. Re-charring casks produces only a small increase in oak lactone levels. The level of eugenol in grain whisky matured in new, toasted American oak casks is around its odour threshold level after four years. Consequently its sensory impact is a lot lower, but cannot be discounted as it may play some role in modifying the sensory impact of other aroma compounds such as oak lactone.

Another group of constituents extracted from oak during maturation are the hydrolysable tannins. These include gallic and ellagic acids and the various complex combinations of these acids with sugars, which are known as gallo-

and ellagitannins (e.g. vescalagin and castalagin). These compounds are generally non-volatile and have no aroma, but may play a role in modifying the mouth-feel and taste of mature whisky. They may also be important as oxidative catalysts in the removal of sulphides from the spirit, and so contribute indirectly to flavour changes during maturation (See Chemical degradation, below). It should be noted that in many older studies Folin-Denis and Folin Ciocalteau reagents were used to determine the level of tannins in whisky. This method more accurately measures total phenolics, which would also include any lignin breakdown products extracted from the cask wood. Hydrolysable tannins gradually deplete with repeated cask use. Although re-charring casks may give increased levels of ellagic acid, it has little effect on the levels of gallic acid.

Another potential source of aroma compounds in refill casks is from the spirit or wine matured in a previous fill. In-drink by the cask during maturation results in the retention of some components of the distillate by the cask wood, and these are then released during subsequent maturations. This effect is most obvious in casks that are used to mature malt and then grain distillates, with the grain spirit acquiring some of the characteristics (such as peatiness) of the malt spirit. It is also possible to identify congeners from sherry and bourbon in ex-sherry and ex-bourbon casks, but it is not known if these constituents make a significant contribution to the distinctive flavour profile obtained from such casks.

After a number of fills, heartwood constituents and thermal degradation products have all but been depleted. The extraction of wood components then relies on a combination of hydrolysis and oxidation reactions. However, the proportion of lignin in the wood that is degraded by these reactions is relatively small (Conner *et al.*, 1993). With repeated use, susceptible lignin in the inner surface of the wood becomes depleted and the locus of degradation retreats further into the stave (Conner *et al.*, 1995). This combination of depletion and migration from deeper in the stave wood slows the rate of extraction and consequently maturation. Eventually a point is reached where the cask fails to produce any sensory improvement, and it is then termed 'exhausted' (Philp, 1989).

Subtractive activity

Changes in distillate character during maturation may be the result of the loss or suppression of aroma compounds. This may involve:

- Evaporation of low boiling point compounds through the cask
- Adsorption/degradation by the charred surface of the cask
- Chemical reactions resulting in a less volatile product or one with different sensory characteristics
- Masking of immature characters either by sensory interaction or by physical changes in the whisky matrix.

Evaporation

Evaporation of volatile compounds through the cask surface occurs during the course of maturation. For a model whisky, the rate of evaporation ranged from 32 per cent of the total present in the spirit for acetaldehyde to 5 per cent for *iso*-amyl alcohols and 1 per cent for ethyl hexanoate and acetic acid (Hasuo and Yoshizawa, 1986). Evaporation is thought to be the main route for the loss of dimethylsulphide (Fujii *et al*., 1992) and dihydro-2-methyl-3(2H)-thiophene (Nishimura and Matsuyama, 1989). Although evaporation occurs from all casks, it is not known how factors such as porosity and stave thickness affect its rate, and in turn spirit quality. Different warehouse conditions (temperature, humidity and the airflow round the cask) will also affect the evaporation rate and again mature quality. As evaporation progresses the level of spirit in the cask decreases, creating an air space. The increased headspace may provide a larger pool of air to replenish the dissolved oxygen in the spirit that is used up in oxidation reactions during maturation.

Adsorption/degradation by char

The 'active' carbon layer on the inner surface of the cask plays an important role in the removal of immature character from the maturing spirit. Experiments have shown that it promotes the oxidation of dimethyl sulphide (Fujii *et al*., 1992) and may reduce the concentration of other sulphur compounds by a combination of adsorption and oxidation (Philp, 1986). Two mechanisms are possible for these reductions. It may be that char preferentially adsorbs these compounds, or chemical degradation may play the major role. Fujii *et al*. (1992) showed that approximately half the dimethyl sulphide in model solutions was oxidized in the presence of char, but was unable to identify the route by which the remainder was lost. Another potential role of char is in retaining both cask extractives and wine/spirit congeners from previous fills. Although fresh char contains few components, after maturation it contains higher concentrations of wood and wine/spirit components than the sub-surface layers. The char may therefore act as an important reservoir for congeners that are released during the subsequent use of the cask.

Chemical degradation

Chemical reactions that alter distillate components include oxidation and acetal formation. Examples of oxidation include the formation of acetaldehyde and acetic acid from ethanol (Reazin, 1981), and the formation of dimethyl sulfoxide from dimethylsulfide (Fujii *et al*., 1992). The breakdown of sulphur compounds may also be enhanced by cask extractives, particularly hydrolysable tannins such as gallic and ellagic acids. Interaction between these tannins, dissolved oxygen and copper ions produces active oxygen and peroxide, which degrade sulphur compounds (Philp, 1986). The dynamics of this reaction change during maturation. Copper in the spirit is an important promoter of this reaction, and is adsorbed by the cask during maturation (Muller and McEwan, 1998). The availability of oxygen may also depend on cask factors such as porosity, evaporation rate and the air space within the cask. The

situation is further complicated by a recent study (McPhail *et al.*, 1999) that identified these cask extractives as important antioxidants in mature spirits with the ability to adsorb free radicals. Therefore, the role of these compounds and the mechanisms of oxidation in maturation need further clarification. If oxidation reactions are initiated by active oxygen and peroxide, then hydrolysable tannins will act as promoters. If, however, it is initiated by free radicals, then these compounds will act as inhibitors!

Acetal/aldehyde equilibria are established for most aldehydes, and are important in whisky aroma. Aldehydes frequently have sour and pungent odours, whereas acetals are pleasant and fruity (Perry, 1986). The equilibrium between free aldehyde, hemi- acetal and acetal is affected by spirit pH (Perry, 1986), and hence is partly influenced by cask type. During maturation the concentration of esters generally increases due to the esterification of free acids by ethanol. A large part of this is due to the formation of ethyl acetate from acetic acid, either extracted from the cask wood or the product of ethanol oxidation (Reazin, 1981). Trans-esterification reactions are also thought to occur, which in the presence of the large excess of ethanol favours the formation of ethyl esters.

Masking

The immature character of a spirit may be masked in a number of ways. The first is a direct sensory interaction, where the presence of strong wood aromas lessens the impact of sulphury or feinty characters. Less dominant wood aromas may also interact by enhancing the perception of positive distillate characters. However, the nature and extent of this type of interaction has not been studied owing to the difficulties in creating realistic models of whisky aroma.

Masking may also occur through changes in the whisky matrix that reduce the volatility of distillate components. The reduction in pH during maturation, which may be cask-dependent, affects the ionization state of weak bases and, consequently, their volatility (Delahunty *et al.*, 1993). Decreases in pH had the greatest effect on pyridines, due to their pKa values, greatly reducing their perceived aroma in whisky.

It has also been known for some years that wood maturation of spirits produces physicochemical changes in the liquid that are detectable by differential scanning calorimetry (Nishimura *et al.*, 1983), small angle light scattering (Aishima *et al.*, 1992) and mass spectrometric analysis of liquid clusters (Furusawa *et al.*, 1990). Recent research has shown that ethanol and water do not form a homogeneous mixture over the whole compositional range (D'Angelo *et al.*, 1994). At spirit concentrations, ethanol exists as a microemulsion in water. Whiskies consist mostly of ethanol and water, with flavour-active components maintained in the aqueous emulsion by ethanol (Conner *et al.*, 1998). The aggregation of ethanol is affected by the presence of wood extractives, which increases the solubility of aroma compounds and consequently reduces their release into the headspace (Conner *et al.*, 1999). This effect, and the physicochemical changes, is consistent with an increase in large ethanol polymer hydrates in wood-matured spirits, which have a

greater capacity for solubilizing aroma compounds. The wood extractives, particularly water-soluble components such as gallic acid, sugars and ionic components, compete with ethanol for water for hydration. This reduces ethanol solubility, and consequently increases the amount of ethanol available to solubilize other congeners such as esters (Piggott *et al.*, 1992). Therefore, this interaction is greatest in casks that generate high levels of extractives.

Cask types

The major cask types used by the Scotch whisky industry are ex-sherry, ex-bourbon, refill and regenerated casks. A similar range of casks is used for Irish, Canadian and Japanese whiskies. A variety of other casks can be used, particularly for finishing spirits. These impart distinctive sensory characteristics, which are not achievable using the standard cask types. Examples of this are malt whiskies finished in port, Madeira or ale casks. American bourbon and straight whiskies are matured in new charred casks, which by law can only be used once and are then sold on for the maturation of other whiskies throughout the world.

Maturation in new charred casks

Maturation in a new charred cask imparts a high level of colour and extractives that give dominant wood-derived aromas to the mature spirit. These aromas are described as woody, vanilla, coconut, resinous, pine, cedar and sap-like, and are derived from a combination of heartwood constituents and thermal degradation compounds produced during charring. Examples of the flavour-active compounds involved are *cis* oak lactone (heartwood) and vanillin (thermal degradation of lignin), although other as yet unidentified constituents also make significant contributions to the final aroma. The main subtractive reactions are char-mediated adsorption or degradation of immature characters by the fresh, highly active char layer. The high levels of wood aromas mask some distillate characters completely, and the levels of extractives may also be sufficient to change ethanol aggregation, reducing the perception of some distillate character. The strong wood-derived aroma produced by maturation in these casks is an essential character of American straight whiskies but is often considered overpowering in other whiskies, masking too much of the character of the original distillate.

Maturation in ex-sherry casks

This includes all casks purchased, either directly or indirectly, from the Spanish sherry industry. These casks are mainly 500-l butts, with smaller numbers of sherry hogsheads (250 l) and puncheons (558 l). Casks may be made from Spanish or American oak, with large differences in whiskies

matured in the different types of oak. Spanish oak produces whiskies with typical sherry characteristics, combining vanilla, fruity and sweet aromas. Despite the mild heat treatment, spirit matured in Spanish oak casks contains high levels of colour and cask extractives. In contrast, American oak casks produce whiskies that are relatively light and floral, and have a limited ability to reduce the immature characters of new-make spirit. American oak casks are generally more suited to clean, more delicate distillates, whose character would be masked by the heavy extract from a Spanish oak cask.

Sherry casks are generally produced uncharred, with only a light toast. Consequently, natural heartwood constituents and hydrolysis and oxidation products dominate the extract at the expense of thermal degradation products. However, the heartwood of Spanish oak contains much lower levels of oak lactones compared with American oak, imparting different sensory characters to the mature whiskies (Mosedale 1995). The importance of wine contact has not been established. Constituents of sherry have been identified in whisky matured in sherry casks, but their sensory impact, if any, has not been established.

The high level of gallo- and ellagitannins extracted in Spanish oak may enhance the oxidation of distillate components and compensate for the absence of a char layer. The limited ability of American oak sherry casks to reduce the immature characters of new-make spirit may be due to the lower levels of tannins. Finally, the high levels of extractives obtained from first-fill Spanish ex-sherry casks may give changes in ethanol aggregation that lead to masking of distillate characters.

Maturation in ex-bourbon casks

In the production of spirits that are matured in used casks, the role of the wood is to integrate the individual characteristics of the distillate and enhance product complexity (Swan, 1994). Oak wood aromas may be present but are not dominant, and the level of immature characters is considerably reduced. Typical characteristics of maturation in ex-bourbon casks include dry, earthy, scented and vanilla aromas (Shortreed *et al.*, 1979).

Cask extractives are produced by each of the mechanisms described previously. Heartwood constituents and thermal degradation products have been reduced by bourbon maturation, but not completely depleted. Hydrolysis and oxidation of wood components also contribute. Constituents of bourbon may be detected in the spirit, but their sensory importance, if any, is unknown.

The major subtractive activity is char-mediated adsorption or degradation, though the activity of char may be lower than for the first fill. The lower levels of wood aromas may enhance positive distillate characteristics, although prolonged maturation may give strong wood aromas that mask some distillate characters. Likewise the levels of extractives on prolonged maturation may be sufficient to change ethanol aggregation, reducing the perception of some distillate character.

Maturation in refill casks

Repeated use of casks results in decreased yields of wood compounds (Reazin, 1981; Sharp, 1983). In tandem with this decrease in extract is a decrease in the development of mature characteristics, such as smoothness, vanilla and sweet, and less suppression of immature characteristic such as soapy, oily and sulphury (Piggott *et al*., 1993). Comparison of first-fill and refill casks shows that the majority of wood aromas present in the first-fill spirit can still be detected in spirit from refill casks, but at much lower levels (Conner *et al*., 2001). However, the relative amounts may change, and the different balance can give rise to changes in the mature character.

In refill casks the levels of heartwood and thermal degradation constituents are all but depleted, and cask extractives are formed and extracted from the sub-surface layers of the stave. The extraction of constituents from previous fills also occurs, and for grain spirits this may have a significant impact on the character of the mature whisky.

The activity of the char layer in these casks is unknown, but almost certainly reduces with each fill. The lower levels of cask extractives will also limit other degradation reactions. It is possible that the loss of immature character/sulphur compounds by evaporation becomes more important as the other routes of degradation are diminished. Again, the lower levels of extracts suggest that sensory or physicochemical masking is unlikely.

Maturation in regenerated casks

Exhausted casks are regenerated by scraping off the old char layer and re-firing the cask on a gas burner, as previously described. Although this does not regenerate all the constituents of a new cask, the levels of extractives can be higher than in ex-bourbon casks. However, regeneration can give variable results, and has been associated with a higher than normal incidence of off-notes in mature spirits.

The thermal degradation products of lignin and polysaccharides dominate the wood components extracted. Heartwood constituents, such as hydrolysable tannins and oak lactones, have been depleted by previous use, and are not regenerated by re-charring. There is very little information on repeated use of regenerated casks, so it is not known whether regeneration also increases the level of hydrolysable lignin that may be degraded during subsequent maturations. Some constituents from previous fills can survive the regeneration process (e.g. peaty character).

The major subtractive activity of these casks is from the generation of a fresh active char layer, which leads to the enhanced removal of immature characters from the new-make spirit during maturation. The higher levels of breakdown products may give strong wood and toasted aromas, and these might mask some distillate character.

Other maturation variables

Although the cask type is the dominant factor in maturation, other variables can influence the mature qualities of the spirit. The most important of these is maturation time but other factors, such as fill strength and warehouse conditions, also play a role.

Maturation time

Time is an important variable in the maturation of distilled spirits. Maturation periods of ten to twenty years are not uncommon, and although these produce high-quality mature spirits the important reactions in generating these qualities have not been identified. One particular problem in identifying reactions over these timescales is that when modelled in the laboratory, no appreciable activity may be observed within a practical experiment time.

The extraction of colour and wood constituents during maturation has been followed in a large number of studies. In first- and second-fill casks, there is a rapid initial extraction within the first six to twelve months. Thereafter the extraction rate is reduced, although a steady increase in colour and cask constituents is maintained throughout the maturation period. The initial rapid extraction is attributed to the rapid diffusion of free extractives from the cask wood. The steady increase thereafter is due to the release of further concentrations of hydrolysable tannins and lignin breakdown products through a combination of spirit hydrolysis and air oxidation. The evaporation of spirit, which increases the concentration of all non-volatile congeners, may also contribute to this steady increase. In refill casks most free extractives have been depleted, and the initial rapid extraction is generally absent: Extractives generally increase linearly through the maturation period, reflecting the slower rate at which congeners are formed by hydrolysis and oxidation. Consequently increased levels of cask extractives generally accompany prolonged maturation, although this will to a large extent be dependent on cask type. The effect of prolonged maturation on the relative amounts of cask-derived congeners has not been investigated, but increased oxidation of these may occur (e.g. coniferaldehyde oxidized to vanillin), which may enhance the mature character of the spirit.

Colour and cask extractives provide easily measured markers for additive reactions during maturation. No such markers have been followed for subtractive reactions, with the result that their contribution to the sensory properties of old whiskies is not known. Evaporation takes place throughout the maturation period, and in Scotland this is accompanied by a decrease in strength. On prolonged maturation large reductions in spirit strength may occur (down to 50 per cent v/v), and this may affect the solubility of wood and distillate components. Consequently, the concentration of long chain ethyl esters, fats and ethanol lignin may decrease, and the concentration of sugars and hydrolysable tannins increase. Oxidation reactions may vary over the

maturation period. Recent studies have shown that char mediated reductions in dimethyl disulphide occur to a large extent within the first eighteen months of maturation. Adsorption of copper by the cask will reduce the level of active oxidant produced in older spirits. However, the development of rancid character in old brandies is attributed to the oxidation of fatty acids to ketones, so there may be other, as yet unidentified, reactions that are promoted by prolonged maturation. Finally, the increase in the level of extractives and wood aromas with longer maturation could lead to both sensory and physicochemical masking of distillate aromas.

Fill strength

Distillates are generally filled into casks at a constant strength – malt whiskies are diluted normally to between 57 per cent and 70 per cent (v/v), while the maturation strength of grain whiskies may be higher but is generally less than 80 per cent (v/v). Spirit strength influences the extraction and formation of flavour congeners in a maturing whisky. Lower alcoholic strengths favour the extraction of water-soluble wood components, such as hydrolysable tannins, glycerol and sugars (Reazin, 1981). Higher strength distillates extract more ethanol-soluble congeners, such as lactones. Overall, increasing the fill strength reduces the levels of colour, solids and volatile acids developed during maturation (Baldwin and Andreasen, 1974; Reazin, 1983). Some wood/distillate reactions may be dependent upon the presence of water and therefore as spirit strength increases the water concentration will decrease and reaction rates will be slower, resulting in lower levels of congeners in the final product. One notable exception is ester formation, which remains constant over the maturation period and is not affected by fill strength. To control the extraction of wood components, casks are filled with distillates at empirically derived strengths. This strength rarely exceeds 80 per cent (v/v), as strengths above this can lead to the extraction of excessive amounts of wood lipids and ethanol lignins, which give filtration problems before bottling.

Warehousing

The maturation of whisky requires that companies have suitable storage facilities for the large cask inventory this entails. Traditionally, maturing whisky was accommodated in stone-built single- or multi-storey warehouses located beside the distillery. However, with the expansion of production, large, centralized multi-storey warehouses were used to increase storage capacity. During maturation the cask is not an impermeable container, allowing the evaporation of spirit (both ethanol and water) and the ingress of air (oxygen). The loss of a small percentage of spirit (called the angels' share) has long been an accepted part of maturation, and this loss has been found to vary with the environmental conditions in a warehouse (Reid and Ward, 1994). Local, regio-

nal and national differences exist in warehouse temperature and environment, and these can affect both evaporative losses and the rate and progress of maturation.

Warehouse types

The traditional maturation warehouse was a stone-built single- or multi-storey building with slate roof on timber sarking. The bottom storey of the warehouse had a cinder floor, with additional levels having wooden floors. Warehouses generally were constructed without damp courses, and humidity often depended on the surrounding soil type and water table. Casks were stored in 'stows', usually two or three high, sitting on top of one another, with wooden runners between each layer.

Large, centralized multi-storey warehouses have a basic construction of brick walls, concrete floors and insulated aluminium or asbestos roofs. Steel racking with wooden runners allows casks to be tightly packed on their sides in long parallel rows, and racking can extend to up to twelve rows high. More recently some warehouses have dispensed with racking, and casks are stored on their ends on pallets, stacked up to six high, for easy access using forklift trucks.

Environmental conditions and evaporative losses

Differences in construction give rise to important variations in ventilation and insulation. The large roof area of warehouses and their generally poor insulation allow relatively high rates of solar heat transfer through the roof to upper levels. If no natural or forced air circulation is provided, then hot, stagnant air builds up around the upper tiers and there is a sizeable temperature difference between the top and bottom of the warehouse. This effect is most marked in continental America during the summer, where temperatures of 50–60°C can develop on the top floor while temperatures at the bottom are only 18–21°C (US Environmental Protection Agency, 1978). In Scotland, which has a maritime climate heavily influenced by the Gulf Stream, temperature variations are less, with top tiers typically 16–20°C in summer and bottom tiers at 10–15°C. Traditional warehouses generally have much better insulation characteristics, and so do not experience the extreme internal temperature gradients of the multi-storey racked warehouses.

Warehouse temperature and humidity can influence evaporative losses. Within a warehouse there is an inverse relationship between temperature and humidity. Under controlled climatic conditions, temperature and humidity have been shown to affect the relative rates at which ethanol and water are lost. A rise in temperatures increases the evaporation losses of both ethanol and water, while humidity influences the relative rate at which ethanol and water are lost. At high humidity more ethanol than water is lost and the strength is decreased; at low humidity more water than ethanol is lost and the strength increases (Philp, 1989). Application of these results to the ware-

house environment is not straightforward, as conditions can vary on a seasonal, monthly and even daily basis. In Scotland, although the location of a warehouse affects the seasonal and monthly temperatures for maturation, this cannot be directly related to losses, owing to the influence of other factors such as ventilation and insulation.

There is a direct relationship between warehouse humidity and water loss during maturation. For large racked and palletized warehouses there are marked differences between top and bottom tiers. The environment around the bottom tiers is generally stable, with a relatively high humidity. On the top tiers there are marked daily decreases in humidity as the temperature increases, and this translates to higher losses of water from the top tier (Reid and Ward, 1994). Consequently, for equivalent casks maturing the same spirit the alcoholic strength can decrease when matured at the bottom of the warehouse but increase when matured at the top. However, total evaporative losses will be higher from the top tier. Humidity also explains international differences in strength changes. In the USA, the relatively hot and dry climate encourages preferential loss of water vapour relative to ethanol, and consequently strength increases during maturation (Reazin, 1981). In Scotland, the cool, humid environment favours the loss of ethanol over water, and strength decreases during maturation.

There has been little work on the effect of warehouse ventilation during maturation, perhaps owing to difficulties in quantifying this parameter. The rate of evaporation will depend heavily on the airflow around the cask. In a well-ventilated warehouse the concentration of ethanol and water around the cask will be reduced, and this will increase the concentration gradient between the barrel and air, thus increasing evaporation. Individual warehouse management is also influenced by factors such as health and safety regulations, and the labour involved in moving barrels and opening and closing windows. Often in Scotland warehouses do not have any active ventilation systems, and so the exchange of air depends the prevailing weather conditions, how sheltered the warehouse is, and the number of times it is opened on a weekly basis.

Effects on quality

The effect on whisky quality of warehouse parameters such as temperature, temperature cycling, humidity and ventilation is not precisely known. Consequently, present methods of warehouse operation have not been developed by design and calculation; rather, each distiller's operation is for the most part the result of tradition and experience. The chemical effects of temperature are straightforward; higher temperatures increase the rates of extraction and reaction and increase the rate of diffusion. Under controlled conditions, the non-volatile content extracted during maturation significantly increases with temperature (Philp, 1989). In large warehouses, spirit matured on the top tier is generally darker and has a higher non-volatile content than spirit matured on the bottom tier. Temperature cycling is also thought to play a role in this

faster extraction. Changes in the internal temperature and pressure of barrels result in expansion and contraction of the spirit volume, which increases liquid motion in and out the cask wood.

Under controlled climatic conditions there are no consistent differences between Highland and Lowland malt spirits that can be related to temperature or humidity. However, for grain spirits some differences have been noticed: Those matured at a higher temperature have been described as sweeter but less clean, whilst smoother and more pleasant whiskies are produced at lower temperatures (Philp, 1989). This suggests that the influence of warehouse conditions is subtle, and can be masked by cask type and distillate character. Comparisons have shown that the physical and chemical reactions typical of maturation proceed at a greater rate in the warmest (top) tier of a warehouse. However, no optimal temperature has been determined for producing the desired product quality (Reazin, 1981), and the effects of different conditions are averaged out during blending.

Wood policy

Distillers strive to produce a range of unique spirits, with recognizable but distinctive characteristics, from their different distilleries. Consistency in character of these individual whiskies is vital, not only for spirits that will go on to be sold as single malts and grains, but also for those that will be used in blending. Since wood is one of the primary influences in whisky character, and accounts for a considerable proportion of the production costs, it is vital that distillers establish a successful and cost-efficient wood policy to control overall product quality.

An important aspect of wood policy is the introduction of new casks. If whisky quality is to be maintained, it is important to have a healthy influx of new cooperage oak wood into cask stocks. Therefore, on reaching the point of exhaustion casks should be removed from the system and new casks introduced. A proportion of these exhausted casks may be sent for regeneration, but this does not recreate the original state of the casks and consequently the number of casks that can be recycled into the system is limited. Most new casks are ex-bourbon barrels from the USA, with a lesser, but nevertheless important, number of sherry butts from Spain. In the case of the bourbon barrel, the construction and heat treatment of this cask is outside the control of Scotch whisky companies. Consequently, the quality of the purchased cask, which is influenced heavily by the heat treatment it undergoes during construction, is to a large extent unknown. Sherry casks can be made from both American and European oak woods. This variable, in conjunction with the heat treatment applied to the wood during cask construction, has a large impact on the maturation performance of the cask and the character of the spirit it will produce. If casks are purchased directly from the sherry producer without any prior knowledge of their history, then the quality of cask performance will again be unknown. To overcome this problem some Scotch whisky

companies have much more involvement with the casks during sherry production, either through obtaining knowledge of the cask history or by providing specifications for variables such as wood type, sherry type and storage period.

An effective wood policy should ensure full utilization of the maturation potential of a cask. This can be achieved by matching cask types and activities with particular spirit types. Cask management involves matching a new-make spirit, with its particular flavour characteristics, with casks that have the ability to mature the spirit to produce the quality of whisky required for its target products. Casks that begin life maturing one type of spirit may end their life maturing another. For example, a cask displaying a high degree of maturation activity may be capable of maturing a malt whisky spirit that has heavy sensory characteristics. After several re-fillings its activity will have dropped, and it may then be used to mature lighter spirit, such as a grain whisky. Other variables are also important in ensuring the maximum use of a cask. These include the number of times a cask is re-filled, the period of spirit storage, and the warehouse environment in which the cask is stored.

In summary, careful management of cask stocks and their uses will ensure a continued supply of quality and diverse whiskies to the blender, who can then assemble the finished products.

Blending

The *Oxford English Dictionary* defines blending as 'mixing so as to be inseparable and indistinguishable'. Malt whiskies and single grain whiskies are the product of individual distilleries, and as such each has its own unique flavour profile. Blending can involve combining around 20 to 50 different malt whiskies with 2 to 5 grain whiskies to produce a whisky with a more rounded character, in which the individual flavour attributes are less easy to separate out. Blends are central to the Scotch whisky market, outselling malt whiskies at a ratio of around nine bottles of blended whisky to one of malt (UK Food & Drink Report, 2000).

Why blend?

Blending was pioneered by Andrew Usher, in Edinburgh, in the early 1860s. Prior to this Scotch whisky only had a limited appeal. It is hypothesized that this was because the unblended malts available at the time were perceived by many as too strongly flavoured for everyday consumption. However, with the introduction of milder blended whiskies the appeal of Scotch whisky spread, first to England and then throughout the world (The Scotch Whisky Association, 1992).

Today, blending provides the distiller with an opportunity to create a product with unique and recognizable flavour attributes. It also enables product

consistency. Even whiskies produced by the same distillery can vary significantly in sensory character, owing to the influences of a wide range of processing and maturation parameters. Blending a large number of diverse products allows the distiller to achieve continuity in character.

What does blending involve?

In the subsequent description of blending, this aspect of whisky manufacture has been divided into four areas:

1. The flavour specialist
2. Strategy
3. Practicalities of blending
4. Innovation.

Traditionally all four areas would have been the sole responsibility of a company's Master Blender. However, as whisky companies have grown and consolidated, this responsibility is often distributed among a large number of people, the blending team.

The flavour specialist

Even today blending remains more of an art than a science, as whisky companies are understandably unwilling to reveal the secrets of blend formulation. Each blender has his or her own flavour style, often inherited from the past. The word 'style' should be emphasized here. The formulation of a blend is not like following a step-by-step recipe. It is more dynamic, with the component whiskies and their addition levels varying from batch to batch.

When creating a new blend, component malts and grains are selected that complement and enhance one another's flavours. The skill and knowledge of the blender is critical. It takes years of experience to become a successful blender, who knows exactly which whiskies and in what proportion must be used to achieve the desired flavour characteristics (Lang, 1983). Once a blend has been established, yet more skill is required to ensure continuity of its character. There are many variables that must be taken into account. Each whisky in the blend will have been selected for its specific flavour profile, but the quality of each component whisky will vary and the blend formula may need to be adapted slightly to account for such differences in quality. There is also the potential problem that one or more of the component whiskies may become unavailable, perhaps due to closure of a specific distillery. The blender must then call upon his or her knowledge to select suitable alternatives, or manipulate the formula to account for their absence.

The role of malt whiskies in the blend

The role of malt whiskies in the blend is to provide the key flavour character. The large number of potential malts available offers the blender an exciting

array of diverse whiskies that can be used to give 'personality' to the blend (Booth *et al.*, 1989). The blender selects these according to the flavour style of the product, and the quantities of each used depend on the individual flavour profiles. Full-flavoured malts, such as the heavily peated Islay malts, are likely to be used in only small quantities in a blend because their characteristics are such that at high levels they are likely to be too dominant. Other malts, such as the Lowland malts, have less intense flavour characteristics, and can be added at higher levels without a major sensory impact.

In theory, a blended whisky could be formulated using only a few malt whiskies. In practice, however, the number used is generally in the range of 20 to 50 malts. If only a limited number of malts were used, the sensory impact resulting from the sudden unavailability of one of these would be much greater than it would be in a blend containing a wide range of malts. A large number of malts in the blend makes the achievement of blend consistency an easier task.

The role of grain whiskies in the blend

Grain whisky is more lightly flavoured than malt whisky, although again each distillery's spirit has its own distinctive flavour profile (Lang, 1983). The continuous distillation process used in the manufacture of grain whisky makes it more economical to produce than malt whisky. However, the grain whisky component of the blend should in no way be considered as a cheap diluent; it plays an important role in rounding the characteristics of the blend (Booth *et al.*, 1989). The addition of grain whisky imparts sweetness, tones down harsh notes, and can aid in revealing certain flavours in the malts.

Although there are relatively few grain distilleries in Scotland (only eight grain distilleries were in operation in 2002), a blender is unlikely to source all of his or her grain whisky from a single distillery. Around two to five different grain whiskies are used when formulating a blend; again, this minimizes problems of inconsistency should a particular grain whisky suddenly become unavailable.

Ratio of grain to malt whisky

Blended Scotch whisky typically contains more grain whisky than malt. However, there are no strict rules as to the relative percentages. Although grain whisky is the cheaper of the two, a higher inclusion of malt whisky does not necessarily equate to a better quality product (Booth *et al.*, 1989). Finished product quality depends more on factors such as the age of the malts used and the ability of the blender to select and combine the most appropriate malt and grain whiskies (The Scotch Whisky Association, 1992).

Assessment of whiskies to be used in the blend

Blenders have at their disposal the most sensitive instrument for measuring the flavour of a whisky: the nose. However, it take years of experience to be

able quickly to judge the sensory characteristics of a whisky, and even longer to know exactly what these will contribute to the overall flavour profile of the blend.

The judgment of a component whisky is based primarily on its aroma characteristics, although the blender may also elect to taste some or all of the samples. Samples of each whisky are drawn from the casks and a small amount is transferred into a tulip-shaped nosing glass. A watch glass is placed on top, in order to trap the aroma volatile in the headspace above the spirit. Whiskies can be assessed either at full strength or, more commonly, after dilution to around 20 per cent alcohol by volume. Dilution to this level decreases the pungency effect of the ethanol and allows the maximum partitioning of aroma volatile into the headspace, revealing the full aroma characteristics of the whisky.

When assessing a whisky for incorporation into a blend, the blender focuses on the following aspects of its aroma:

- Is the sample free from off-notes?
- Is the sample of the style and type desired?
- Is the sample of appropriate overall quality?

Strategy

As stated earlier, consistency is one of the key factors in a successful blend. However, for a consistent blend to be produced the required component whiskies must be available, in the correct quantities and of the desired quality, every time that that blend is being produced. Although the blend formula is not rigid, and certain malts or grains can be substituted with others if required, it is vital to ensure that appropriate replacements can be obtained. Since many of the whiskies in a blend will have been matured for a number of years, and all of them for at least three years, it is vital to forecast sales of a blend well into the future so that required stocks can be laid down. This is a considerable task, which if not carried out correctly could have major implications on the future of the blend. It is usual for some malts to be obtained from a company's own distilleries. However, to ensure the full spectrum of flavour character in the component whiskies, other malts may need to be purchased from competitors. Future sales are not always easy to predict. For example, recent years have seen an increased demand for deluxe blends, with distillers having to ensure that they have sufficient stocks of more mature whiskies in order to meet these needs.

Practicalities of blending

Each blend contains a large number of individual whiskies, which have been maturing for various periods of time in various locations. The identification, locating and retrieval of these casks in preparation for formulating the blend is not a minor task, and the absence of just one cask could potentially be disas-

trous. Strict inventory management is therefore another essential aspect of blending.

Once the required casks have been gathered together they are drained into stainless steel troughs, which covey the spirit into a blending vat. In the vat the whiskies are mixed thoroughly, either by mechanical agitators or by using compressed air. All of the component whiskies may be combined in the blending vat, though some companies prefer to vat their malts and grains separately, only bringing the two together before bottling (The Scotch Whisky Association, 1992). At the vatting stage the blender assesses the product, giving his or her approval, highlighting any problems and providing recommendation for any further additions that may be required (Booth *et al*., 1989).

Vatting may be followed by a 'marrying period' of around three to six months, where the blend is rested in butts or larger wooden containers. The aim of this period is not of further active maturation. Although these vessels are constructed of wood, their maturation potential has been exhausted through use and re-use over a number of years. Not all companies carry out marrying. However, certain blenders believe that it is an essential step in the optimization of product quality, allowing time for the components of the blend to come together fully, giving the well-rounded sensory characteristics desired. As with many aspects of blending, little or no scientific research has been carried out to date to establish if, when and how such changes occur.

Once the blend has received approval from the blender, it enters the final production stages. Water is added to reduce the alcohol strength of the blend to bottling strength (generally 40 or 43 per cent alcohol by volume). Rough filtration, to remove barrel char etc., is followed by chill filtration. This removes any components in the whisky that might form haze during storage, the primary culprits being long chain fatty acid esters. Caramel is added to provide uniformity of colour, and the blend is then ready for bottling, packing and dispatch.

Innovation

Although many companies have key blends that have remained relatively unchanged over a number of years, innovation is still essential. A number of companies have diversified over recent years. In particular there has been the development of more deluxe blends. These are superior quality products, formulated using only more mature whiskies, typically twelve years old and above. Other growing markets must also be explored. One such market is the increased consumption of whisky with mixers. Only blends that are compatible with a range of typical mixers will be competitive in this growing market.

Blending of whiskies other than Scotch

The preceding description has focused on the blending of Scotch whisky. However, whisky blending is not unique to Scotland.

In some countries the procedure is very similar to that used for Scotch. Blending of Japanese whiskies, for example, follows the same basic principles. However, since there are only a small number of Japanese distilleries they have greater difficulty obtaining the full spectrum of malts required to formulate a blend, and have to make greater attempts to differentiate the spirits being produced by single distilleries through the manipulation of process and maturation parameters (Booth *et al.*, 1989).

Legislation in certain countries permits the addition of other constituents. Neutral spirit can be added, within legislated proportions, to both American and Canadian whiskies. These counties also permit the addition of low levels of specific flavouring agents to certain types of blends. These flavourings may include wines, sherries, rum, brandy, or other types of whiskies.

As well as differences in the blend formula, the blending procedure itself can also differ. For example, two different approaches are used in the blending of Canadian whiskies. The first approach, similar to that of Scotch whisky, involves the blending of mature whiskies. However, the alternative approach is to pre-blend the spirits before they are matured, with the flavouring agents being added immediately prior to bottling (Booth *et al.*, 1989).

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Chapter *8*

Co-products

Bob Pass and Ian Lambert

Introduction

The history of distilling is inseparable from that of agriculture. Surplus cereals were used to produce *uisage beatha* – the 'water of life' – and it was natural to feed the residues to stock; this was so commonplace that it is not surprising that it was not documented in the early days of distilling.

The first recorded evidence appears to be that given in *The First Statistical Account of Scotland* (1790), which refers to the substantial Kilbagie Distillery, Clackmannanshire (Sinclair, 1978):

> . . . previous to the year 1788, the quantity of corn used annually at the distillery at Kilbagie alone amounted to above 60 000 bolls, and the annual quantity of spirits made, to above 3000 tons. The black cattle fed annually were about 7000; swine 2000. The cattle were sold to butchers, who drove them to Edinburgh and Glasgow markets; the swine were killed and cured into bacon and pork for England. The work people employed were nearly 300.

The archaic Scottish boll has been variously described as equivalent to 63.5 kg (*Economist*, 1994), 76.2 kg (Anon, 1869), or 6 bushels (Craig, 1994), this last measure being equivalent to 136 kg of barley (*Economist*, 1994).

Assuming the 3000 tons of spirit were at 70 per cent alcohol, then this was equivalent roughly to 2.3 million litres of pure alcohol. At an assumed yield of 325 l p.a. per tonne (t) of malt, this would need about 7000 t of malt. If a boll is taken to be six bushels, this ties in reasonably well with 60 000 bolls of, presumably, barley (8160 t barley producing about 7000 t malt).

The malt would have produced 7000 t of draff – sufficient to feed the cattle noted, with, presumably, the pot ale being fed to the pigs. The fattened cattle were driven to Edinburgh or Glasgow (50 and 45 km distant respectively).

At the time this interdependence of agriculture and distilling was mirrored in brewing, which had a thriving trade in brewers' spent grains (Donnachie, 1979).

By the end of the nineteenth century the use of residues for animal feed was frequently recorded in Alfred Barnard's epic tour of British Distilleries, originally published in 1887 (Barnard, 1969). Of the 129 Scottish distilleries he visited, draff and/or pot ale feeding to cattle and pigs is mentioned in no fewer than 35. Such enterprises are commonly thought of as relatively small scale, and indeed Duncan *et al*. (1994) quote a reasonably-sized Highland distillery in 1825–26 as using about 20 t malt per annum. Such production would have only produced a moderate 20 t or so of draff, which would readily be consumed by over-wintering ten cattle. However, at the other end of the scale, then (as now) the Lowland distilleries such as Kilbagie were of much greater size. The Caledonian Distillery in Edinburgh required a trainload of draff to be taken away every twelve hours (Barnard, 1969).***31

Barnard conveys the impression of agricultural enterprises thriving on the co-products. Cattle, horses and pigs were fed on both draff and spent wash or pot ale. For example, Port Dundas Distillery in Glasgow fed 400 hundred pigs, described thus: 'Some of them are highly bred animals of great size, and on entering the breeding shed the visitor is surprised to see the wall literally covered with prize-cards'.

At the time, many of the larger Lowland distilleries kept pigs, finding a ready market in the expanding urban conurbations.

The Islands, now considered to be relatively remote, were able to take full advantage of sea transport, leading to the development of thriving distilleries on Islay (which may have originally been illicit but, once established, remained well served by sea communications). Caol Ila, on Islay, shipped draff off the island, taking advantage of the vessel delivering malt and other supplies. (Barnard, 1969).

The links between agriculture and distilling were not always without problems. A famine in 1782 led to a ban on distilling in Argyll to conserve grain, and this was repeated on Islay in 1801 and 1802, with barley being extremely scarce on the island. At the other extreme, an enterprise to raise pigs on draff on the distillery farms of Laphroaig and Ardenistiel and on the tiny islet Texa, (off Islay) came to grief, ending in bankruptcy (Wilson, 1985).

A number of sources have also suggested that the innovation of drying plants is a relatively new phenomenon. Certainly large dark-grain plants, where the residues from both mash tun (draff) and first distillation (pot ale or burnt ale) are dried together, are a recent innovation. Drying has, however, been practised for a considerable time. Barnard (1969) quotes an instance of draff drying at Loch Katrine distillery (closed in 1902; Craig, 1994), with the dried product being sold in bags for cattle feeding at £5.10/ton (equivalent to a highly respectable £224/tonne in today's money!).

By the early years of the twentieth century a number of distilleries were drying pot ale to produce a manure or residue (Royal Commission, 1909); as was noted in the Royal Commission report, the main difficulty was the disposal of the liquid effluent, with pollution of the rivers Spey and Fiddich causing concern.

The use of land to 'purify' pot ale was also considered, and had already been practised for many years at some distilleries (for example at Glenkinchie;

Barnard, 1969). Nowadays, pot ale is regarded as a useful fertilizer (Bucknall *et al.*, 1979).

In 1909, at the time of the Commission Report, pot ale was being sprayed into chimneys (at Convalmore and Benrinnes), and at Benrinnes the chimney was deliberately set on fire weekly! Processes at Knockando and Longmorn were somewhat more sophisticated, with evaporating trays of pot ale producing a dried charred residue.

The consortium plant at Rothes, serving four distilleries, was a much more sophisticated operation. At Rothes a multiple-effect evaporator, with four of five effects acting under partial vacuum, produced a pot ale syrup. The syrup was finally dried in evaporating pans to produce a charred residue. Ground to a powder, this was sold at an average price of £4.15/ton (roughly equivalent to a staggering £187/tonne in today's money, for a fertilizer with, generously, an analysis of 5.44 per cent nitrogen, 10.06 per cent P_2O_5 and 5.53 per cent K_2O**32 (Bingham and Sinclair, 2000; personal communication). Despite the high value of manure, the process was 'far from being self supporting' (Royal Commission, 1909).

Plus ça change

Writing in 1994, Duncan *et al*. noted that:

> By product recovery and waste treatment in malt and grain distilleries remains a singularly unattractive investment ... The factual evidence to date shows that with adequate dilution and dispersion, direct sea disposal offers the most environmentally acceptable way of dealing with distillery wastes.

He was referring to the dilute liquids; the solid residues (draff etc.) have not been the same problem.

Since the Commission's report, expansion of the industry in the twentieth century has resulted in more evaporation and drying of the liquid residues (in the main, pot ale). The inevitable consequence of this development has been an increase in the proportion of draff used in drying plants to absorb the syrup. The market for the evaporated pot ale (pot ale syrup) is relatively limited in size (see below), and therefore increased production of dark grains has been the logical development, with the capacity in the industry now being some 300 000 tonnes p.a.

For the future, it is likely that production and sale of co-products suited to the ruminant feed market will continue to be most important, other uses having a small impact overall.

Production of co-products

Although the biochemistry of malting and fermentation is complex in detail, when considering the co-products it is more useful to contemplate the simplified chemical conversion:

$$\text{Starch} + \text{Water} \rightarrow \text{'Sugars'} \xrightarrow{\text{Fermentation}} \text{Alcohol} + \text{Carbon dioxide} + \text{Heat}$$

$$(C_6H_{10}O_5)_n + nH_2O \rightarrow C_6H_{12}O_6 \rightarrow 2C_2H_5OH + 2CO_2$$

$$\text{M.W } (162)_n + (18)n \rightarrow 180.16 \rightarrow 92.14 + 88.02 \qquad \text{**33}$$
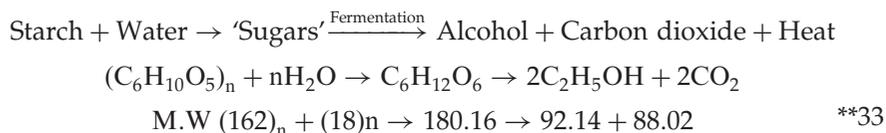
It therefore follows that from the alcohol produced per tonne of malt or cereal mashed, the approximate weight of co-products remaining can be calculated (assuming that the yeast pitching rate is also known).

Annual whisky spirit production is published (Gray, 2000). However, to obtain useful estimates of the volumes of individual products available, knowledge of both co-product production processes and industry structure is required.

Whisky spirit production uses either malted barley as the sole cereal substrate (for malt whisky production; see Chapter 2) or a mixture of unmalted cereal grain together with sufficient malted barley to provide the enzymes necessary to convert the cereal starch (for grain whisky; see Chapter 3). Although for grain whisky production any cereal may be used, current economics dictate that the choice is between wheat and maize.

Apart from the cereal used, there are other important differences between the two processes that can affect the nutritional properties of their respective co-products.

Malt spirit is batch distilled in copper stills from a fermented clear wort or wash. Grain spirit is continuously distilled from a fermented wash that may include the cereal, malt and yeast residues, and the stills may allow 'sacrificial' copper to come into contact with the wash. Details of the respective processes are given below.

Malt whisky co-products

The malt distiller mills dressed malted barley to produce a suitable grist. The soluble sugars and starch are dissolved using repeated hot water extraction, always ensuring that malt enzyme activity is maintained by recycling later, hotter waters. The solubilized starch and sugar liquid (wort) is drained off and fermented by added yeast. The residue remaining in the mash tun (known as (malt) draff) can then be used directly as animal feed or used as a component of barley dark grains (see Figure 8.1).

After a fermentation of typically 48 hours the wash is distilled in the copper wash still. The relatively low pH of the fermented wash (about 4.0) means that some still erosion is inevitable. It is also beneficial, as reactive copper helps remove some of the less desirable sulphur containing compounds (Whitby, 1992). The amount of still erosion will vary with the fermentation time of the wash, design of still, purity of copper used and type of still heating.

The still residue, known as pot ale or, historically, burnt ale, contains yeast, yeast residue, soluble protein and carbohydrates, and a significant but variable quantity of copper (40–140 mg/kg dry matter). With a total solids content of 4.0–4.5 per cent and a high biological oxygen demand (BOD), only a small
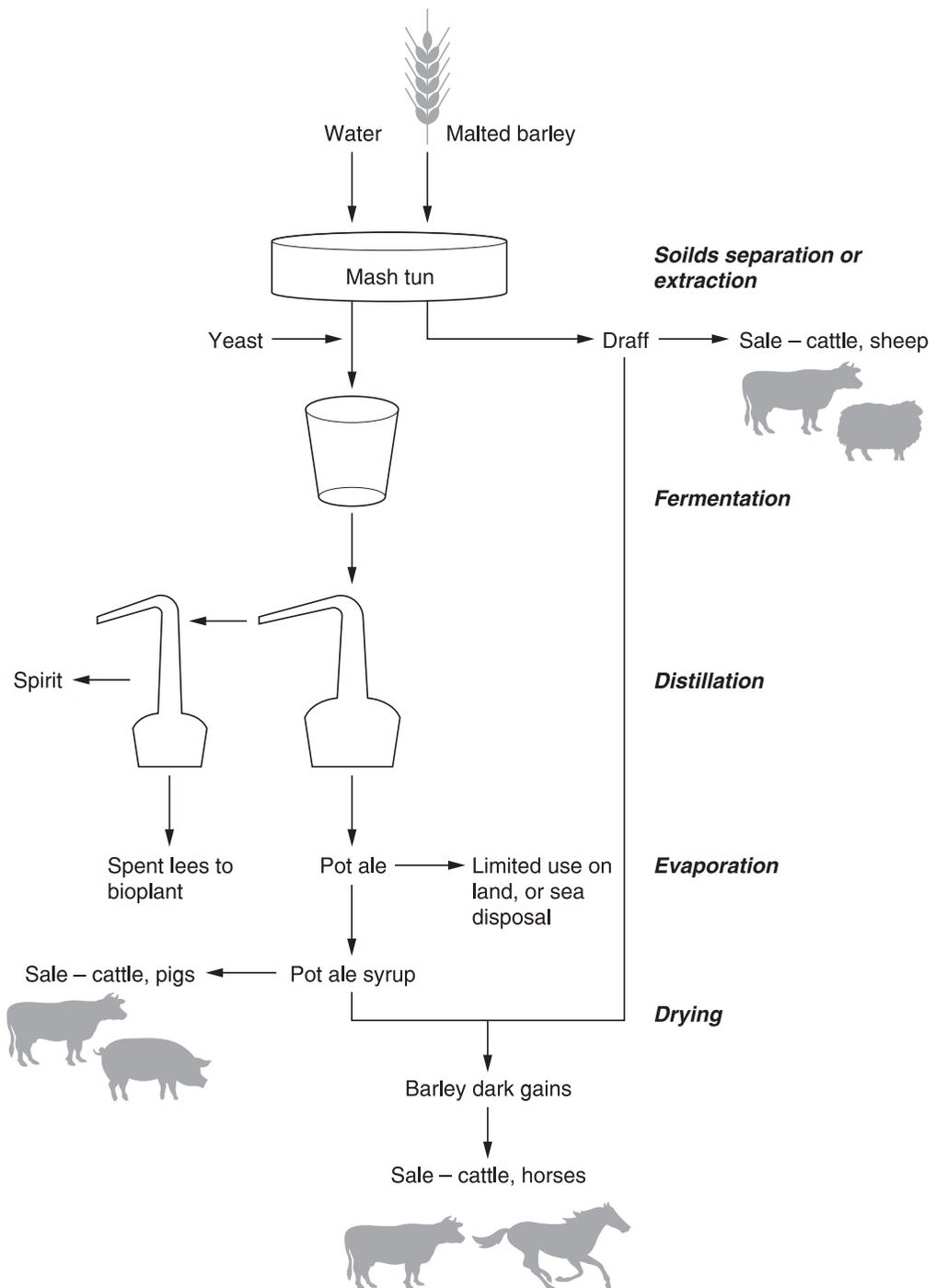
Figure 8.1
Co-products from malt distilleries.

proportion is now used as a fertilizer or as pig feed owing to the large volume produced. Some reaches the sea via long pipelines, but most is further processed to pot ale syrup (PAS) or barley dark grains (BDG).

The pot ale is first evaporated to pot ale syrup. Pot ale syrups have dry matter of between 40–50 per cent, viscosity similar to that of molasses, and vary in colour from mid- to dark brown (depending on the design of the evaporator, evaporation under partial vacuum giving a paler syrup).

The higher dry matter concentration of pot ale syrup allows it to be sold, without further processing, as a cattle or pig feed. As is demonstrated below, the total market for this liquid feed is somewhat limited and therefore most pot ale syrup is blended with draff and then dried to produce barley dark grains.

The low wines from the wash still are then passed on to the spirit still for the second distillation to whisky spirit, and the spirit is filled into oak casks for maturation into malt whisky.

Spent lees remain as the spirit still residue with a BOD of 1500 mg/l and a low pH; this dilute solution of organic acids and alcohols is normally treated by conventional biological effluent treatment.

In the production of barley dark grains, draff is first dewatered in rotary screw presses, and is then mixed with pot ale syrup before being dried, pelleted and cooled.

Grain whisky co-products

Figure 8.2 illustrates the processes involved in the production of grain spirit and associated co-products.

Grain distilleries have a wider range of production techniques than malt distilleries, and the more varied nature of the co-products reflects these differences.

The starch in the cereal must first be solubilized to render it susceptible to the malt enzymes. This is achieved through a combination of grinding and high-pressure and -temperature cooking. The severity and extent of cooking will vary between distilleries and the choice of cereal. The partially liquified cereal must then be cooled before sufficient malted barley is added to convert the starch to fermentable sugars.

Some distilleries then remove the solids before adding yeast to ferment a clarified wort, whereas others ferment all the wash, including cereal and malt residues.

After fermentation the alcohol is distilled in continuously operated distillation columns of varying designs. Most incorporate a facility for allowing chemically reactive copper to come into contact with either the boiling liquid or vapour phases (or both). As in malt distilleries, this contact removes less desirable sulphur-containing compounds and congeners.

Following distillation, the spent wash solids are removed by centrifuges or filter presses. Where possible, the dilute centrate or filtrate is discharged into the sea, with the moist solids being sold following the addition (where neces-
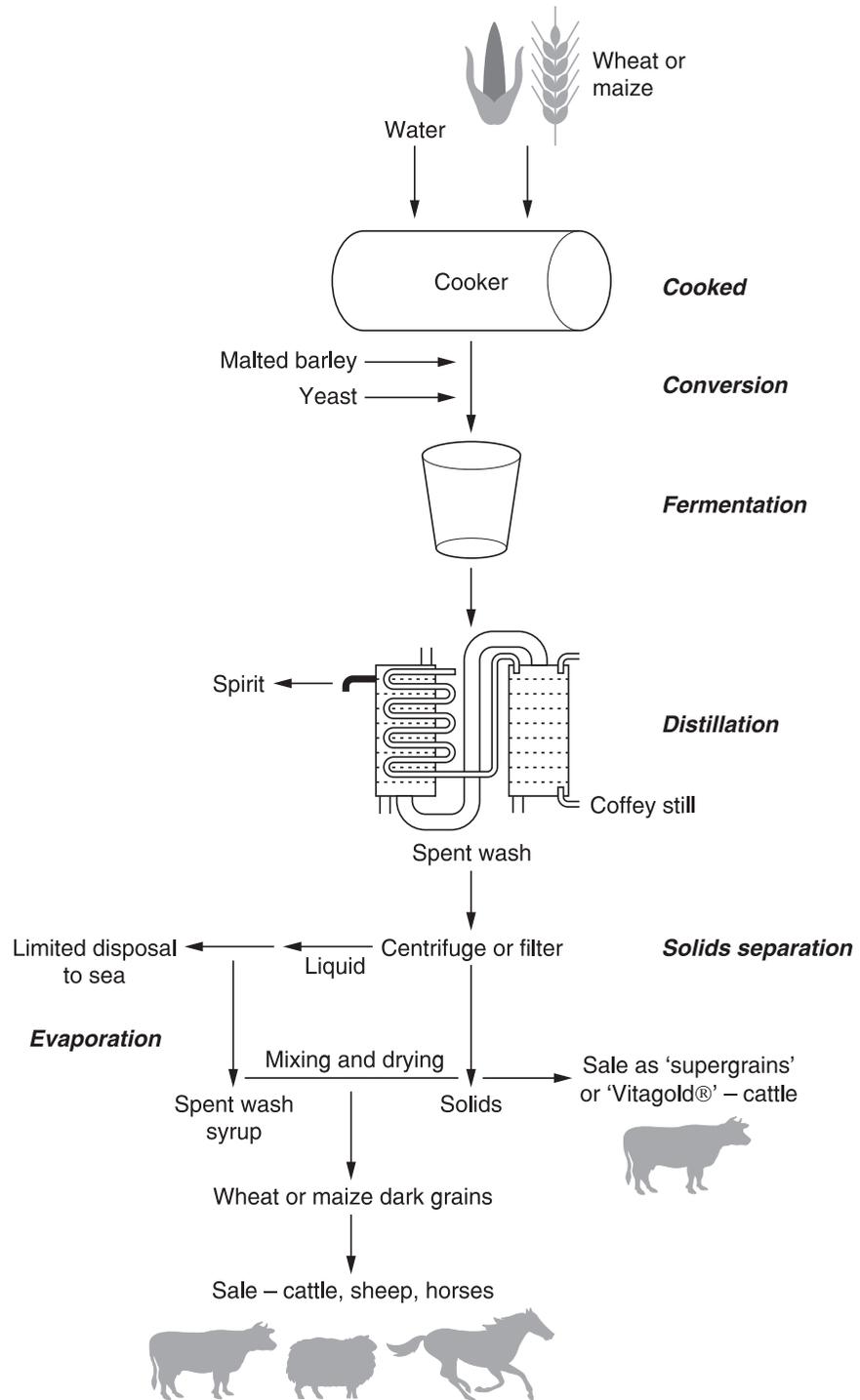
Figure 8.2
Grain distilling co-products – typical process.

sary) of a suitable preservative to extend shelf life. Where local factors prevent coastal discharge, the liquid is evaporated to spent wash syrup before being mixed with the solid fraction and dried and cooled to produce dark grains for sale.

The differences in both processes and still design significantly affect co-product properties. Cereal and yeast protein are denatured to a variable extent depending on the degree of both initial cooking and any subsequent drying.

Copper levels in the co-products also depend on the extent to which wash has been distilled in a copper-rich environment. It can vary from the background low level found in the cereal to over 150 mg/kg dry matter.

Volumes produced

Broadly speaking whisky production has always been cyclical, with recurring peaks and troughs as optimistic demand growth assumptions have led to overstocking followed by reductions in production.

Figure 8.3, compiled from data given by Gray (2000) and Craig (1994), shows estimates of production for both malt and grain whiskies. The dramatic increase from 1955 until 1966 can clearly be seen. Production throughout the Second World War had been very low, and post-war market growth was
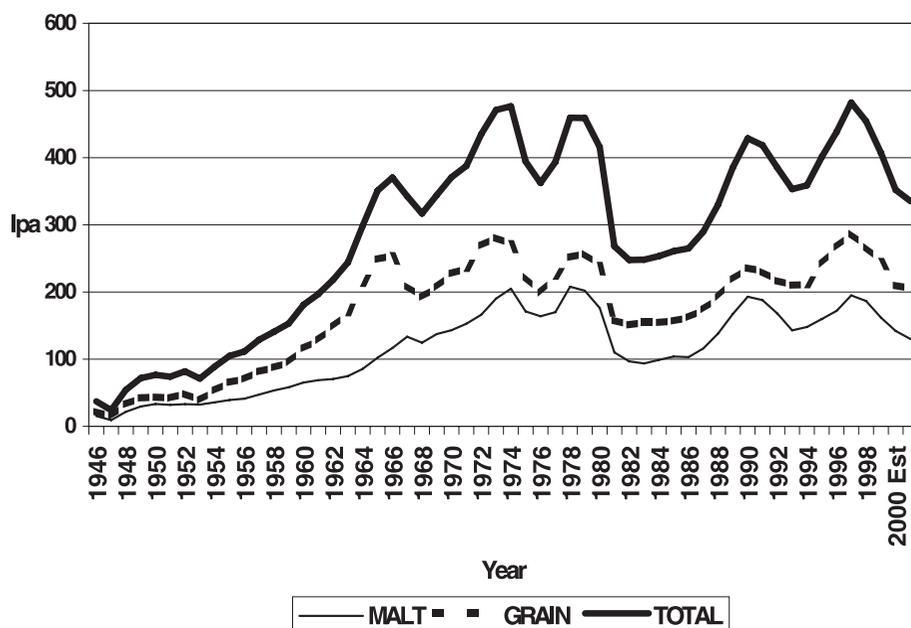
Figure 8.3
Malt and grain whisky distillation 1947–2001 (Sources: Craig, 1994; Gray, 2000).

dramatic as increases in cereal availability coincided with the western world's increasing affluence.

As already noted, co-product utilization is not a new phenomenon, but the substantial growth in production in the 1960s led to significant changes in production methods.

Figure 8.4 (see p. 258) shows a materials flow for malt distilleries. For every 100 t of malt, some 330–345 t of pot ale with a BOD_5 of 25 000 mg/l (Duncan *et al*., 1994) is produced. Grain distilleries produce a somewhat greater volume of spent wash for every tonne of cereal with a very similar BOD_5.

Thus in 1966, with a malt and grain spirit production of 349 million litres of pure alcohol (116 million litres of malt and 233 million litres of grain) (Gray, 2000), around 4 million tonnes of pot ale and spent wash would have been produced (at an assumed spirit yield of 376 and 385 litres p.a./t of malt and grain mashed respectively). For comparison, current total UK beer production is around 5.7 million tonnes (BLRA, 2001). By the mid-1960s the volumes of dilute co-products available in Scotland had become very substantial, and this was the driver behind the building of large drying plants. Where drying of the dilute co-products is not required (for example, where sea discharge is possible), then current economics make the sale of wet cereal residues (draff or the grain distilling equivalent) more desirable.

The industry's growth in the 1960s saw a parallel necessary increase in the industry's drying capacity, ensuring that the dilute co-products did not cause environmental difficulties.

By contrast, the UK brewing industry has not required this investment as the dilute products of fermentation are consumed directly and the moist cereal residues (brewers' grains) are produced over a sufficiently large geographic area to find markets (see Crawshaw, 2001).

Current co-product volumes can be calculated in a similar manner from spirit volumes, spirit yields per tonne of malt or cereal (given in Gray, 2000). The same source also gives an indication of both individual distillery capacity and the industry's overall capacity utilization.

From these figures, and using the simplified stoichiometry given in the process descriptions above, the tonnage of co-products produced can be calculated. It is summarized for relevant years in Tables 8.1 and 8.2.

The effective tripling of the tonnage of residues from the process in the ten years from 1955 led to significant changes in recovery and marketing strategies, with a rise in the number of plants producing dried or evaporated co-products.

Over the last ten years there have been further changes, with smaller malt distilling drying plants closing (e.g. Aultmore, Glenallachie, Teaninich and the Livet Feeds dark-grain plants, and Ardmore and Glendronach dried distillers' solubles plants). The cost of renewing ageing plants to meet modern environmental standards has increased the economies of scale, so that current drying plants serving the malt distilling industry are large and take both draff and pot ale from a number of distilleries. All grain distilleries are relatively large (with capacities of between 10 and 63 mla/annum**34), and can justify integrated co-product recovery facilities.

Table 8.1
Grain distillery co-product volumes

Year	Grain whisky production ($\times 10^6$ l p.a.)	Yield alcohol (l/t)	Residues (1000 t dry matter)
1955	65.5	385	48
1966	233.0	385	171
1997	287.2	377	225
1998	267.6	376	211
1999	245.2	377	192
2000 (estimated)	210.0	377	165
2001 (estimated)	205.0	377	161

Table 8.2
Malt distillery co-product volumes

Year	Malt whisky production ($\times 10^6$ l p.a.)	Yield alcohol (l/t)	Residues (1000 t dry matter)
1955	39.3	376	39
1966	116.0	376	116
1997	194.8	410	154
1998	186.7	407	151
1999	161.9	409	129
2000 (estimated)	142.0	409	113
2001 (estimated)	130.0	409	104

Calculating tonnages of co-product by type

From the above information on distillery capacities and capacity utilization, if it is assumed that all malt or grain distilleries operate at the same capacity utilization for the sector it is possible to calculate the actual tonnages of co-product by type. The calculation is then corrected for those distilleries that discharge pot ale to sea (for example on Islay) or use products as fertilizers. Calculations for malt and grain distilleries are given below.

Malt distilleries

Malt draff production (Table 8.3):

Table 8.3
Draff production

Year	Yield assumed	Capacity utilization	Calculated malt	Calculated draff
1997	410	88	195 500	185 700
2000 (estimated)	409	66	147 000	139 600
2001 (estimated)	409	60	133 600	126 900

	Spirit ($\times 10^6$ l p.a.)
Island distillery capacity – draff sale	20.57
Other known sea/land/sewer disposal of pot ale	26.97
Pot ale syrup production, with draff sale	43.53
TOTAL	*91.07*

Pot ale syrup production (Table 8.4):

Table 8.4
Pot ale syrup production

Year	Calculated malt	Pot ale dry matter[1]	Pot ale syrup tonnage[2]
1997	94 400	12 461	27 691
2000 (estimated)	71 000	9372	20 827
2001 (estimated)	64 00	8514	18 920

[1]Assuming 3.3 m^3/t malt at 4 per cent dry matter.
[2]Assuming an average of 45 per cent dry matter.

	Spirit ($\times 10^6$ l p.a.)
Capacity of malt distilleries selling pot ale syrup	44

Barley dark grains (Table 8.5):

Table 8.5
Barley dark-grain production

Year	Total malt spirit production (kla)[1]	Barley dark grains (tonnage)[2]
1997	114 658	102 300
2000 (estimated)	81 894	73 700
2001 (estimated)	79 358	67 800

[1]Reduced to take account of volume sold as draff.
[2]From stoichiometry and assuming a dry matter of 90 per cent.

Although the above figures can only be approximate, they do illustrate both the variable volumes of co-product available and the changes in production that have occurred.

As a further example, in 1973 in the Highland and Islands Development Board area alone draff sales were estimated at almost 300 000 t p.a., a figure that fell to 128 000 t p.a. in 1975 (primarily due to a fall in spirit production (Mackel, 1977). For comparison, draff use in Scotland in 1979/1980 was about 400 000 t and it was estimated that potential use was double that figure (Lilwall and Smith, 1983).

As the above calculations show, draff sales are now running at between 125 000 and 200 000 tonnes per annum and are unlikely to increase significantly unless alternative methods of pot ale processing can be developed.

The alternative strategy to producing barley dark grains is to sell the draff and produce pot ale syrup. The list of PAS evaporators and their potential maximum production is given in Table 8.6.

Current production is little more than half that given in Table 8.6, with some of the PAS being produced and then taken to dark-grain plants. In the last ten years some older evaporation plants have closed, and total production in the early 1990s will have reached or exceeded the current capacity of some 36 000 t p.a. At the high level of production seen in 1991 the market showed signs of distress in the summer, with stocks being very high and prices collapsing to a low of about £4/t to the distiller.

Table 8.6
Pot ale syrup production capacity

Distillery	Capacity (kla)	Maximum pot ale syrup production
Aberfeldy	1800	1300
Aberlour	3200	2300
Blair Athol	1700	1200
Dufftown	4000	2900
Glenallachie	2500	1800
Glenfarclas	3000	2100
Glenfiddich, Valvenie and Kininvie	20 000	14 300
Inchgower	2300	1600
MacDuff	2500	1800
Tomatin	7000	5000
Tomintoul	2800	2000
TOTAL		36 300

The present combination of draff, pot ale syrup and barley dark-grain sales volumes, primarily driven by the need to process pot ale, has fortuitously largely kept the market in balance, with the more perishable draff being consumed mainly in Scotland and Northern England and the more concentrated, higher value PAS and BDG being transported and used throughout Britain.

Grain distilleries

Both dried and moist feeds are produced by grain distilleries, the latter where it is possible to dispose of the dilute centrate or filtrate by extended sea pipeline (Supergrains and Vitagold) or evaporation to spent wash syrup (Loch Lomond Gold).

Calculations of the volumes of grain distillers feeds are complicated by the fact that most grain distilleries produce both grain spirit (for maturation in oak to grain whisky) and the more highly rectified neutral spirit for use in gin, vodka and other spirit-based drinks. Published statistics for grain spirit production exclude neutral spirit volumes, which are commercially confidential. Thus the figures given in Tables 8.7 and 8.8 for potential grain co-product production will be approximately correct, but figures for actual production will certainly be an underestimate (by probably about 20 per cent) as they do not include the co-products derived from neutral spirit production.

Figure 8.5 (see p. 265) shows the distribution of potential co-product production with the relevant livestock population for the Scottish Regions.**35 The major consumers – dairy and beef cattle – are concentrated in the Southwest of Scotland (where milder winters, higher rainfall and better land

Table 8.7
Grain moist feeds – calculated production at 61 per cent utilization (Year 2000)

Distillery (product name)	Capacity (kla/annum)	Cereal and yeast residue[1] (t dry matter)	% Recovery[2]	% Dry matter	Actual tonnes of product
Cameronbridge (Supergrains)	56 000	27 518	50	25	55 000
Girvan (Vitagold)	60 000	29 484	60	30	59 000
Loch Lomond (L. Lom. Super)	10 000	4914	50	25	10 000
(Loch Lomond Gold)	10 000	4914	50	38	6500
TOTAL					130 500

[1]At assumed yield of 377 l alcohol/t cereal mashed.
[2]Centrifuge processes assumed to be less efficient than filtration. Dried feeds (drying losses only) assumed to be 3 per cent.

Table 8.8
Grain dried feeds – calculated production at 61 per cent utilization (Year 2000)

Distillery (product name)	Capacity (kla/annum)	Cereal and yeast residue[1] (t dry matter)	% Recovery[2]	% Dry matter	Actual tonnes of product
Dumbarton	35 000	17 200	97	90	18 500
Invergordon	38 000	18 700	97	90	20 000
North British	63 000	31 000	97	90	33 000
Port Dundas	39 000	19 200	97	90	21 000
Strathclyde	32 000	15 700	97	90	17 000
TOTAL					109 500

[1] At assumed yield of 377 l alcohol/t cereal mashed.
[2] Centrifuge processes assumed to be less efficient than filtration. Dried feeds (drying losses only) assumed to be 3 per cent.

encourage dairying; in the East of Scotland, cereal growing has encouraged beef production as a complementary enterprise to arable farming). In general there has been a movement of co-products to the South and West, with many also being used in Northwest England. The dried feeds can be distributed throughout the British Isles, and are also exported, often from northern ports, to the Benelux countries, Scandinavian and Ireland.

Agri-Food Market Analysis (2000) is a map showing brewing and distilling in the UK, and illustrates the difficulties that can arise in more remote geographical locations, where distillery production can be relatively large compared to livestock numbers.

Islay is a good example of the problems that can occur. The Dunlop cheese plant sadly closed in the spring of 2000, and dairy cattle numbers declined from about 700 to 150. The dairy herd and followers were major consumers for the draff from the island's distilleries. At capacity, the seven working distilleries on Islay and neighbouring Jura (Ardbeg, Bowmore, Bunnahabhain, Caol Ila, Isle of Jura, Lagavulin and Laphroaig) produce around 36 000 t p.a. of draff. This is sufficient for almost 3 t per head per annum for all the cattle on the two islands. This is a very high rate of draff usage; the 1983 survey by Lilwall and Smith found an average usage rate of 0.7 t per head per annum, with maximum usage rates of 2.2 t and 1.2 t per head per annum for beef fatteners and dairy specialists.

With the recent drastic reductions in the dairy herd there is therefore an urgent need to increase beef enterprises to sustain both rural and distilling sections of the local economy. If this is not done, then distilleries may be left with the undesirable option of either regarding the draff as waste or transporting it to the Scottish mainland; both at considerable cost.

Although there may be difficulties in areas where draff transport is very expensive, Scotland as a whole could use more draff and other moist feeds were it possible to utilize the pot ale. Lilwall and Smith (1983) estimated 1979/1980 draff usage at 416 000 t p.a., and considered that uptake could be more than doubled. They stated that an improved marketing strategy of longer-term allocation between drying plants and direct sale would increase volumes. However, they did not address the central problems of dealing with fluctuating supplies, and the use of the pot ale released, were draff sales to increase.

This latter constraint means that in the longer term moist co-product sales will decline from the current figure of 284 000 p.a. with an increase in dried feeds.

Waste waters and effluents

The remaining co-products, which have no commercial value, must be processed and/or disposed of economically and in compliance with environmental legislation. These comprise the second malt distillation residues – spent lees, washing waters from cleaning process vessels and pipelines – together with condensate where pot ale or spent wash is evaporated to produce syrup or, by further processing, dark grains. In addition there may be liquors produced by vapour scrubbing of dark grains plant dryer emissions, as well as blowdown from steam boilers and purge-water from cooling systems. Figure 8.4 provides an illustration of the derivation and quantities of the individual wastewaters produced during the making of malt whisky and dark grains.

Several disposal routes are available, all now regulated by various pieces of legislation, the principle one being the EC Urban Wastewater Treatment Directive (UWWT), which controls both the direct discharge of wastewaters into inland freshwaters, estuaries and the sea, and discharges made indirectly via public sewage systems. In a few instances, wastewaters are spray irrigated over farmland, or lagooned. This relatively new piece of European legislation lays down the minimum degree of treatment necessary prior to direct discharge into any of the 'controlled' waters mentioned above. In addition, the regulatory authority enforcing the legislation – the Scottish Environment Protection Agency – requires that several Environmental Quality Standards (EQS) are achieved; notably those for dissolved copper and dissolved oxygen in the receiving waters.

At certain distilleries, located on estuaries and adjacent coastal waters where good dispersive and mixing conditions prevail, pot ale or spent wash are disposed off directly via long sea outfalls along with the weaker wastewaters and, in some instances, cooling water, but only where the local conditions allow the UWWT regulations and EQS achievement to be complied with.

Disposal of wastewater via public sewerage systems normally necessitates pre-treatment on site in order to meet Water Authority requirements, so that they in turn are able to comply with UWWT regulations.
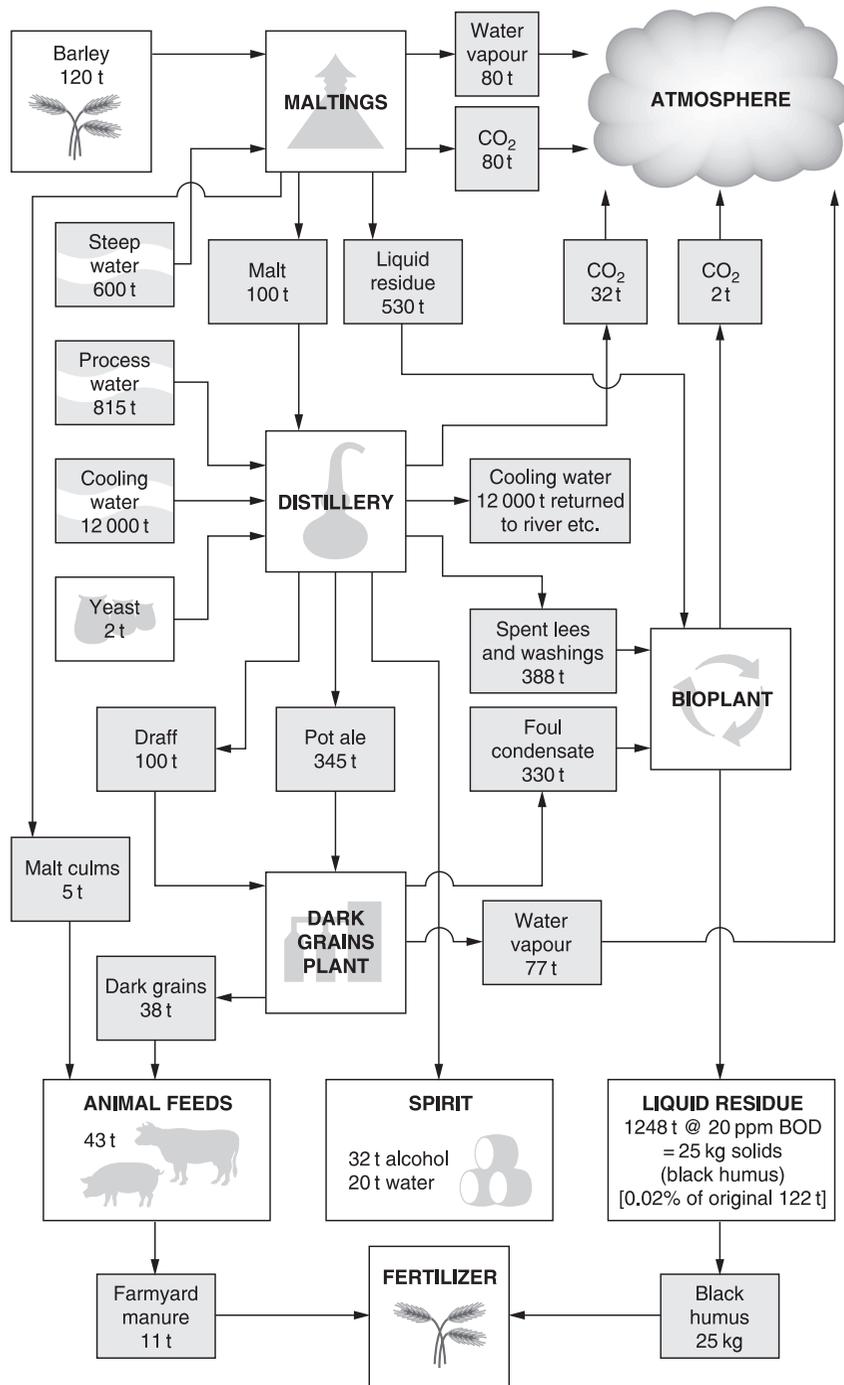
Figure 8.4
Weekly material flow for a medium-sized distillery with associated maltings, dark-grains plant and bioplant.

At inland sites without access to public sewers, on site treatment is obligatory before discharge to a watercourse that is, more often than not, one capable of supporting salmonid fish. Consequently the degree of treatment required is high, and the consent conditions imposed by the Scottish Environmental Protection Agency (SEPA) are correspondingly stringent.

The nature of the wastewater lends itself to biological treatment provided the acidity and lack of nutrients (nitrogen and phosphorus) are first rectified.

Figure 8.4 illustrates the quantities of sludge derived from the biological treatment of wastewaters, which approximates to 0.5 kg dry matter for each kilogram of biochemical oxygen demand (BOD) degraded.

The high concentration of copper in the spent lees gives rise to high levels of copper in the surplus biomass produced during treatment, effectively removing copper from the treated effluent. Subsequent applications of the 'bioplant sludge' to the farmland give rise to elevated levels of copper in the soil, which must be monitored and managed carefully in order to comply with the Code of Practice associated with the disposal of sludge to agricultural land. The copper deficiency of much of the land in Scotland exemplifies the benefits of this practice, provided the appropriate precautions are taken.

In anticipation of an overall tightening of environmental legislation relating to potentially toxic elements, the industry has developed methods of removing copper from spent lees in order to ensure that current disposal routes for wastewater and surplus biomass are secure.

Copper in spent lees is present, for the most part as soluble Cu^{2+} ions, which lends itself to removal by ion exchange, electro-winning, or precipitation and membrane filtration. In fact all three techniques have been investigated, and as a result two successful methods have been developed; one based on a combination of cation exchange followed by electro-winning of the eluted soluble copper concentrate, and the other using precipitation by sodium hydroxide and ultra-filtration to concentrate the precipitate. However, only the first method recovers copper as a metal.

The other major emission from the whisky distillation process is of water used for distillate cooling. Where water resources are sufficient, water is used on a total loss basis amounting to $120 \, m^3/t$ of malt mashed. At many sites, however, water supplies are limited, resulting in full or partial recirculation through a cooling tower.

Limitations on the temperature are imposed under the Freshwater Fisheries Regulations in order to protect freshwater fisheries, especially where salmonids occur. Restrictions are placed on cooling water discharges such that a receiving water does not become heated above a certain point, which poses problems, especially during the summer months when ambient temperatures are high and stream flows are low. Traditionally these months were avoided by distilleries for whisky making, but production requirements have meant that whisky has to be made when conditions are less than favourable in terms of temperature and water availability. Consequently, steps must be taken to reject heat prior to return to a watercourse – usually by means of a cooling tower.

Markets for co-products

The calculations above show the considerable volumes of co-products produced by the whisky industry.

The distillery uses the starch in the cereal to produce alcohol, and the residue is thereby enriched by plant structural fibre, protein and oil, and yeast.

The requirements of the industry are for relatively large markets capable of absorbing the fluctuating supplies of available co-product. Smaller niche markets do exist, but are of little economic significance. Possible markets therefore include:

- Animal feeds
- Human food
- Fertilizer
- Fuel
- Production of biomass.

All the above markets are large, and their respective potential can be compared using dark grains as an example.

Animal feed

The UK market for animal feed is large, with some 20 million tonnes worth £2.2 billion being purchased in 2000 (Dean, 2001).

The percentages of purchased compounded (manufactured) feed for each class of livestock are as follows (*Feed Facts Quarterly*, 2000):**37

	1999 volumes (%)
Cattle and calves	38 per cent
Pigs	21 per cent
Poultry	27 per cent
Sheep	7 per cent
Others (including horse feeds)	7 per cent

In terms of market size, the ruminant (cattle and sheep) market is the largest single sector. It is also nutritionally less demanding than the others. Ruminants have evolved since the appearance of grass in the Miocene period some 20 million years ago, and use pre-gastric fermentation where micro-organisms degrade the plant cell wall. The ability to ruminate (i.e. regurgitate, rechew and reswallow) the feed reduces the particle size and aids this breakdown. In addition, the presence of large numbers of micro-organisms allows the use of a wider range of protein sources and non-protein nitrogens (Van Soest, 1982).

Monogastric animals and birds lack the enzymes required to break down plant structural carbohydrate, and also require protein with a higher content of essential amino acids.

The essential amino acids of wheat dark grains, extracted soya bean meal and fish meal are compared in Table 8.9.

In modern pig and poultry diets the principle limiting amino acids are commonly lysine, threonine and methionine. Wheat dark grains are relatively low in these amino acids, and are particularly deficient in lysine.

The relative poor amino acid profile of the distillers' feeds, together with their elevated cereal fibre content, means that they generally find their highest economic value in ruminant feeds. Small volumes of pot ale, pot ale syrup and grain dark grains are used in other species, where they have desirable particular attributes (for example, the low fibre in pot ale syrup makes it suitable for pigs, and the high xanthophyll pigment content in maize dark grains makes them attractive for pigmenting egg yolks).

Table 8.9
Comparison of essential amino acids (g/16gN)
(Sources: ARC, 1981; MAFF, 1990)

	WDG	SBM	FM	IP	WDG % IP
Arginine	3.31	7.90	5.10	–	–
Histidine	3.24	3.18	1.89	2.3	141
Isoleuc ine	3.80	5.11	3.64	3.8	100
Leucine	8.50	8.19	6.69	7.0	121
Lysine	2.15	6.77	5.67	7.0	31
Methionine + cystine	2.98	2.80	3.78	3.5	85
Phenylalanine + tyrosine	7.97	9.32	7.16	6.7	119
Threonine	3.31	4.15	4.39	4.2	79
Valine	4.83	5.84	5.34	4.9	98

WDG, wheat dark grains; SBM, extracted soya bean meal; FM, white fish meal; IP, ideal protein (pigs).

Human food

The nutritional properties outlined above make entry into the human food market problematic, and there have been a number of studies of co-product use (Tsen, 1982; Cooler *et al.*, 1986; O'Palka, 1987) in both baked goods and in canned meat products.

Although the co-products could be included at low levels without significant adverse effect, at higher levels the lack of functional gluten, the dark colour and flavour adversely affected product quality. It has to be concluded that, without the use of low temperature drying, the removal of at least some

cereal fibre and the production of lower copper products, their monetary values in human food applications will remain low.

Fertilizer

As noted above, dilute liquid co-products have been used as fertilizers for many years, and numerous studies have been conducted by the Scottish Agricultural College. Use on hills encourages grass growth at the expense of heather (Bucknall et al., 1979), and applications of over $1000\,m^3$/ha per year have converted rough grazing into productive grassland. They have also been used on grassland and in forestry, but such use has only been on a limited scale as access problems through the winter and the dangers of run-off result in limited widespread adoption.

Recently, year-round application of pot ale to grassland has been practised at those sites remote from access to further processing. Rigorous control of soil copper levels and avoidance of run off, either directly or via field drainage systems, have had to be established in order to satisfy the regulatory authorities.

The dried co-products can also be used as a fertilizer, but here the primary constraints have been economic rather than logistical. A recent study, (Bingham and Sinclair, 2000, personal communication) chemically analysed typical co-products (see Table 8.10).

Scottish Agricultural College experiments showed a positive plant response to the above co-products, but in comparison with commercially available fertilizers they are very dilute nutrient sources.

From quoted market information it can be calculated that the value of nitrogen, phosphor and potassium in a range of inorganic fertilizers (5:24:24, 13:13:20 and 20:10:10) is currently £3.78, £1.25 and £2.87 per unit of N, P_2O_5 and K_2O respectively (Bojduniak, 2001a, 2001b).**38

Table 8.10
Chemical analysis of co-product
(Source: United Distillers & Vintners (Distilling) Ltd)

Product	Dry matter (%)	pH	N (%)	P_2O_5 (%)	K_2O (%)	Cu (mg/kg dry matter)	Farm value (£/t)
Steeped barley	40	3–5	0.82	0.73	0.48	2	5.40
Malt draff	22	3–5	0.70	0.47	0.19	4	3.79
Barley dark grains	90	N/A	3.89	3.71	2.21	50	25.74
Malt residuals	90	N/A	3.31	2.76	3.75	15	26.77
Pot ale syrup	45	3–5	2.45	4.53	2.49	29	22.12

The monetary value of the co-product nutrients is given in the last column of Table 8.10. With a range of typical value from below £4/t to £27/t, sale of the products in competition with inorganic fertilizers is unattractive.

Research carried out by Diageo**39 some years ago demonstrated that a peat substitute could be produced from draff by composting. However, the relatively small market, combined with high transport and marketing costs, has so far not resulted in any sustained commercialization.

Fuel

The use of fossil fuel to dry the co-products first so that they can then themselves be used as fuel would at first seem perverse. However, sugar cane residues ('bagasse') has been used as a fuel for many years (Morrison, 1947), and more latterly as a source of energy for fuel alcohol production.

Using gross energy figures for the dried animal feeds (Gizzi, 2001) and DTI data (Department of Trade and Industry, 2001) on the cost and gross energy of coal for UK power generation, it can be calculated that the value of barley, wheat and maize dark grains are £21.71/t, £22.01/t and £23.06/t respectively. These values assume that the dark grains could be used as a direct replacement for coal with no other modification (in practice at least some changes would probably be necessary, as, for example, the dark grains could not be exposed to the weather).

Again, except as a last resort the use of the feeds as fuel is very unlikely to be competitive.

In conclusion, of all the potential large markets examined use as ruminant feed remains the most attractive – as has historically been the case.

Although such dependence on a single market has its dangers (as the recent bovine spongiform encephalopathy (BSE), 2001 foot and mouth disease epidemic and low UK milk prices all illustrate), other major markets have not yet proved more economically attractive.

Fuel may, however, be derived indirectly from pot ale and spent wash by means of anaerobic digestion, whereby a mixed culture of bacteria break down the soluble organic components into methane and carbon dioxide. Unfortunately the digestion process requires the prior removal of the dead yeast cells, since their refractory nature prevents degradation, which would give rise to accumulation in the reactor and eventual process failure. Methods for removing dead yeast cells include high-speed centrifugation, and coagulation with carboxymethyl cellulose followed by belt pressing and membrane ultra-filtration. Equipment is costly to install, but the recovered yeast, at around 20 per cent dry matter, could be incorporated with wet draff for direct feeding, or transported to a dark grains plant for processing there.

Yields of methane would be equivalent to $0.34\,m^3/kg$ chemical oxygen demand (COD) destroyed, which, for a clarified pot ale with a COD of $45\,kg/m^3$ and 90 per cent degradation by anaerobic digestion, would produce around $14\,m^3/m^3$ – equivalent to approximately 14 l of heavy fuel oil. Unfortunately, the digestion process releases most of the organically bound

nitrogen and phosphorus as inorganic ammonium and phosphate salts – typically 1500 mg/l and 650 mg/l respectively. Consequently the only viable disposal route is by application to farmland, where the nutrients would be useful fertilizer additions – especially for grassland.

Several schemes have been trialled in the past, which confirm the effectiveness of anaerobic digestion, but to date no full-scale system has been installed. Instead, at sites economically remote from dark-grains plants or without access to well-sited marine outfalls, pot ale is applied directly to grassland at carefully controlled rates as a fertilizer.

Production of biomass

The production of microbial biomass from pot ale and spent wash is an attractive proposition at first glance, bearing in mind the relatively large amounts of amino acids and unfermented sugars present in these two co-products. Indeed, over the years several attempts have been made to grow selected micro-organisms for harvesting as animal foodstuffs.

For some years, product formulated from evaporated spent wash was marketed as a culture medium for lactobacilli (trade name 'Kickstart') and sold in the farm market. The concentrated culture medium was diluted on farms, and a vial of freeze-dried lactobacilli was added and grown. The resulting bacterial culture was then used as an inoculant on grass to improve silage quality. Although used successfully, the project came to an end with the closure of Cambus Distillery in 1993.

Other notable work was undertaken by Wm Grant & Sons, where a fungi imperfecti and a yeast were grown in sequence to exploit these amino acids and sugars. Unfortunately, transferring the process from laboratory pilot to full-scale use gave rise to insurmountable problems, and the scheme was abandoned.

Another process, trialled by Long John Distillers in the 1980s at Tormore Distillery, utilized the so called Malimo process. *Aspergillus niger* was the organism selected following screening for best results. Again, problems were encountered that led to abandonment of the process.

Work in Northern Ireland in the 1980s (Barker *et al.*, 1983), where mixed cultures of filamentous fungi and yeasts were grown on pot ale, also showed promise. However, as with other biomass production projects, process and EU approval difficulties have not yet allowed commercialization.

Nutritional characteristics

Distillers' feeds have been used throughout the world to great effect, and scientific study of them has also been extensive. A 50-year world literature search of *Nutrition Abstracts and Reviews* (Commonwealth Agricultural Bureau, 1974, 1978, 1980) and, since 1986, the Dialog database, produced

861 articles on brewing and distilling co-products used in animal feeds (see Figure 8.5). It may be that the sharp annual increase from 1973 to 1983 was partly due to rising fuel costs and environmental pressures stimulating research interest.

The recent decline in numbers of publications over the last four years has no particular significance, except that as the feeds have become thoroughly understood then further research is progressively less cost-effective.

Most recently, both the moist and dried co-products have been extensively reviewed (by Crawshaw, 2001 and Gizzi, 2001, respectively). To avoid repetition, only nutrients of major economic significance are considered below.

Using April 2001 raw material prices (Agricultural Supply Industry, 2001;**40 Bojduniak, 2001a, 2001b) to formulate a typical compound dairy feed (with energy and protein levels of 12.4 MJ/kg and 20 per cent of dry matter), then the marginal value**41 of, for example, barley distillers' dark grains is £93.49/t. In this typical example the nutrients of value were metabolizable energy, protein, phosphorus and salt. Table 8.11 illustrates their relative importance.

Table 8.11 shows that the energy content of the co-products is the most important single determinant of value, followed by protein. Other nutrients are far less significant. It is for this reason that the ruminant metabolizable energy value (ME) of the co-products has been particularly well researched, despite the high cost of such trials in animals.

In the UK alone there have been 26 *in vivo* ME determinations for the dried feeds (Gizzi, 2001) and 8 for brewers' grains and draff. This number compares well with major competing feeds, such as extracted soya bean meal or maize gluten feed (MAFF, 1990).
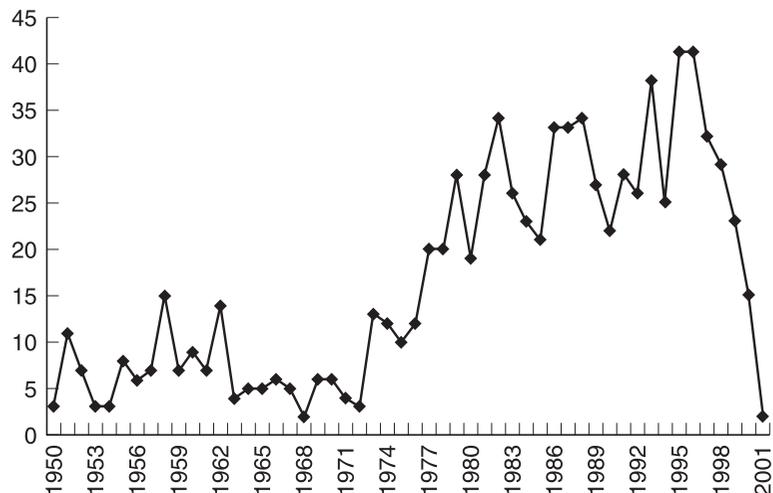

Figure 8.5
Brewing and distilling feeds: scientific publications 1950–2000.

Table 8.11
Effect of 10 per cent reduction in level of limiting nutrient in barley distillers' dark grains on its marginal value in a dairy compound

Nutrient	% Reduction in value
Metabolizable energy	13.3
Protein	3.4
Phosphorus	1.1
Salt	0.1

The dried feed data set have also enabled the production of a prediction equation (Gizzi, 2001):**42

$$ME = 0.966\ GE + 0.006\ NDF + 5.11\ Cereal\ ME - 80.5$$

(S.E.s 0.831, 0.232, 0.002, 1.647 and 22.121 respectively.

$$Adjusted\ r^2 = 0.623^{***},\ n = 23$$

where ME and GE are the values of metabolizable and gross energy of the dark grains (expressed in terms of MJ/kg organic matter (OM), NDF is the concentration of neutral detergent fibre in the dark grains (expressed as g/kg OM), and cereal ME is a constant based on the average ME of the predominant base cereal (expressed in terms of MJ/kg OM – maize = 14.00, wheat = 13.95, barley = 13.68 (MAFF, 1990)).

The use of such equations allows a reasonably accurate figure for ME to be calculated despite relatively wide variations in manufacturing and process conditions.

Protein is the next most economically important nutrient in the distillers' feeds, and the amino acid profile of the feeds is more typical of vegetable protein and is thus best suited to ruminants. However, there has long been controversy about the extent and importance of the heat damage that may be produced by the processes (see Gizzi, 2001). The measure of undegraded and indigestible nitrogen proposed for forages (Van Soest, 1965), acid detergent insoluble nitrogen (ADIN), is a useful indicator of heat damage, although much of the ADIN itself is digested (Whyte, 1993).

Practical feeding experience has not indicated any problems with protein availability (Gizzi, 2001). McKendrick and Hyslop (1992) demonstrated that performance of dairy cattle on wheat or barley dark grains was better than that on a conventional concentrate.

No discussion of co-products from whisky production would be complete without mention of copper, as any of the feeds that include still residues may contain high levels.

In the development of distilling, copper had the obvious advantages of malleability, ready availability, high thermal conductivity, no taint, relative resistance to corrosion, and low toxicity. As noted elsewhere, the use of copper was serendipitous as it removes undesirable congeners; however, relatively high levels can be found in the co-products. Copper is an essential trace element for all mammals, but symptoms of both deficiency and toxicity are mostly reported in ruminants (Underwood and Suttle, 1999).

The ability of copper-rich distillery feeds to increase liver copper in lambs has been studied (Suttle *et al.*, 1996), and copper availability determined. Respective availabilities were 0.011, 0.038, 0.064 and 0.069 for PAS, barley dark grains, wheat dark grains and Supergrains respectively. For comparison, copper sulphate was found to have an availability of 0.057. Reasons for the wide range of availability seen are not readily forthcoming. It may be that the copper in PAS is in the form of a heat-labile sulphur and/or amino acid complex, and is thus unavailable unless the product is dried. Alternatively, it has been proposed that the formation of copper soaps from cereal lipids may both prevent the formation of unavailable complexes in the rumen and allow digestion (Gizzi, 2001). This could explain why availability may be higher than in copper sulphate.

As certain breeds of sheep are highly susceptible to copper toxicity (Underwood and Suttle, 1999) and there is a wide variability in copper content availability, the guidance of the Scottish Agricultural College is best followed (Lewis, 2001).

Relevant legislation and Codes of Practice

UK feed legislation is relevant to co-products sold as feeds. It is mainly derived from relevant EU Directives and Regulations and is therefore consistent across the EU, with only minor national differences. The Feeding Stuffs Regulations 2000 (Statutory Instrument 2000 No.2481) are applicable in England, with equivalents in Scotland and Wales.

The legislation is both complex and comprehensive, with the dried co-products specifically mentioned and a derogation**43 from inappropriate labelling requirements for the moist feeds. An interpretation of current legislation with future proposals is given in Knock (2002), and matters specific to moist co-products are also summarized in Crawshaw (2001). Briefly, a label must accompany each feed consignment, and must include a description of the feed, information on provenance, and, in the case of the dried feeds, net weight and protein content. There are also proscribed limits for undesirable substances, pesticides and aflatoxin.

In addition to the legislation, the industry undertakes to comply with the relevant non-statutory codes of practice in place (GAFTA, 1999; BFBi, 2000; UFAS, 2000), including the relevant salmonella code (MAFF, 1995). These codes cover manufacturing, storage, transport and recommendations for farm storage of the moist feeds. Hazard analysis critical control point (HACCP) techniques are widely used by the industry to ensure compliance.

The legislation and codes further reinforce the safety and integrity of these inherently safe feeds. Potential hazards are few, and include:

1. Spoilage. Inappropriate storage of moist feeds (e.g. allowing air to cause aerobic breakdown) or water damage to dry feeds can lead to microbial breakdown. Such spoilt products should never be fed. as they may contain fungal and/or microbial toxins.
2. Foreign bodies and cross-contamination. The nature of distilleries and co-product processing plants is such that reasonable diligence should prevent the former, and cross-contamination is not usually possible, due to the dedicated nature of the process.
3. Undesirable substances, pesticide residues and salmonella. These are all monitored with no adverse reports. Incoming cereals, to maltings or distillery, come with a 'pesticide passport' detailing any treatments with storage pesticides. The extent of heat treatment through the process ensures the absence of coliforms at the point of production, and after many years of testing no positive salmonella isolations have been reported.
4. Mycotoxins. The high molecular weight and low volatility of these natural fungal toxins ensures that they will not distil, but can remain in cereal and distillation residue as a contaminant. The use of maize normally requires analysis of aflatoxin in the cereal, as the toxin is heat stable (Chu *et al.*, 1975), and the levels are controlled by feed legislation (Statutory Instrument 2000, No.2481). Ochratoxin A, a toxin produced when cereals are stored in poor conditions, is unlikely to be found in barley purchased for malting, as germination potential would be affected. Other cereals used in the processes could potentially be contaminated, but destruction of the toxin during cooking, fermentation and co-product recovery can be substantial (Krogh *et al.*, 1974; Chu *et al.*, 1975; Scott, 1996). Levels in co-products will normally be very low, as UK cereal suppliers follow the CODEX principles for the prevention of cereal mycotoxins (World Health Organization, 2001). Any minor contamination is unlikely to have any adverse effects on animal performance as the limited data available indicate that ochratoxin is of relatively low toxicity to cattle (Miller, 1995; World Health Organization, 1996), perhaps because microbial fermentation in the rumen has been shown to degrade the toxin (Kiessling *et al.*, 1984). This degradation also makes it highly unlikely that any residues would be found in milk or meat from cattle (Scudamore, 1996).

It may be concluded that distillers' feeds are inherently safe. Any nutritional or other hazards have been identified, and appropriate measures and controls put in place.

Conclusions

Over 200 years of practical experience of feeding distillery co-products has shown them to be valuable, safe feeds.

Concern for the environment has always been a major priority for the industry. Drying plants were originally developed in the first part of the twentieth century, primarily to avoid problems with water pollution. Drying the diluted co-product streams from the industry has seldom been viable in its own right, and this continues to be the case.

Where appropriate, the use of the dilute liquid co-products as fertilizers could be further developed. At coastal locations, when rapid and efficient dispersion and dilution is possible, there would seem to be no reason why these same materials cannot be used as informal fish nutrients.

The industry's growth was almost exponential from the end of the Second World War until the mid-1960s. Thereafter production has been cyclical, and co-product volumes have reflected this pattern, always finding a sustained, reliable feed market. This promises to continue.

The major attributes of the feeds are consequences of their cereal origin and removal of starch by fermentation (thereby proportionately concentrating other nutrients). High in ruminant metabolizable energy, protein, digestible fibre and unsaturated oils, they are (on a dry basis), frequently of higher value than the cereal from which they are derived. It does appear that, at first sight, the distiller has the benefit of using the cereal to produce alcohol and selling a co-product of higher value than the cost of the input material. Unfortunately the cost of drying the dilute cereal residues normally exceeds the value of the end product, thus more than neutralizing any possible financial benefit. Feed analysis indicates that they are best suited to use in ruminants and, without further modification, this is likely to be their major market.

For the future, continued dependence on the cattle feed market seems inevitable and, as previously, process developments will continue to have a strong environmental component. To protect the environment (and despite much of Scotland's soil being copper deficient), processes to remove ionic copper from the spent lees have been developed. It is likely that techniques will also be developed to either remove the copper from co-product streams such as pot ale (where most copper is complexed; Quinn, *et al.*, 1980) or prevent high levels occurring whilst still allowing the beneficial effects of copper compounds on spirit quality.

Similarly, the heat damage to protein may have very little impact on animal performance, but a reduction in protein digestibility will increase the animal's nitrogen excretion. Again there is a need to reduce nitrogen deposition. For

this reason, low temperature processing and co-product drying will become of greater interest. Indeed, trials using membrane filtration techniques proved to be effective at preconcentrating pot ale to 20 per cent dry matter, together with reverse osmosis concentrating the expressate to another 20 per cent dry matter concentrate (Jones, 1994, personal communication).

Current concerns with safety, integrity and the welfare aspects of modern food production are likely to increase and further influence EU and government policy. However, the safety and integrity of whisky industry co-products is recognized in the marketplace, and demand is likely to continue to outstrip supply.

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Chapter *9*

Whisky analysis

Ross Aylott

Introduction

Whisky, like most other distilled spirits, when purchased by the consumer is by analysis essentially a mixture of water, ethyl alcohol and congeners in the approximate proportions 59.9 per cent, 40 per cent and 0.1 per cent by volume, respectively. The alcohol is fermented from cereals and concentrated by distillation. The congeners are the trace secondary components, and are present as a result of the raw materials used, and the fermentation, distillation and maturation processes. It is the congeners that give whiskies their unique sensory characteristics. Analysis of the congeners is integral to modern fermentation and distillation process control, process research and development, quality assurance, and brand protection.

Although whisky has a history going back well over 500 years, little was known about its composition (other than alcohol) until the late nineteenth century. Early work encompassing Scotch whisky by Allan and Chataway (1891) described methods for the detection of alcohols, aldehydes, esters, amines and furfural. The authors made two very important points:

> The secondary constituents of spirits are by no means to be regarded in the light of impurities, as they have been wrongfully called and considered by some. They are the associated bodies which give the alcohol its special and valued characters . . .
> The secondary or bye products of spirits are naturally most abundant in those spirituous liquids manufactured in apparatus where no, or but little, fractionation occurs. This is the case with the spirit distilled in Scotland in pot stills, and made wholly from fermented malt, and in Ireland from a mixture of malted and unmalted barley.

Our analytical knowledge of whiskies has expanded rapidly in recent years, starting in the 1960s with the introduction of gas chromatography (Singer and Styles, 1965; Duncan and Philp, 1966), gas chromatography–mass spectrometry (Kahn, 1969), and then high performance liquid chromatography (HPLC;

Lehtonen, 1983a). There are now known to be many hundreds of congeners in whiskies, and these include alcohols, acids, esters, carbonyl compounds, phenols, hydrocarbons, and nitrogen- and sulphur-containing compounds (Swan *et al*., 1981). Whiskies distilled on pot stills are richer in congeners than those whiskies made with greater rectification on continuous stills. Congener concentrations typically cover concentration ranges from the high parts per million (mg/l) down to the low parts per billion (μg/l).

Congeners are natural constituents of the production process; some clearly contribute sensory character and some others do not. However, together all the congeners help make each whisky unique. The presence of each congener is associated with specific parts of the production process – for example, the cereals used, or the fermentation, distillation or maturation processes. These processes affect the final concentration of each congener in the final product. For example, congeners such as higher alcohols formed in fermentation may be reduced in concentration and even eliminated by rectification during distillation. Ethyl esters formed in fermentation may increase in concentration during maturation, and wood-related congeners not present at all at the end of distillation are extracted from casks into the liquid during maturation.

Whiskies of the world and their regulatory definitions

Whisky is a distilled spirit fermented from cereals, distilled at less than a maximum alcoholic strength (normally <94.8 per cent vol.) and matured in oak casks for a minimum period (typically three years). The normal minimum bottling strength for consumption is 40 per cent vol. Additives are not normally permitted, except for spirit caramel in some whiskies for the purpose of colour standardization and water for alcoholic strength reduction from maturation to bottling strength.

The first whiskies are believed to have been made in Scotland and Ireland. The first record of whisky production in Scotland dates from 1494 (Anon. 1494 Craig, 1994), although whisky manufacture only started to become a regulated process in the nineteenth century. Whisky manufacture spread to the USA and Canada, where individual styles developed. Other countries also started making whisky, and nowadays production may also be found in Australia, India, Japan, New Zealand, Pakistan, Spain, and the Czech Republic (Murray, 1998). The reader is referred to specific articles on the production of Scotch and Irish whiskies (Lyons, 1999), American whiskies (Ralph, 1999) and Canadian rye whisky (Morrison, 1999), along with more general texts (Lyons and Rose, 1977; Piggott *et al*., 1989; Piggott and Connor, 1995).

Whisky definitions may be found in the laws of most countries. These laws help protect the interests of the consumer, the business of the manufacturer, and the tax revenue of the state. The most important definitions are those of countries where that particular whisky originates. Many other countries either refer to the definition of the home country in their own regulations, or use words and phrases taken from the home definition. The major international

whisky brands come from Scotland, Ireland, the USA and Canada. The following sections review the definitions for these whiskies in their countries of origin.

Scotch whisky

Scotch whisky is made in Scotland from cereals, yeast and water only. There are two types of Scotch whisky; malt whisky and grain whisky. Scotch malt whisky is fermented from malted barley and the resulting alcohol concentrated in a batch distillation in copper pot stills. Scotch grain whisky is fermented from a mash containing other non-malted cooked cereals (such as barley, wheat or maize) and a smaller proportion of malted barley. The resulting alcohol is concentrated in a continuous distillation process; the most common design is known as the Coffey or Patent still. Both Scotch malt and grain new-make spirits are then reduced with water to approximately 65–70 per cent vol. alcoholic strength, and are matured in oak casks for at least three years. At the end of maturation the whisky is either blended or taken as a single distillery product, and is reduced further with water for bottling at a minimum 40 per cent vol. alcoholic strength.

Scotch whisky is defined in UK law in the Scotch Whisky Order 1990, made under the Scotch Whisky Act 1988. It is also defined in the related European Union Regulation 1576/89 (EEC, 1989), which defines all spirit drinks made in member states. Both the UK and EU laws define the process and not analytical parameters (with the exception of the minimum bottling strength of 40 per cent vol.).

The Scotch Whisky Order 1990

For the purpose of the Act, 'Scotch whisky' means whisky:

- which has been produced at a distillery in Scotland from water and malted barley (to which only the whole grains of other cereals may be added) all of which have been: (i) processed at that distiller into a mash; (ii) converted to a fermentable substrate only by endogenous enzymes; and (iii) fermented only by the addition of yeast;
- which has been distilled at an alcoholic strength by volume of less than 94.8 per cent so that the distillate has an aroma and taste derived from the raw materials used in, and the method of, its production;
- which has been matured in an excise warehouse in Scotland in oak casks of a capacity not exceeding 700 litres, the period of maturation being not less than three years;
- which retains the colour, aroma and taste derived from the raw materials used in, and the method of, its production and maturation; and
- to which no substance other than water and spirit caramel has been added.

The minimum alcoholic strength of Scotch whisky is specified at 40 per cent vol.

European Union Regulation 1576/89

Article 4(a) of the European Union Regulation 1576/89 describes whisky or whisky as a spirit drink produced by the distillation of a mash of cereals and:

- saccharified by diastase of malt contained therein, with or without other natural enzymes;
- fermented by the action of yeast;
- distilled at less than 94.8 per cent vol., so that the distillate has an aroma and taste derived from the raw materials used; and
- matured for at least three years in wooded casks not exceeding 700 litres in capacity.

The minimum alcoholic strength by volume of whisky in the Community is also 40 per cent. Annex II of the Regulation describes geographical designations for whisky produced in Europe as 'Scotch whisky', 'Irish whiskey' and 'Whisky Espanol'.

Irish whiskey

Like Scotland, Ireland also makes malt and grain whiskies using mixtures of malted and unmalted cereals. Irish malt whiskies are triple distilled in three pot stills, and Irish grain whiskies are continuously distilled on three column systems, making them lighter in character than many of their Scottish equivalents. Like Scotch whisky, the minimum maturation period is three years. Production and maturation may take place in the Irish State or Northern Ireland, and plants may currently be found in Cork and near Dundalk (in Eire), and in Bushmills in Northern Ireland. A legal definition may be found in the Irish Whiskey Act 1980, and also within EU Regulation 1576/89.

American whiskey

The Standards of Identity for Distilled Spirits are documented in the Code of Federal Regulations:

> Whiskey is an alcoholic distillate from a fermented mash of grain produced at less than 190° proof in such a manner that the distillate possesses the taste, aroma and characteristics generally attributed to whiskey, stored in oak containers and bottled at not less than 80° proof . . .

In particular, bourbon whiskey:

> . . . is whiskey produced at not exceeding 160° proof from a fermented mash of not less than 51 per cent corn and stored at not more than 125° proof in charred new oak containers . . . Straight bourbon whiskey has to be stored for a period of two years or more in charred new oak containers . . .

The other internationally popular American whiskey is from Tennessee, and that has a geographical designation.

Canadian whisky

Canadian whisky is defined in Canada under the Food and Drugs Act (1993):

> Canadian Whisky, Canadian Rye Whisky and Rye Whisky shall:
>   be a potable alcoholic distillate, or a mixture of potable alcoholic distillates, obtained from a mash of cereal grain or cereal grain products saccharified by the diastase of malt or by other enzymes and fermented by the action of yeast or a mixture of yeast and other micro-organisms.
>   be mashed, distilled and aged in small wood for not less than three years in Canada.
>   possess the taste, aroma and character generally attributed to Canadian whisky.
>   be manufactured in accordance with the requirements of the Excise Act and the regulations made there under; and
>   may contain caramel and flavouring.

Other sections relate to age claims and a definition of Highland whisky.

Whisky is the major category of distilled spirits worldwide. There are 35 brands of various 'whiskies' in the top 100 brands of distilled spirits (*Drinks International*, 2002). Each of these brands sells over one million cases annually in international and regional markets (Table 9.1).

Other whiskies and related products

Finally, some regional products are referred to as 'national whiskies'. Many of these are admixtures of neutral alcohol flavoured with a smaller proportion of a malt whisky or a similar product. As such these products do not qualify as 'whisky' under the regulations described above, owing to their use of one or more of the following components:

- Alcohol that has not been distilled at < 94.8 per cent vol. or < 190°US proof
- Alcohol that has not been fermented from cereals
- Alcohol that has not been subject to a minimum period of maturation
- Additives or flavourings.

It is clearly important that such 'national whiskies' do not claim the provenance of Scotch, Irish, American and Canadian whiskies. Analysis plays a key role in the identification of such products.

Composition and analysis of whisky

As already noted, whiskies contain many hundreds of congeners (Kahn, 1969, Maarse and Visscher, 1985a, 1985b). Whiskies are analysed for a

Table 9.1
Whisky brands selling over 1 million 9-litre cases in 2001, and their ranking order in the top 100 international and regional distilled spirits (*Drinks International*, 2002)

Ranking order	Brand	Category	Brand owner	Sales in 2001 (million cases)	Status
10	Johnnie Walker Red Label	Scotch whisky	Diageo	6.8	International
12	Jack Daniels Tennessee	American whiskey	Brown Foreman	6.4	International
13	J&B Rare	Scotch whisky	Diageo	6.3	International
16	Bagpiper	Indian whisky	UB Group	5.3	Regional
17	Jim Beam	Bourbon	JBB Worldwide	5.1	International
18	Ballantines	Scotch whisky	Allied Domecq	5.1	International
23	Willian Grant's	Scotch whisky	Willian Grant & Sons	4.0	International
25	Dewar's White Label	Scotch whisky	Bacardi Martini	3.7	International
26	McDowell's No.1	Indian whisky	UB Group	3.6	Regional
27	Officers Choice	Indian whisky	BDA Ltd	3.5	Regional
28	Johnnie Walker Black Label	Scotch whisky	Diageo	3.3	International
29	Crown Royal	Canadian whisky	The Seagram Co.	3.3	International
33	Chivas Regal	Scotch whisky	The Seagram Co.	3.0	International
34	Director's Special	Indian whisky	Shaw Wallace	3.0	Regional
38	7 Crown	American whiskey	The Seagram Co.	2.7	Regional
41	Suntory Kakubin	Japanese whisky	Suntory	2.6	Regional
43	Famous Grouse	Scotch whisky	Highland Distillers	2.5	International

46	Canadian Mist	Canadian whisky	Brown Foreman	2.4	International
48	Kerala Malt	Indian whisky	Shaw Wallace	2.3	Regional
50	Hayward's	Indian whisky	Shaw Wallace	2.3	Regional
58	Whisky DYC	Spanish whisky	Allied Domecq	2.0	Regional
59	Canadian Club	Canadian whisky	Allied Domecq	2.0	International
60	Bell's	Scotch whisky	Diageo	2.0	International
63	Cutty Sark	Scotch whisky	Berry Bros. & Rudd	1.9	International
66	Vat 69	Scotch whisky	Diageo	1.7	International
69	VO Canadian	Canadian whisky	The Seagram Co.	1.6	International
70	Clan Campbell	Scotch whisky	Pernod Ricard	1.6	International
77	Teachers	Scotch whisky	Allied Domecq	1.5	International
79	Clan MacGregor	Scotch whisky	Willian Grant & Sons	1.4	Regional
81	Suntory Red	Japanese whisky	Suntory	1.4	Regional
82	Jameson	Irish whiskey	Pernod Ricard	1.4	International
87	Windsor Canadian	Canadian whisky	JBB Worldwide	1.3	International
88	Old Tavern	Indian whisky	Shaw Wallace	1.3	Regional
97	Early Times	American whiskey	Brown Foreman	1.1	Regional
99	Suntory Old	Japanese whisky	Suntory	1.0	Regional

wide range of purposes, from research and process improvement to routine production quality assurance. Nowadays, with the exception of alcoholic strength measurement and certain process monitoring techniques, the whisky analyst relies heavily on gas and liquid chromatography as the techniques of choice. Chromatography offers advantages in selectivity, sensitivity and speed over many of the older spectrophotometric and titrimetric methods it replaces. Most of the well-known chromatographic detectors have a role, from flame ionization to mass spectrometric detection for gas chromatography, and ultraviolet to fluorescence detection for liquid chromatography. Sample preparation has been minimized, and direct injection of the whisky sample in the presence of an appropriate internal standard is used wherever possible. This section reviews the current methods available, the context in which they are used, and typical results obtained.

Alcoholic strength measurement

Alcoholic strength measurement is the key quantitative measurement in whisky production, as it helps measure the efficiency of the overall carbohydrate conversion process, determines the excise tax paid to the government, and ensures that consumers receive the alcohol declared on the product's label. Alcoholic strength is normally defined as the concentration of ethyl alcohol in a solution by volume at a specific temperature. Alcohol concentration is normally expressed as a percentage or as degrees proof, where pure alcohol at 100 per cent by volume is equivalent to $200°$ US proof. Methods for alcoholic strength determination are published in the *AOAC Official Methods of Analysis Handbook* (AOAC, 2000) and in the EC Commission Regulation No 2870 (2000).

Two versions of the measurement are used; real and apparent alcoholic strengths. Real alcoholic strength is the percentage alcohol by volume in the distillate of a test sample, so that any dissolved solids are removed. This gives the true result. Apparent alcoholic strength is the percentage alcohol by volume in a test liquid without prior distillation. Here, the true result is obscured by dissolved solids. The presence of dissolved solids increases the density of the sample, and results in an apparent alcoholic strength measurement lower in value than the corresponding real alcoholic strength measurement. The difference between real and apparent alcoholic strength is known as the 'obscuration':

Obscuration, per cent vol. = real alcoholic strength − apparent alcoholic strength

The amount of dissolved solids in most whiskies is low. Obscuration is normally less than 0.2 per cent vol. in Scotch whisky, and therefore distillation is not normally required before taking a strength measurement for revenue or trading purposes. The United States Alcohol Tobacco and Firearms (ATF) regulations require sample distillation prior to alcohol strength mea-

surement when dissolved solids exceed 600 mg per 100 ml. Between 400 and 600 mg dissolved solids per 100 ml, the obscuration is added to the determined proof (ATF Code of Federal Regulations 27, 30, 31: Determination of Proof).

Alcoholic strength measurement is usually based on the density of the solution. Pure ethanol has a density of 789.24 kg/m^3 at 20°C, while water has a density of 998.203 kg/m^3 at 20°C. Ethanol/water mixtures will have a density between these two values; the lower the density the higher the alcoholic strength. The three main techniques based upon liquid density involve the use of hydrometers, pycnometers and electronic densitometers.

Hydrometers have traditionally been the method of choice for alcoholic strength measurement in the production environment. The hydrometer is a calibrated, weighted device that floats in the test liquid to a depth corresponding to the density of the liquid. The reading is taken where the liquid intersects the hydrometer scale. The measurement is normally precise to up to ±0.1 per cent vol., depending on the hydrometer used and the volume of liquid under test.

More precise alcoholic strength measurements are obtained using pycnometers and electronic density meters. A pycnometer is a small (usually Pyrex ® glass) vessel of typically 100 ml volume, which is used to determine the specific gravity/density of the test liquid. Alcoholic strength is then read from tables that give alcoholic strength by volume as a function of the density of water alcohol mixtures. An international table has been adopted by the International Legal Metrology Organization (OIML) for this purpose. The procedure is highly skilled and slow, and accurate temperature control and measurement is required. Results are precise to at least ±0.1 per cent vol.

During the 1980s, electronic densitometry became the preferred method for alcoholic strength determination in laboratories, larger distilleries and bottling plants. Measurement is based on resonant frequency oscillation of a sample in an oscillation cell. The oscillation frequency of the cell is modified by the added mass of the test sample, and this can be used to determine its density and corresponding alcoholic strength. Small test volumes are required (< 5 ml) and precision up to ±0.1 per cent vol. is possible. Once calibrated, instruments are stable and can be automated. Commercial instruments are made by Anton Parr ® and Kyoto ®.**48

More recently, near-infrared spectroscopy has been applied to the real alcoholic strength measurement of obscured liquids. This approach, as developed using the Foss Rapitech ® instrument, eliminates the need for distillation prior to analysis, and is particularly useful for alcoholic strength measurement in obscured liquids such as whisky-based liqueurs and lower strength ready-to-drink formulations.

Finally, gas chromatography may be used to separate the test liquid, with a specific peak for ethanol from which a value for real alcoholic strength can be calculated. Results are typically precise to ±1 per cent vol. The method can be automated, but is not precise enough for revenue purposes.

Major volatile congeners

After ethanol, the major congeners in whisky are the higher alcohols, namely n-propanol, isobutanol, and 2- and 3-methylbutanols. Much useful work on these congeners in various distilled spirits has been published (Duncan and Philp, 1966; Kahn, 1969; Shoeneman and Dyer, 1973). The gas chromatographic analysis for these congeners usually also includes separation of methanol, acetaldehyde and ethyl acetate. All these congeners are formed during fermentation, and their final concentrations are influenced particularly by the distillation and blending processes. The major volatile congeners are analysed at the mg/l level in one direct injection gas chromatographic separation, using a polar stationary phase on a packed or capillary column (Martin *et al.*, 1981). Common internal standards include 1- or 3-pentanol, and the method has been validated in comprehensive inter-laboratory trials (Kelly *et al.*, 1999).

A typical higher alcohol chromatogram for a Scotch blended whisky is shown in Figure 9.1 (using Carbowax ® 20M on Carbopak ® B as stationary phase), and quantitative results for typical Scotch, Irish, bourbon and Canadian whiskies are given in Table 9.2. The results show that Scotch grain whisky from the continuous Coffey still distillation contains very few congeners after isobutanol, while Scotch malt whisky from a double pot still distillation is much richer in the less volatile congeners that elute after isobutanol. The less volatile congeners, particularly 2- and 3-methyl-
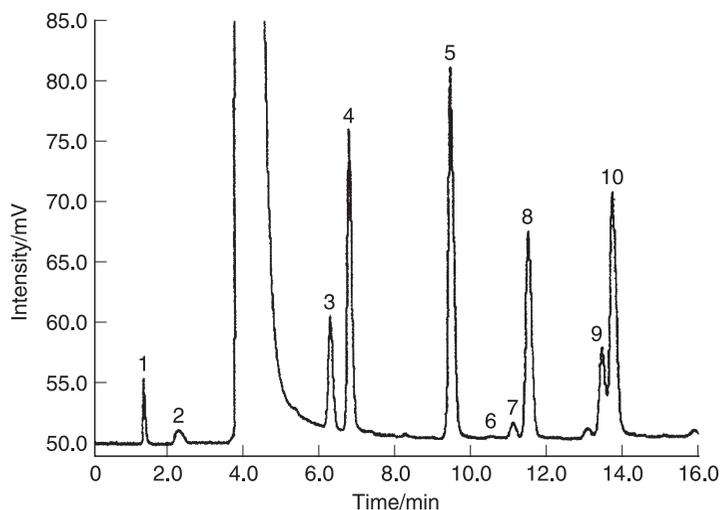

Figure 9.1
Typical higher alcohol gas chromatogram for a blended Scotch whisky. Peaks 1, acetaldehyde; 2, methanol; 3, ethyl acetate; 4, propanol; 5, isobutanol; 6, butanol; 7, diethyl acetal; 8, internal standard; 9, 2-methylbutanol; and 10, 3-methylbutanol. (Aylott *et al.*, 1994, reproduced by permission of the Royal Society of Chemistry.)

Table 9.2

Major volatile congener concentrations (g/100l abs. alc.) in single samples of Scotch malt, grain and blended whiskies, and Irish, American and Canadian whiskies

Whisky type	Acetaldehyde	Methanol	Ethyl acetate	n-Propanol	Isobutanol	2-methylbutanol	3-methylbutanol	2- and 3-methylbutanol	Ratio 1	Ratio 2
Scotch malt whisky	17	6.3	45	41	80	62	114	176	2.2	2.8
Scotch grain whisky	12	8.5	18	72	68	8	15	23	0.3	2.8
Scotch blended whisky	5.4	8.9	23	55	62	26	46	72	1.2	2.8
Irish whiskey	4.1	10	13	28	15	18	32	49	3.1	2.8
Kentucky bourbon whiskey	15	17	89	28	160	143	242	385	2.4	2.7
Canadian whisky	3.3	7.9	7.1	6.2	6.9	7	9	16	2.3	2.2

Ratio R1 = 2- and 3-methyl butanol/isobutanol.
Ratio R2 = 3-methylbutanol/2-methylbutanol.

butanol and above, are eliminated from grain spirit in the rectifier section of the Coffey still, and are recovered as fusel oils. Blended Scotch and Irish whiskies have congener profiles representing the various malt and grain whiskies used in their blends. Bourbon whiskey is very rich in congeners, as there is little if any rectification in the bourbon distillation process beyond the beer still, in the doubler. Canadian whiskies, being blends of matured grain neutral spirits and a bourbon-style distillate, have relatively low congener concentrations compared to the other whiskies examined. Of particular note are the relatively low methanol concentrations encountered in all whiskies fermented from cereals (normally $<25\,\text{g}/100\,\text{l}$ abs. alc.) and the ratios of 3-methylbutanol/2-methylbutanol (typically between 2.5 and 3.5). Other trace congeners that may be detected in this analysis also include n-butanol and acetic acid.

The normal concentration unit used in whisky congener analysis is grams per 100 litres absolute alcohol ($\text{g}/100\,\text{l}$ abs. alc.). Thus congener concentration is related to the volume of pure alcohol, and not simply the volume of sample liquid. This concentration unit at first appears strange, but it proves very useful in that it can relate to a whisky that will occur at many different alcoholic strengths during its manufacturing process. For example, a Scotch grain spirit may be distilled at approximately 94.6 per cent, matured at 65 per cent, and then blended and finally bottled at 40 per cent vol.

This major volatile congener analysis is the most widely used gas chromatographic method applied to whisky, and finds applications throughout the lifecycle of the product. For example, it may be used to monitor the efficiency of rectification in the continuous stills used to distil Scotch grain spirit and Canadian grain neutral spirits (GNS), in competitor product analysis to determine the percentage malt whisky in a blended Scotch whisky, and in consumer protection activity to confirm brand authenticity. The spectrophotometric and titrimetric methods from the pre-chromatography era (Singer and Styles, 1965; Shoeneman and Dyer, 1973) are now all but redundant, and are only occasionally required for use in supporting dated regulatory requirements. When total esters are determined by these older methods as ethyl acetate, over 50 per cent of the total esters is ethyl acetate. Similarly, the major volatile acid is attributable to acetic acid. If required, methods for total aldehydes, esters, fusel oil, furfural, colour and extract can be located in the *AOAC Official Methods of Analysis Handbook* (AOAC, 2000) and *Methods for the Analysis of Potable Spirits* (Research Committee on the Analysis of Potable Spirits, 1979).

Trace congeners

The next major groups of congeners are longer chain alkyl alcohols, aldehydes and esters at low and sub-mg/l levels. A few of these congeners present at higher concentrations can be determined by direct injection on a packed temperature programmed polar column (such as Carbowax Ⓡ 20M

on Chromosorb Ⓡ B). However, the best separations are achieved on polar capillary columns using an organic solvent extract of the test liquid. Chlorinated fluorocarbons (CFCs) proved ideal extracting solvents until their withdrawal from use on environmental grounds. Pentane is also a good extracting solvent (with a 10-ml sample extracted into 1-ml pentane). A capillary column chromatogram of a typical blended Scotch whisky extract is shown in Figure 9.2.

Capillary column systems can use both vaporizing splitless and on-column injection. More recently, solid-phase micro-extraction (SPME) has developed into a reliable and straightforward method of sample preparation and introduction prior to capillary column separations (Lee *et al.*, 2001a). These high-
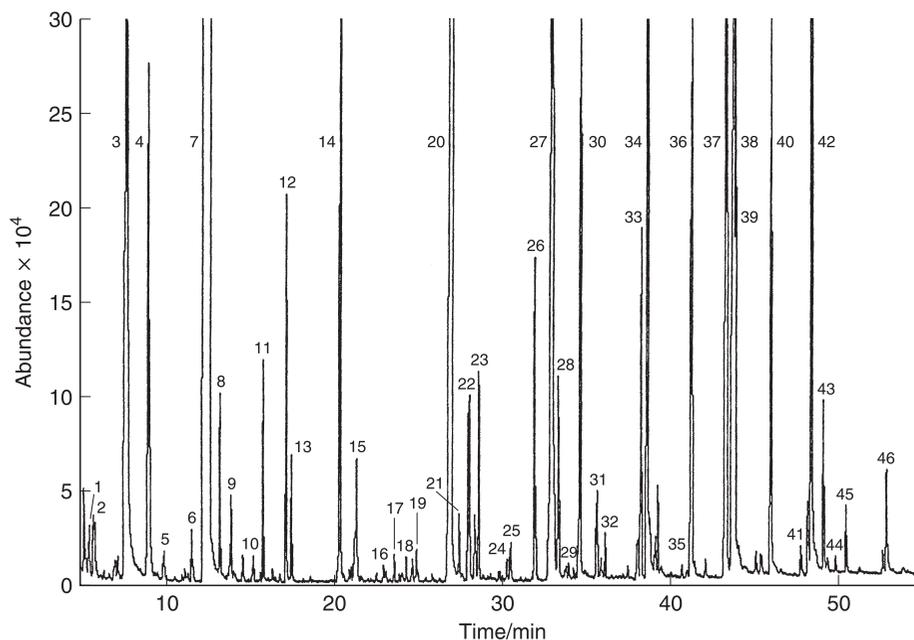

Figure 9.2
A capillary column gas chromatogram of a typical blended Scotch whisky extract. Peaks 1, ethyl butanoate; 2, propanol; 3, isobutanol; 4, isoamyl acetate; 5, butanol; 6, 3-ethoxypropanal; 7, isoamyl alcohol; 8, ethyl hexanoate; 9, diethoxypropane; 10, furfuryl formate ; 11, triethoxypropane; 12, ethyl lactate; 13, hexanol; 14, ethyl octanoate; 15, furfural; 16, acetyl furan; 17, benzaldehyde; 18, ethyl noonanoate; 19, ocatanol; 20, ethyl decanoate; 21, isoamyl octanoate; 22, diethyl succinate; 23, internal standard; 24, ethyl undecanoate; 25, decanol; 26, β-phenyl ethyl acetate; 27, ethyl dodecanoate; 28, isoamyl decanoate; 29, *trans* oak lactone; 30, β-phenyl ethanol; 31, *cis* oak lactone; 32, dodecanol; 33, ethyl hexadecenoate; 34, octanoic acid; 35, ethyl pentadecanoate; 36, tetradecanol; 37, ethyl hexadecanoate; 38, decanoic acid; 39, ethyl hexadecenoate; 40, hexadecanol; 41, ethyl octadecanoate; 42, dodecanoic acid; 43, ethyl octadecenoic acid 44, vanillin; 45 , β-phenyl ethyl butanoate; and 46, tetradecanoic acid. (Aylott *et al.*, 1994, reproduced by permission of the Royal Society of Chemistry.)

resolution separations are often linked to mass spectrometric detection with the MS operating in electron impact mode.

Maturation congeners

Whisky maturation at a molecular level involves processes of congener addition, congener reduction and congener production (Philp, 1986). During these processes the pungent and harsh characteristics of new-make spirit (new distillate) diminish and the smoother, more complex character of mature whisky develops.

Scotch and Irish whisky maturation casks are normally made from Spanish and American oak. Bourbon whiskey casks are made only from new charred American oak and are used only once. This results in a flourishing market for used American oak barrels, with the Canadian and Scotch whisky industries being major buyers.

Maturation warehouses in Scotland and Ireland tend to be cool all year around, while those in the USA and Canada can become very hot in the summer and very cold in the winter. Some bourbon whiskey companies heat their warehouses in winter in order to accelerate their maturation process. As the character of the spirit develops, the volume in each cask decreases by 1 to 2 per cent per year due to evaporation.

The process of congener addition is one of extraction from the oak cask into the liquid through interaction of ethanol with wood lignin (Reazin, 1979). The resulting congeners are known as lignin degradation products (LDPs) and polyphenolics, which may be determined by HPLC (Lehtonen, 1983a, 1983b) and oak lactones, which may be determined by capillary column gas chromatography. Whilst new-make whiskies are clear liquids prior to the start of maturation, they acquire varying amounts of colour and particles of charred wood from the oak casks during the maturation process.

Direct-injection reversed-phase gradient elution HPLC with ultraviolet and/or fluorescence detection is the most commonly used analysis within the maturation process (Aylott *et al.*, 1994). This analysis can be used to quantify LDPs, which include gallic acid, vanillic acid, syringic acid, vanillin, syringealdehyde, coniferaldehyde, ellagic acid and scopoletin, as well as furfural (found in Scotch malt spirit) and 5-hydroxymethyl furfural (which is mainly associated with spirit caramel). A typical chromatogram is shown in Figure 9.3.

The congener reduction process involves volatilization (such as the loss of dimethyl sulphide), adsorption onto the charred wood, and oxidation of carbonyl compounds. The congener production process involves oxidation to form acetals, esterification to form more esters, and hydrolysis to form quinones.

Sulphur-containing congeners, such as the volatile dimethyl sulphide (DMS), dimethyl disulphide (DMDS) and less volatile dimethyl trisulphide (DMTS), may be determined by temperature programmed capillary column gas chromatography with flame photometric detection (Beveridge, 1990).
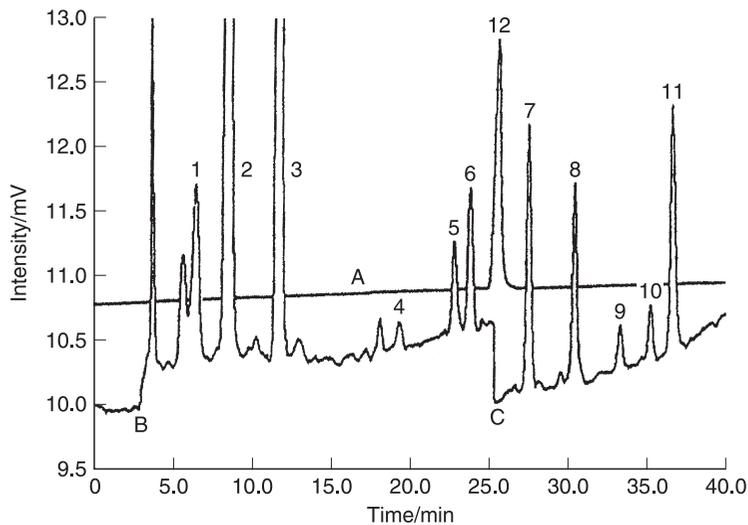
Figure 9.3
A high performance liquid chromatogram of cask-related and other congeners in blended Scotch whisky with (A) fluorescence detection (excitation at 345 nm and emission at 450 nm), and ultraviolet detection at 280 nm (B) and 340 nm (C). Peaks 1, gallic acid; 2, 5-hydroxymethyl furfural; 3, furfural; 4, vanillic acid; 5, syringic acid; 6, vanillin; 7, syringealdehyde; 8, internal standard; 9, coniferaldehyde; 10, synapaldehyde; 11, ellagic acid; and 12, scopoletin. (Aylott *et al*., 1994, reproduced by permission of the Royal Society of Chemistry.)

DMTS is an important flavour compound in Scotch malt whiskies. These ultra-trace compounds were concentrated from the test sample by a dynamic headspace concentration technique onto Chromosorb ® 101 prior to thermal desorption, chromatographic separation and detection at the low µg/l level.

Whisky age

The concentrations of oak-derived congeners in a given cask of whisky increase with maturation time. It is therefore possible to start to draw a graph plotting maturation congener concentrations against age. However, there are a great variety of barrel types in use, from the new charred oak used in the bourbon whiskey process (which will produce relatively high concentrations of addition maturation congeners) to the second or third refill cask used for a Scotch grain whisky (which will result in less congener addition). Casks may also be rebuilt and rejuvenated. This means that age determination on the basis of maturation congener concentrations is imprecise. However, the chromatographic profiles for maturation congeners are consis-

tent, and can prove useful in checking false authenticity claims in spurious products where use of wood flavour additives is suspected.

Natural ^{14}C in the atmospheric carbon dioxide is absorbed by metabolism into all plants, including the cereals used for whisky manufacture. The natural levels of ^{14}C increased between 1945 and 1960, corresponding to the start and end of atmospheric nuclear testing (Baxter and Walton, 1971), and have now fallen back to near the pre-1945 levels. Analysis of the ^{14}C levels in ethanol concentrated from whiskies samples has been used to estimate the year in which the cereal was grown, and then to relate this year to age. Analytical precision is limited, and this obviously reduces the accuracy of the resulting date.

pH, residues, ash, anions and cations

The natural pH of whisky at 40 per cent vol. bottling strength is normally in the range 4–4.5. This mild acidity is as a result of trace organic acids, with acetic acid being the main component. Calibration solutions for the pH meter should be prepared in 40 per cent ethanol. The pH of whisky can be made higher when the maturing spirit has been reduced to bottling strength with softened rather than demineralized water.

Whisky residues are low (typically $< 0.2\,g/100\,ml$), and generally represent non-steam volatile material derived from the cask during maturation. Samples are prepared for this analysis over a steam bath. Ash values are much lower (typically $< 0.02\,g/100\,ml$), and represent involatile inorganic compounds remaining when the test sample is taken to dryness in a furnace. The ash will include trace metals such as calcium, magnesium, sodium and potassium (all typically at low mg/l levels), which are derived from the water and casks used in the whisky process. These trace metals may be analysed in whisky directly by atomic absorption spectroscopy with flame atomisation, or by ion chromatography with electrochemical detection.

Various trace sugars are also present, and are derived as cask extracts and low molecular weight carbohydrates present in spirit caramel (when used). Concentrations in Scotch whisky are typically considerably less than 200 mg/l. A twelve-year-old bottled deluxe blended Scotch whisky was found to contain 50 mg/l glucose, 50 mg/l fructose and 20 mg/l sucrose. Test samples may be analysed by direct injection ion exchange chromatography with conductivity detection, or by gas chromatography (after taking the sample to dryness and separation of the sugars as their trimethyl silyl ethers derivatives). Other sugars derived from carbohydrate extracted from oak wood may be detected at much lower relative concentrations.

Volatile phenolic congeners

A range of volatile phenolic compounds is found in whiskies where peat is used in the kiln-drying of the malted barley. These flavour congeners are

present in many Scotch malt whiskies, particularly those from Islay, an island with eight distilleries off the west coast of Scotland. In addition, volatile phenols are found in many of the blended Scotch whiskies made with such malt whiskies.

Volatile phenol congeners are best determined by direct-injection reversed-phase gradient elution HPLC with fluorescence detection in the presence of an internal standard such as 2,3,5-trimethyl phenol (Aylott *et al.*, 1994). This analysis detects phenol, guaiacol, isomers of cresol and xylenol, eugenol, and other phenol derivatives (Figure 9.4). Alternatively, many of these compounds may be separated by capillary column gas chromatography and enhanced sensitivity and selectivity obtained as their 2,4-dinitrophenyl derivatives (Lehtonen, 1983b).
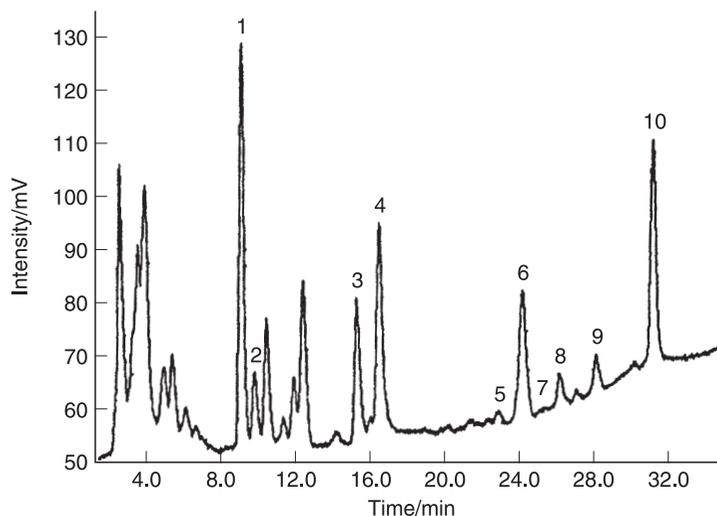
Figure 9.4
A high performance liquid chromatogram of volatile phenolic congeners in blended Scotch whisky with fluorescence detection (excitation at 272 nm and emission at 298 nm). Peaks 1, phenol; 2, guaiacol; 3, *m-, p*-cresol; 4, *o*-cresol; 5, 3,5-xylenol; 6, 4-ethyl phenol (2,5-xylenol); 7, 4-ethyl guaiacol; 8, 2-ethyl phenol; 9, eugenol; and 10, internal standard. (Aylott *et al.*, 1994, reproduced by permission of the Royal Society of Chemistry.)

Sensory analysis

Sensory analysis is the key quality control technique for the whisky blender and other process colleagues. Researchers investigating those congeners responsible for the unique characteristics of each whisky have also developed sophisticated sensory methods. Finally, the informed whisky enthusiast seeks an understanding of the complexities of whisky flavour (Hills, 2000).

At the process end, new-make spirits (also known as 'new distillate' in the USA) are monitored for conformance to specification before being put into casks for maturation. Similarly, as single casks of mature whisky are selected for disgorging ('dumping' in the USA), the blender will check that each specific cask is contributing the correct characters for the blend. These samples are normally assessed by nose only. New-make spirit or whisky (25 ml) is typically placed in a tulip-shaped nosing glass with an equal volume of demineralized or distilled water in order to bring alcoholic strength down to approximately 20 per cent vol. The nosing glass is covered by a watch glass and is left to equilibrate for about 30 minutes to allow a build-up of congeners in the headspace above the liquid. The samples are then nosed by the blender and/or a trained sensory panel, and their colour checked. Any abnormalities are recorded and action is taken to resolve any unsatisfactory casks of whisky. At this stage the sensory language used (in terms of the number of descriptors) is normally limited and specific to the particular process being monitored. Sensory assessment after blending is designed to ensure that the product has consistent characteristics that meet the requirements of the brand and correspond to a reference standard. A trained panel will nose and sometimes taste the samples, often using triangle and duo–trio sensory tests in order to validate the results statistically.

At the research end, descriptive terminology has been developed and specific molecules/congeners associated with specific character (Swan *et al*., 1981). This important work at the Scotch Whisky Research Institute (known at that time as Pentlands Scotch Whisky Research) resulted in the Scotch whisky flavour wheel. Furthermore, the effluent from a capillary gas chromatographic column may be spilt into two streams, with one stream going to a mass spectrometer (or other detector) and the other to a nosing point, where a trained panellist makes sensory comments. This method is commonly known as GC–sniff analysis, and can help identify those congeners contributing to particular odour characteristics. GC-sniff can also be useful in detecting trace contaminants whose peaks are hidden in complex chromatograms of whisky and other multi-component matrices.

The Scotch whisky flavour wheel has recently been revised, and reference compounds have been defined for assessor training purposes against each flavour term (Table 9.3) (Lee *et al*., 2001b). The revised flavour wheel is shown in Figure 9.5. The primary tier terms include a range of common descriptors for whisky, including: peaty, grainy, grassy, fruity, feints, woody, sweet, stale, sulphury, cheesy, and oily.

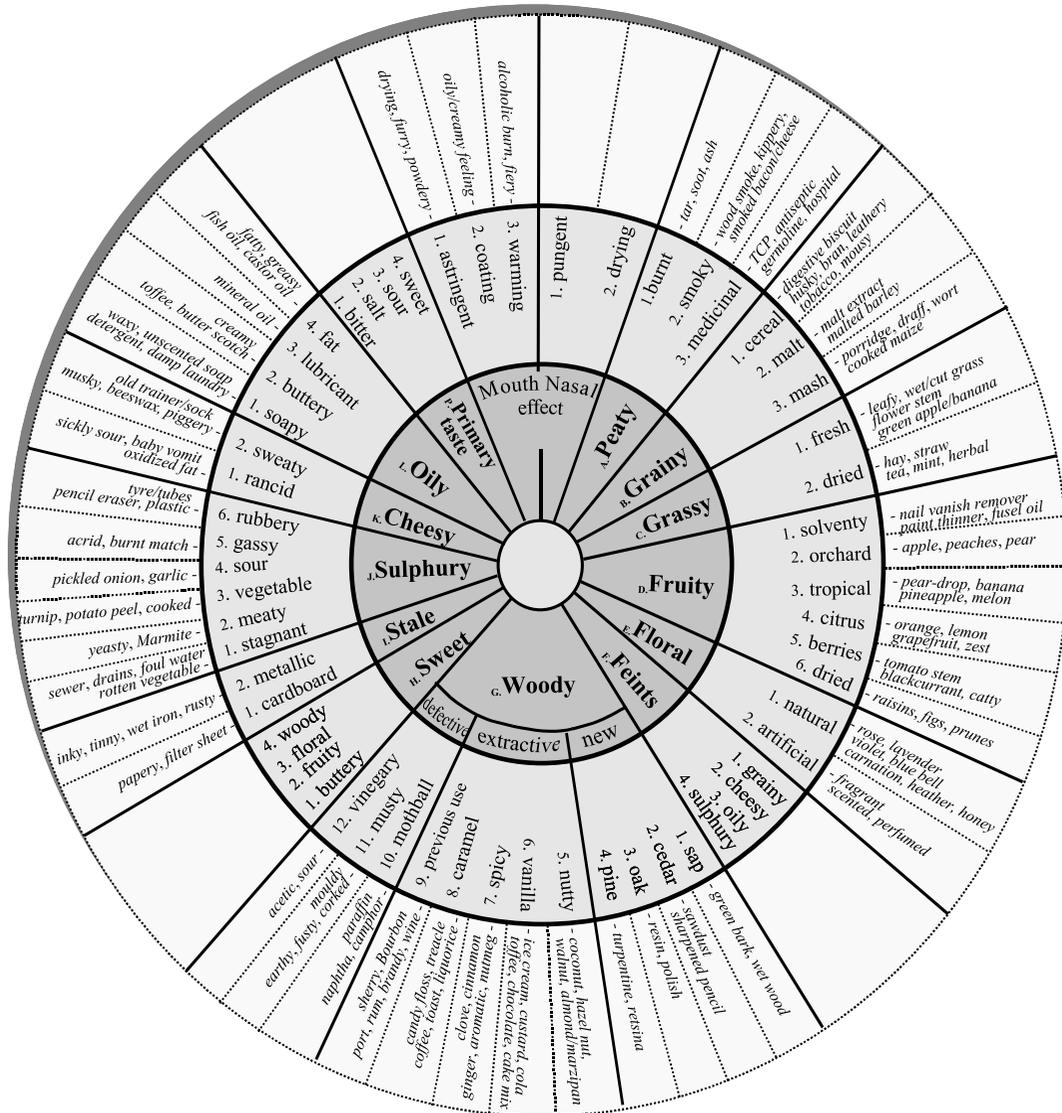# Revised Scotch Whisky Flavour Wheel
For Industrial Purposes

Figure 9.5
The revised Scotch whisky flavour wheel. (Lee *et al.*, 2001b, reproduced with permission from *Journal of the Institute of Brewing*.)

Table 9.3

Whisky descriptors and reference compounds (Lee *et al.*, 2001b, reproduced with permission from *Journal of the Institute of Brewing*)

Code	Attributes	Reference compounds	Concentration (mg/L)
N.1	*Pungent*	Formic acid[a]	10×10^3
A.1, 2	*Burnt/smoky*	Guaiacol[b]	27
A.3	*Medicinal*	o-Cresol[d]	1.75
B.2	*Malty*	Malted barley[a], 2-and 3-Methyl butanal, 4-Hydroxy-2(or 5)-ethyl-2 (or 5)-methyl-3(2H) furanone, 4-hydroxy-2,5-dimethyl-3 (2H) furanone	– 0.6 (2-methyl butanal)[e], 1.25 (3-methyl butanal)[e]
C.1	*Grassy*	Hexanal[b] cis-3-Hexen-1-ol[a]	5 1.00×10^3
D.1	*Solventy*	Ethyl acetate[d], 2-Methyl propan-1-ol[a]	1.12×10^3 1.00×10^3
D.2	*Fruity (apple)*	Ethyl hexanoate[b]	2
D.3	*Fruity (banana, pear-drop)*	iso-Amyl acetate[b]	7
D.5	*Berry* *Catty*	Thiomenthone[d] Thiomenthone[d] Sodium sulphide + mesityl oxide[a]	3×10^{-3} 1.26 100 each
E.1	*Floral (Natural – rose – violet)*	Phenyl ethanol[b]	1.52×10^3 $>3 \times 10^{-3}$
	Floral (Artificial – scented, perfumed)	α-, β-Ionone[d] Geraniol[b]	19
G.5	*Nutty (coconut)*	Whisky lactone[b]	266
	Marzipan	Furfural[b]	839
G.6	*Vanilla*	Vanillin[b]	43
G.7	*Spicy*	4-Vinyl guaiacol[b]	71
	Spicy (clove)	Eugenol[a,b]	1–55
G.8	*Caramel (candy floss)*	Maltol[b]	1.14×10^3
G.10	*Mothball*	Naphthalene	$>8 \times 10^3$
G.11	*Mouldy*	2,4,6-Trichlorocanisole[a]	10
	Earthy, musty	Geosmin, 2-methyl iso-borneol	
G.12	*Vinegary*	Acetic acid[b]	5.32×10^3
I.1	*Cardboard*	2-Nonenal[d]	0.08
J.1,6	*Stagnant, rubbery*	Dimethyl tri-sulphide (DMTS)[c]	3
J.2	*Yeasty* *Rotten egg* *meaty*	Hydrogen sulphide (H$_2$S)[c] Hydrogen sulphide[c] Methyl (2-methyl-3-furyl) disulphides[d]	–> 0.02 –> 0.14
J.3	*Vegetable (sweet corn, cooked cabbage)*	Dimethyl sulphide (DMS)[c]	>0.6
J.5	*Gassy*	Ethanethiol[c]	>0.072
		3-Methyl-2-butene-1–thiol[c]	$>7.2 \times 10^{-4}$
K.1	*Rancid*	n-Butyric acid/ethyl butyrate[d]	>2
	Sweaty	Iso-Valeric acid[b]	2
L.	*Oily*	Heptanol[a]	1
L.1	*Soapy*	Ethyl laurate[b]	12
		1-Decanol[a]	100
L.2	*Buttery*	Diacetyl[b]	0.1

[a] in 23% ethanol solution; [b] in 23% grain whisky; [c] in lager; [d] >threshold; [e] threshold in beer.

For reference papers to standards please see the original paper "Origins of Flavour in Whiskies and a Revised Flavour Wheel: A Review" by K.-Y.M. Lee, A. Paterson, J.R. Piggott and G.D. Richardson, published in 2001 in the Journal of the Institute of Brewing, Vol 107 (5) pp 287–313.

Quality assurance and analysis in the whisky production process

Malting, fermentation, distillation and maturation

Distillers require cereals with specific properties, such as low nitrogen content, known moisture content and appropriate malting characteristics. Malting barley varieties may require authentication. Potential distillery yield may be predicted by testing fermentation efficiencies in laboratory trials, and an array of techniques are available for distillery process troubleshooting (Hardy and Brown, 1989). Further routine process analyses during fermentation, distillation and maturation are normally limited to sensory assessments and alcoholic strength measurement.

Blending and bottling

The main analysis (along with sensory assessment) in the blending and bottling process is alcoholic strength measurement, and modern facilities employ electronic density measurement.

The quality of the water used for final reduction to bottling strength is very important. The town water supply is normally used, but it is usually subjected to a demineralization and carbon and ultraviolet light treatment process at the bottling plant in order to reduce trace anions and cations to a minimum and eliminate the risk of any off-odours. Conductivity measurement is used to monitor the water treatment process and the characteristics of bottled product in association with pH measurement.

Colour consistency is also an important quality parameter, particularly to those whiskies that include addition of trace spirit caramel for colour standardization. Colour is normally monitored by visible spectrophotometry at a wavelength of around 500 nm. The resulting 'tint' is usually quoted as the absorbance at a chosen fixed wavelength $\times 100$.

Whiskies are usually filtered through both coarse and fine sheets or cartridge filters in order to ensure a clear, bright product. Chill filtration is often used, where the liquid is chilled before filtration in order to achieve enhanced stability under prolonged cold environmental storage conditions. Clarity or turbidity is measured on a nephelometer using instruments made by Sigrist ® and Hach ®.**49

Whisky stability

Once in the bottle, whisky is a very stable product. Providing the closure gives a good seal on a glass bottle, alcoholic strength will remain constant and the product character will not change. Storage at warm temperatures can result in slightly elevated acetaldehyde and ethyl acetate concentrations. If the closure is loose, then ethyl acetate and acetaldehyde concentrations will diminish through volatilisation, and alcoholic strength will be lost. Stability testing

with Scotch whisky packaged in polyethylene terephthalate (PET) 5-cl bottles over a two-year test period showed that while sensory properties remained stable, alcoholic strength slightly increased due to preferential migration of water through the bottle wall. Ethylene terephthalate cyclic trimer was also detected by HPLC as a trace migrant from PET into spirit at the sub-µg/l level (Aylott and McLachlan, 1986).

Whilst filtered whisky is normally a clear, bright product, two forms of flocculation have occasionally been experienced in Scotch whiskies. The first form is known as 'reversible floc', and can form when whisky is stored for prolonged periods at very cold temperatures (as may be encountered in cold winter transit). The whisky develops a haze, which disappears when the liquid is warmed and shaken. The main congeners detected in reversible floc are the ethyl esters of long chain fatty acids and larger alkyl esters, both detectable by capillary column GC–MS. As whiskies are produced for distribution around the world to a wide range of climatic conditions, it has been found that reversible floc formation may be minimized by chill filtering whisky prior to bottling. This process reduces the concentrations of reversible floc-forming material, and has no effect on product character.

The second form is known as 'irreversible floc'. This shows itself as very small, hair-like crystals of calcium oxalate, which slowly form and settle in the whisky when natural low mg/l concentrations of oxalic acid in the whisky react with similarly low concentrations of calcium ions. Oxalic acid may be determined by ion chromatography and calcium by flame atomic absorption spectrophotometry. Irreversible floc formation is eliminated by ensuring that final calcium concentrations are kept to a minimum by demineralizing the water used for final reduction. Tennessee whiskies, whose process includes percolation of new distillate through maple charcoal prior to maturation, also pick up calcium from the wood, and levels may be reduced using an ion exchange treatment.

Off-odours as contaminants in whisky

Off-odours are occasionally reported in bottled product. The more common off-odour issues result from whisky being transported and stored in unsuitable environmental conditions. For example; transit and storage close to very smelly chemicals can result in odour ingress into the bottle. Bottles capped with roll-on pilfer-proof (ROPP) closures are most resistant to odour ingress. In each of the examples described below, GC–sniff used in parallel with GC–MS helped to identify the offending contaminants.

Ingress of naphthalene odours (mothball smell) has been detected on various occasions as a contaminant in consumer complaint samples returned from distant markets when product had been stored under unsuitable environmental conditions. The sensory threshold of naphthalene in whisky is very low (typically at µg/l levels in product), and its presence may be quantified by HPLC with fluorescence detection or GC–MS with selected ion monitoring. Musty-smelling compounds can also be encountered as contaminants in

whisky. The first example is 2,4,6-trichloroanisole (TCA), a particularly smelly compound that has been reported on many occasions (Saxby, 1996). TCA contamination at the sub-µg/l level has been found when good quality bottled product was stored in hot, wet and humid climatic conditions such as found in southern Asia. It was thought that trace trichlorophenols in the pulp used to make cartons and cases underwent microbiological degradation to form TCAs, which then entered the product through the closure/bottle interface. The second musty-smelling example is that of geosmin contamination resulting from microbial activity on cereals stored under damp conditions prior to their being taken for fermentation. Trace µg/l concentrations of this musty/earthy-smelling compound were carried through fermentation, distillation and maturation to contaminate the resulting whisky.

Consumer issues

Analysis can prove very useful in the examination of samples returned by consumers as complaints. Considering the enormous number of whisky bottles sold globally, the number of consumer complaints is usually very small. This is due to the inherent stability of whiskies and the very high quality standards applied throughout whisky manufacturing processes. The first thing that a quality laboratory has to do on receipt of a consumer complaint is to confirm its validity and the likely cause. Valid consumer complaints may be divided into two categories; those resulting from packaging and/or those from liquid issues. Complaints in the packaging category normally arise from quality issues on the production line or damage caused during distribution.

Packaging line issues may be minimized by employing appropriate standards and quality assurance procedures in the supply of materials to the manufacturing process. Modern glass bottles are treated at the hot end of the glass-making process with tin oxide in order to toughen the glass, and at the cold end of the process with surfactants and oleic acid in order to give the glass resistance to scuffing during handling. Over-application of oleic acid can give rise to contamination by small, oily globules of the acid in product. Many manufacturers apply codes to their bottles that enable product traceability back to specific production batches, or to production dates and time of bottling. These lot codes are usually found near the neck of the bottle, on the side of the bottle near its base, or on the glass behind a label.

Sometimes a consumer may inadvertently contaminate whisky with material that renders the whisky unstable. As an example, traces of milk result in a heavy precipitate of insoluble milk and whisky components. This contamination can be characterized through the detection of lactose (from milk) in the supernatant and dairy fats in the precipitate (analysed as their methyl esters after derivitization with boron trifluoride/methanol).

In a similar way, low µg/l concentrations of iron react with maturation congeners and cause a green discoloration, which is virtually black at the 10 µg/l iron level. Rust in any post-distillation process can be very damaging,

and process engineers will demand high-grade stainless steel in the construction of tanks and pipe work.

Whisky authenticity

Whisky authenticity falls into two categories: brand and generic authenticity. Brand authenticity is concerned with whether the liquid in the bottle purchased by the consumer corresponds to the brand name on the label; generic whisky authenticity is concerned with whether the liquid in question is entitled to the broad description 'whisky'. Liquid analysis plays a major role in both cases by allowing the analytical fingerprint of the suspect sample to be compared with that of the genuine brand or whisky type.

Brand authenticity

Whisky brand authenticity analysis enables enforcement agencies to protect their consumers, and producers to protect their brands against illegal substitution and counterfeiting. Substitution occurs when one brand is deliberately and illegally substituted in bars by another (usually cheaper) brand. When this occurs, the consumer, the brand owner and often the bar owner are defrauded. In some countries, such as the UK, consumer protection officers (from local authority Trading Standards or Environmental Health departments) collect suspect samples from bars and submit them to their support laboratory (known as the Public Analyst) or the brand owner for authenticity analysis.

In the case of blended Scotch whisky, it has been found that analysis of the major volatile congeners (particularly the higher alcohols) allows normal congener concentration ranges to be set (Aylott *et al*., 1994). As an example, Table 9.4 shows the normal analytical range data for three different brands of blended Scotch whisky during 2001. The three brands contain quite different blends of malt and grain whisky, as can be seen in their unique amyl alcohol (2- and 3-methylbutanol) concentration ranges. As the less expensive brands tend to contain lower proportions of malt whisky, these are usually the type of product used illegally to refill the more expensive products.

The analyst will therefore determine alcoholic strength and the major volatile congeners in a suspect sample, and compare the results with the normal ranges for the genuine brand. If the suspect sample's results fall within the normal ranges, then that sample's authenticity is usually accepted. If its results are outside the normal ranges, the sample is not authentic. Table 9.5 contains the results for three suspect samples, numbered B1, B2 and B3, which were collected from bars by consumer protection officers and sold in bottles labelled as brand B. The result for sample B1 fell within the normal ranges for brand B and was therefore accepted as genuine brand B. Sample B2 had congener concentrations within the normal ranges for the brand but an alcoholic strength of 30 per cent vol.; it was concluded that this sample was the genuine

Table 9.4

Major volatile congener concentration ranges (g/100l abs. alc.) for three different brands of Scotch whisky comprising samples taken from 42, 54 and 42 production batches, respectively

Sample	Declared alcoholic strength (%)	Acetaldehyde	Methanol	Ethyl acetate	n-Propanol	Isobutanol	2-methylbutanol	3-methylbutanol	2- and 3-methylbutanol	Ratio 1	Ratio 2
Brand A	40	3.4–8.4	6.4–9.9	20–24	50–58	61–70	10–13.7	32–39	42–53	0.7–0.8	2.8–3.1
Brand B	40	3.3–8.6	6.2–10	21–27	51–66	61–72	16–21	46–56	62–76	0.9–1.1	2.7–3.0
Brand C	40	6.6–13	6.0–9.2	34–42	58–87	64–76	22–26	60–70	82–96	1.2–1.3	2.6–2.8

Ratio R1 = 2- and 3-methyl butanol/isobutanol.
Ratio R2 = 3-methylbutanol/2-methylbutanol.

Table 9.5
Major volatile congener concentrations (g/100l abs. alc.) in Scotch whisky brand authenticity investigations

Sample	Alcoholic strength (%)	Acetaldehyde	Methanol	Ethyl acetate	n-Propanol	Isobutanol	2-methyl-butanol	3-methyl-butanol	2- and 3-methyl-butanol	Ratio 1	Ratio 2	Authenticity	Conclusion
Suspect sample B1	40.0	7.2	8.5	25	60	70	17	48	65	0.93	2.82	Genuine	. . .
Suspect sample B2	30.2	5.0	6.0	22	55	70	20	54	68	0.97	2.7	Not genuine	Diluted with water
Suspect sample B3	40.0	9.0	5.0	15	39	75	13	35	48	0.64	2.7	Not genuine	Other Scotch whisky
Suspect sample C1	40.1	11.7	8.8	32	70	73	22	62	84	1.15	2.8	Genuine	. . .
Suspect sample C2	39.3	6.4	6.9	15	43	46	13	38	51	1.10	2.8	Not genuine	Other Scotch whisky
Suspect sample C3	47.2	1.7	1.7	4	7	9	5	15	21	2.18	2.9	Not genuine	Admixture
Suspect sample C4	38.0	8.0	4.0	16	30	35	11	31	42	1.15	2.8	Not genuine	'Stretched'

Ratio R1 = 2- and 3-methyl butanol/isobutanol.
Ratio R2 = 3-methylbutanol/2-methylbutanol.

brand but had been adulterated and stretched by dilution with added water. Sample B3 had many congeners outside the normal ranges for brand B (particularly n-propanol, isoamyl alcohol and ratio R1), and it was concluded that it was not the genuine brand B but another blended Scotch whisky with less malt whisky in its blend composition compared to brand B. This analytical approach regularly contributes to prosecution evidence against those who substitute popular brands in the on-trade with cheaper products.

In a similar way, samples C1–C4 were suspect counterfeit samples collected in Asia and purporting to be deluxe Brand C. The results for suspect sample C1 fell within the normal ranges for Brand C and therefore sample C1 was concluded to be genuine. Sample C2 results were outside the required ranges, particularly isoamyl alcohol and ratio R2, which suggested the liquid was not genuine but was another Scotch whisky with much less malt whisky in its blend composition compared to Brand C. Sample C3 was not genuine; its higher alcohol concentrations were very low, but ratios 1 and 2 were characteristic of Scotch malt whisky. This suggested that Sample C3 was an admixture based on neutral alcohol flavoured with a smaller proportion of malt whisky. Sample C4 was also not genuine, with all congener concentrations at approximately one-half of their expected values for genuine product. This suggested that Sample C4 was based on genuine Brand C ,which had been adulterated (or 'stretched') by the addition of an equal volume of cheaper neutral alcohol.

There are needs for similar analytical strategies to be established for checking the brand authenticities of Irish, Canadian and bourbon whiskies. However, many brands, each with their own unique blend compositions, can show overlapping analytical fingerprints, and the methodology described above then becomes limited. There is a need for more research in this area.

Generic authenticity

Generic authenticity analysis is more complicated than brand authenticity analysis because the analytical data employed must encompass all the whiskies in that category, rather than the more narrow analytical fingerprints of specific brands (Lisle *et al.*, 1978; Simpkins, 1985; Aylott, 1999). Generic authenticity is primarily a concern in the less regulated markets, where there are less stringent legal definitions for whisky and less stringent consumer protection laws.

Chromatographic techniques contribute qualitative and quantitative information for suspect samples to be compared with the known analytical ranges of genuine product. It is also useful to check for the presence of components in the suspect product that are foreign to the generic whisky, or for the absence of required congeners from the suspect product. The presence of foreign components or the absence of key congeners will raise suspicions about generic authenticity.

The major volatile congener profile can be very useful in generic authenticity analyses. For example, an abnormally high methanol concentration may

suggest the use of a non-cereal alcohol. The presence of 3-methylbutanol without any 2-methylbutanol may suggest the addition of 3-methylbutanol as a synthetic flavouring. The maturation congener profile can be used in a similar way. The absence of maturation congeners may suggest that the suspect product has not been subjected to the required period of maturation, and the presence of a congener (such as vanillin) on its own without related maturation congeners may suggest its addition as part of a synthetic flavouring. Analytical conclusions from such examples may be used to demonstrate that a suspect product purporting to be whisky has not been produced in compliance with the regulatory requirements. The analytical evidence can then contribute to the disqualification of suspect products as whisky.

Isotopic techniques have been assessed for use in checking that the alcohol suspect samples are derived only from cereal fermentation and not a cheaper source such as cane or beet. One potential stable isotope measurement is the $^2D/^1H$ ratio in different positions on the methyl and methylene hydrogens in ethanol, determined using site-specific natural isotope fractionation–nuclear magnetic resonance (SNIF–NMR). The methyl hydrogens are primarily influenced by fermented substrate, and the methylene hydrogens are strongly influenced by the ratio in the fermentation water (Martin *et al.*, 1995). $^{13}C/^{12}C$ stable isotope ratios are influenced by the photosynthetic pathway used by the fermentable substrate to assimilate CO_2 (Parker *et al.*, 1998). Barley, wheat and beet use the C_3 (Calvin) pathway, and maize (corn) and cane use the C_4 (Hatch–Slack) pathway. Unfortunately, this technique cannot help authenticate the many whiskies made from both barley and maize. Lastly, synthetic alcohol is obviously not permitted in whisky, and ^{14}C analysis by liquid scintillation counting may be used to discriminate between synthetic and natural ethyl alcohol in spirits and fortified wines (McWeeny and Bates, 1980).

Recent issues

Nitrosamines

N-Nitrosodimethylamine (NDMA) was discovered in the mid-1970s at low µg/l levels in certain beers and whiskies. NDMA is a suspected carcinogen, and therefore methods of analysis were required in order to determine its concentration and to understand and control its formation. NDMA is analysed by gas chromatography with a thermal energy analyser (TEA) as detector. This analysis requires no sample preparation for whisky samples, yet offers sensitivity to below 1 µg/l.

Research at that time on Scotch whisky found that NDMA formation was influenced by the kilning conditions for malted barley. The presence of oxides of nitrogen (NO_x) in the kiln gases enhanced its formation. NDMA could be reduced by preventing NO_x from entering the system, or by ensuring the presence of sulphur dioxide in the gas stream (Duncan, 1992). Sulphur-dioxide

levels were enhanced in those malt kilns fired by heavy fuel oil rather than natural gas. Now that the NDMA issue has passed, its regular analysis in Scotch whisky is all but over, with only occasional analyses required for very aged samples.

Ethyl carbamate

Ethyl carbamate (urethane) was discovered as a trace component at the µg/l level in a wide range of foods and alcoholic beverages in the mid-1980s. As ethyl carbamate (EC) is a carcinogen to laboratory animals at much higher dose concentrations, Health and Welfare Canada set maximum concentrations in table wines (10 µg/l), fortified wines and sake (100 µg/l), distilled spirits (150 µg/l) and fruit spirits, liqueurs and grape brandy (400 µg/l) (Connacher and Page, 1986). There then followed an intense five-year period in the European and North American whisky industries, first to determine natural concentrations of EC in whisky with adequate sensitivity, selectivity and precision, and then to understand its method of formation so that control measures could be implemented (Zimmerli and Schlatter, 1991). Various surveys have shown that most whiskies contain EC concentrations at less than 100 µg/l (Dennis *et al*., 1989; Battaglia *et al*., 1990; Food Standards Agency UK, 2000), although higher concentrations could be found in certain single whiskies. Similarly, certain bourbon whiskies exceeded the Canadian 150-µg/l limit. United States distillers later agreed with BATF a 125-µg/l EC target limit for new whisky distillate. More recently, a 400-µg/l EC limit has been introduced in Germany due to the greater EC levels encountered in certain fruit spirits.

Methods for the determination of EC in whiskies and other distilled spirits require capillary column gas chromatography coupled to nitrogen-specific or mass spectrometric detection (Aylott *et al*., 1987). Initially EC was concentrated by solvent extraction, but subsequently sample preparation was minimized and the method automated by employing direct injection of whisky samples with added n-propyl carbamate as an internal standard. GC–MS on a polar capillary column with mass detection at m/z 62 enabled good selectivity and a limit of detection of less than 5 µg/l in whiskies. It was found that the chromatographic peak shape for EC was better when analysing distillation and maturation strength samples (at > 55 per cent vol.) compared to those at 40 per cent vol. bottle strength. Therefore, bottle strength samples were first diluted with absolute alcohol (containing no EC) in order to bring sample alcoholic strength up to about 70 per cent vol. prior to injection.

Intense research was initiated throughout the alcoholic beverage industry in order to understand EC formation. The breakthrough for whisky came when post-distillation EC formation from trace cyanide and cyanate precursors was discovered in the Scotch grain whisky process (Aylott *et al*., 1990). Trace cyanide was found to come from the thermal decomposition during distillation of the cyanohydrin of isobutyraldehyde (IBAC), which is present in fermented wash (or beer in the USA). IBAC arises during fermentation by the hydrolytic action of yeast β-glucosidase on a naturally occurring cyanogenic glycoside

known as epiheterodendrin (EPH). EPH is located in the acrospires (growing shoots) of malted barley (Cook, 1990, Cook et al., 1990). Laboratory-based radiochemical studies verified this chemical pathway (McGill and Morley, 1990).

The knowledge that the relatively low EC levels in new-make grain spirit from a Coffey still could increase by post-distillation EC formation from cyanide and cyanate precursors led to investigations into their formation, using ion chromatography with conductivity and pulsed amperometric detectors (MacKenzie et al., 1990). The precursors cyanide, copper cyanide complex anions, lactonitrile and IBAC were collectively determined as 'measurable cyanide' (MC), along with cyanate and thiocyanate ions. A practical relationship was established between MC concentrations in new-make spirit and final EC concentrations after all the available MC precursor had converted during the first few weeks of maturation into EC (Aylott et al., 1990), giving the formula:

$$\text{Final EC} = 0.5\,\text{MC} + 15$$

Ion chromatographic or colorimetric MC analyses were introduced into distillery process control. During this period of research the presence of sacrificial copper surfaces in Scotch grain whisky Coffey stills and the addition of sacrificial copper rings into stainless steel American bourbon beer stills was found to mop up MC, and thus help reduce post-distillation EC formation.

More importantly, it was found that different barley varieties have differing levels of EC precursors (Cook, 1990, Cook et al., 1990). First this knowledge enabled maltsters and distillers to select low MC-potential varieties, and then enabled growers to develop new varieties of malting barley with low levels of epiheterodendrin precursor. Ethyl carbamate in all whiskies can now be monitored, and its formation minimized through distillation process control and the use of malting barley varieties with low MC-potential.

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Marketing Scotch whisky

Grant E. Gordon

Introduction

Before exploring the theme of the marketing of Scotch whisky it would be helpful to define what marketing is about and why it is an important discipline for the successful conduct of any business today.

Philip Kotler, of the Kellogg School of Management at Northwestern University, and one of the leading academics in the field, defines marketing as 'the set of human activities directed at facilitating and consummating exchanges' (Kotler, 1999). This definition is concerned with decisions by people, either individually or in groups, conducting voluntary activities. Marketing covers exchanges that are either one-time transactions or those that concern building lasting relationships between buyers and sellers. The marketing process is a two-sided discipline, where both buyers and sellers can be actively involved. In terms of what is being exchanged, the key point is that it concerns something that has value to one or both parties.

A narrower definition of marketing that is relevant to the practice of the discipline is 'the management of all the functions required to [copyright] satisfy consumers and maximize profit in the long run for the company'. This definition puts the spotlight on the consumer, being the person who counts most in transactions and is the focus of attention. It also highlights the motivation of companies, which is the pursuit of profits over the long run, which in turn depend upon the revenues derived from recurring customer and consumer demand. Returning again to Kotler, marketing management is 'the analysis, planning, implementation, and control of programmes designed to bring about desired exchanges with target audiences for the purpose of personal or mutual gain'. Reliance is placed upon coordinating and adapting the key variables for achieving an effective response from the consumer. McCarthy popularized a four-factor classification of the marketing decision variables, the 'four Ps':
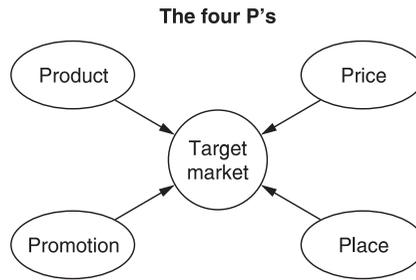
The four P's

Figure 10.1
The four Ps. (Acknowledgments: E. Jerome McCarthy and Philip Kotler.)

Product, Promotion, and Place, Price (Figure 10.1). This chapter explores these four variables and shows how firms practising marketing use these tools to influence sales and pursue long-term profitability. It will also become clear that each of the 'Ps' is in reality a collection of instruments.

Emphasis in these definitions is put on the management process to achieve a desired end result. Without a proactive effort in terms of time, effort, skill and dedication the marketing process will just not happen, and achieving any set of objectives will be hit and miss. Marketing demands engagement, and is best carried out with passion, imagination and flair tempered with discipline. Marketers need a sense of what levers to pull in order to capture the custom of the consumers they are targeting. Successful entrepreneurs usually have an ingrained understanding and appreciation of marketing, but even if marketing is not a science it can be taught and most certainly learnt through practice. Those who practise marketing should not be afraid of error, because very often the best lessons are gleaned from failed experiments.

Marketing books have become popular, and can be found in many bookstores. They sport catchy headlines such as *The Customer is King*, *How Brand X Conquered the Market for Widgets*, *Understanding the Psyche of the Consumer*, etc. Such books, along with many others, have a whole raft of topical messages to deliver. The subjects covered are varied, and offer good guides for those who want to grasp the essentials of best practice. They often include useful lessons in marketing from leading practitioners in the field. However, for all marketers there is nothing that can replace learning through the experience gained from real business situations.

The origins of demand

Historical development

It is quite likely that distilled spirits in Scotland were an import from Ireland during the Middle Ages, brought over by missionary monks. The Scottish Exchequer Rolls for 1494 hold the first written reference to the product, noting

that Friar Cor had used 'eight bolls of malt wherewith to make *aqua vitae*'. The product that became known as whisky, from the Gaelic *uisge* baugh ('water of life'), has helped to make Scotland famous around the world. In the early days the distilling of Scotch whisky was a cottage industry and a natural complement to the traditional farming that supported life in the Highlands. The government soon woke up to the opportunity of using the popularity of the product as a means to raise taxes, and the Excise Act of 1664 fixed duty at 2s and 8d per pint of *aqua vitae*. As a complement to this decision by the Scottish Parliament, the Excise Officer made his appearance. From the eighteenth century onwards there was an emergence of demand on a significant scale for the water of life, but it took a very long time for the industry to settle down and achieve the transition from the freewheeling days of illicit distilling to the modern, regulated industry, with its strict licensing of producers.

One of the key forerunners in the development of the modern-day industry was The Distillers Company, which that was formed on 1 May 1877 as an amalgamation of six Lowland distilleries, all of which produced grain whisky. This was a key milestone in the development of the industry, as it moved it beyond the craft industry stage into a business run much more along commercial lines. Indeed, the advent of grain whisky distilling provided the technology that allowed producers to achieve two objectives: first, they managed to produce a whisky very economically, and secondly, producers now had a quality of spirit offering a lighter and usually more consistent taste than traditional malt whiskies. A new profession emerged, known as blending, along with a new type of whisky that could be mixed to good effect with the traditional malt distillates. Leading blenders would soon launch their own brands, offering consistency of product with a distinct cost advantage. Gradually the blending houses were able to build consumer franchises for their brands. The names that emerged at that time, such as Johnnie Walker, are today amongst the leading brands in the industry.

Demand in the United Kingdom for Scotch whisky gradually grew throughout the nineteenth century. The companies that were best able to meet this demand were the houses that controlled the supply of the grain whisky. This had become the main ingredient in the end product that consumers now demanded, with malt whisky already playing a subsidiary role. Brands that were unscrupulous with their consumers would include as much as 90 per cent grain whisky, offering doubtful quality. Selling a matured product was yet to become the norm, with consumers still accustomed to drinking whiskies that were bottled straight after distillation – just as vodka is today. In these early days of the modern industry it can be supposed that product quality was varied, but demand grew nonetheless.

With a growing industry, more and more players were being attracted into the market to meet the rising demand. For historians it is probably not a great surprise that before the end of the Victorian era the industry had entered a real crisis. Indeed, the growth in popularity with consumers had led to a boom in the production of all types of whisky, which caused a glut in the market at approach of the turn of the century. In 1898, with the bankruptcy of Pattisons, there was a collapse in confidence in the market. However, from the ashes of this crisis there

emerged a number of players who managed to survive. These companies, of which five are the most famous – Haig, Dewar, Buchanan, Walker and Mackie – were leaders in the development of the industry at the dawn of the twentieth century. Scotch whisky was about to spread its wings and build demand, starting mainly in the English-speaking nations of the world.

In the run up to the First World War, pressure was increasing to introduce measures to define quality standards in the industry. A benefit of establishing tighter rules of the game would eventually be to improve the product's reputation with consumers, who had grown accustomed to finding products of varying quality. The Immature Spirits (Restrictions) Act was passed on 19 May 1915, making it compulsory to keep spirit in bond for two years, with the period extended to three years in May 1916. This new legislation was one of the key building blocks that would pave the way to better quality products and, in turn, greater demand. This new legislation led to the demise of the cheaper, immature whiskies that at that stage still accounted for the bulk of consumer demand. After the First World War demand was reduced, as the more expensive whiskies now being marketed were beyond the reach of the mass market.

There was another major barrier to the development of demand during the first part of the twentieth century. In the home market, as in other countries, a movement emerged against the consumption of alcohol as a significant number of people blamed alcohol for creating social disorder in certain sections of the community. This was aside from any impact excessive drinking was having on health standards overall. The temperance movement, as it was called, began to take hold on both sides of the Atlantic, resulting in a period where the consumption of all forms of alcohol was banned in the USA. This 'Prohibition' era did not last very long, as the benefits that its supporters were looking for did not materialize and popular opinion moved firmly in favour of restoring consumer freedom of choice. However, before the period of restrictions had drawn to a close the trend against alcohol had spread to other corners of the globe, and continued to cast a cloud over the industry's efforts to market its products right up to the advent of the Second World War.

As markets reopened in the 1930s, the industry's fortunes were able slowly to start to recover. This recovery was driven largely by exports. In 1937, for the first time in the industry's history, the export market was larger than the home trade. By this stage there was also a clear pattern emerging in terms of leading brands that commanded the loyalty of consumers at home and abroad. Names such as Haig, Dewar's and Johnnie Walker were now well established as the most popular choices, and were leaders in the market. Apart from a strong consumer following, these brands shared another thing in common; they were all owned by the same organization – the Distillers Company Limited (DCL). The DCL was all-powerful in the industry, and enjoyed a quasi-monopoly status. Indeed the company, through a series of amalgamations, had collected a portfolio of leading brands. With such a portfolio of products the DCL had a tight grip on the market, and was able to set prices and thereby define the returns available to all players. The DCL set the agenda in terms of the way the product was marketed, and most of the credit for establishing the foundations

of Scotch whisky marketing goes to the efforts of the people around the world who worked in the company, or as its agents and distributors, building its brands. Other companies were generally followers, and their chance of making a mark would have to wait until after the war years.

Winning and losing consumers

The Second World War also had a particular impact on the fortunes of the industry. American GIs streamed through the continent of Europe in the defence of the Allies, and these soldiers brought with them their consumer habits – one of which was the fashion for drinking whisky. Films of the time showed the heroes relaxing with a glass of whisky, and helped to spread the image of a fashionable alcoholic beverage. An immediate consequence of the war, however, was to curtail production of whisky in Scotland, and this reduction in supply was to have beneficial consequences in the years after the war. Indeed as the world emerged from the horror of the fighting to turn to the task of rebuilding peace and prosperity, the demand for Scotch was about to grow against a backdrop of tight availability. A real product shortage was created, and this kept prices firm and helped underwrite the sense of exclusivity of the beverage. Scotch was to become in peacetime the aspirational alcoholic drink sought after by many.

During the post-war era the industry started to see the emergence of the USA as the main market for Scotch whisky as it took on fashion-drink status throughout the USA. With a flourishing market across the Atlantic the industry entered its real take-off phase – a phase that was to propel it to the ranks of the leading international spirit of choice for drinkers around the world. Demand for Scotch whisky grew annually at close to double-digit percentages in the three decades after the end of the war up to 1970. These were the glory days for an industry that grew from a volume of 10 million cases sold in 1950 to over 51 million cases in 1970, driven by strong export growth. However, the growth levels the industry had grown accustomed to could not continue indefinitely, and more challenging times lay ahead. Indeed history was about to repeat itself, and the industry was on the verge of creating the largest hangover it could possibly imagine. Faced with a plateauing world demand, producers continued to maintain output as if growth was still in the offing and, by failing to curtail production, huge excesses of stock were accumulated. The problem gradually built up during the 1970s, and by the mid-1980s the dam finally burst when prices in the industry collapsed severely as a result of the high volumes of cheap whisky that were flooding the market. Damage to the reputation and image of Scotch whisky was a consequence of the glut that allowed low-priced generic brands to take hold in the market.

In fact, the failure to keep demand and supply in equilibrium was to have a long-lasting impact on the industry as Scotch whisky moved down the path of other consumer products and became commoditized. The worst of the effects were felt in mature markets, where the low-priced products were able to make large inroads in market share terms at the expense of the leading brands. As

cheaper substitutes became available brands started to lose their pre-eminence in consumers' minds, and they gradually placed less value on higher-priced products. In the USA the demand boom of the after-war era had taken the industry to new heights, but the consumer following lost momentum as fashion turned the consumer towards other substitute drinks. The cycle had turned, and marketers were unable to keep the product's leading image at the top of the consumers' set of preferred choices while other products were able to make inroads and grow in popularity. However, efforts to build demand in other parts of the world started to pay dividends, and the industry's dependence upon the USA declined along with its sales in its largest market.

The latter part of the twentieth century saw a new phase of the industry's development where a series of markets started to emerge closer to home, and especially in Europe. Here drinkers were attracted by the image and taste of the product that had previously been the trend in the USA. In Asia the popularity of Scotch whisky also grew as drinkers found, in the product's quality and image, values that are hallmarks of many luxury western-branded products. For many of the newly acquired customers in these countries Scotch whisky was a natural beverage accompaniment to social occasions with friends.

The other new element of change that came over the horizon (or more precisely re-emerged from the long past history of the industry) was the emergence of single malt whiskies. To those who could afford to experience the best, the availability of relatively unknown and sought-after brands of single malt heralded the start of a new trend. The result has been the emergence of a new premium category within the industry, identified by products that have their origins in the Highlands and Islands of Scotland. The folklore and mystery surrounding these brands and their deep historic roots give these products a strong traditional background. This imagery, allied to the variety of tastes, has won the loyalty of many new Scotch whisky lovers around the globe. By taking the consumer into new territories in terms of taste and general satisfaction, marketers have managed to create renewed appeal for the category overall while at the same time moving the image onto a higher platform in terms of perceived value.

The industry that has emerged from all these changes and cycles of demand is thriving, and has firmly established a place at the top table in terms of beverages of choice for drinkers around the world. At the beginning of the new millennium, Scotch whisky is a leading category of choice in consumers of spirits. Scotch whisky sales totalled 74 million cases in 2000 compared with 63 million cases in 1985, showing the resilience of demand for the category (see Figure 10.2). One feature of the pattern of sales during recent history is the rising importance of premium and malt whiskies as sub-categories together representing 18 per cent of total volume demand in 2000. There has been a steady switch of consumption towards the higher value added brands, and premium whiskies, dominated by twelve-year-old Scotches, have led this trend. Single malts have also played an important part in this trend of trading up by consumers, almost doubling its share to 5 per cent over the same period. The rise in popularity of these two premium categories has been at the expense of standard brands, with low-priced products maintaining share overall.

Low price   Malt
Standard   Premium
Standard plus/medium   Super premium
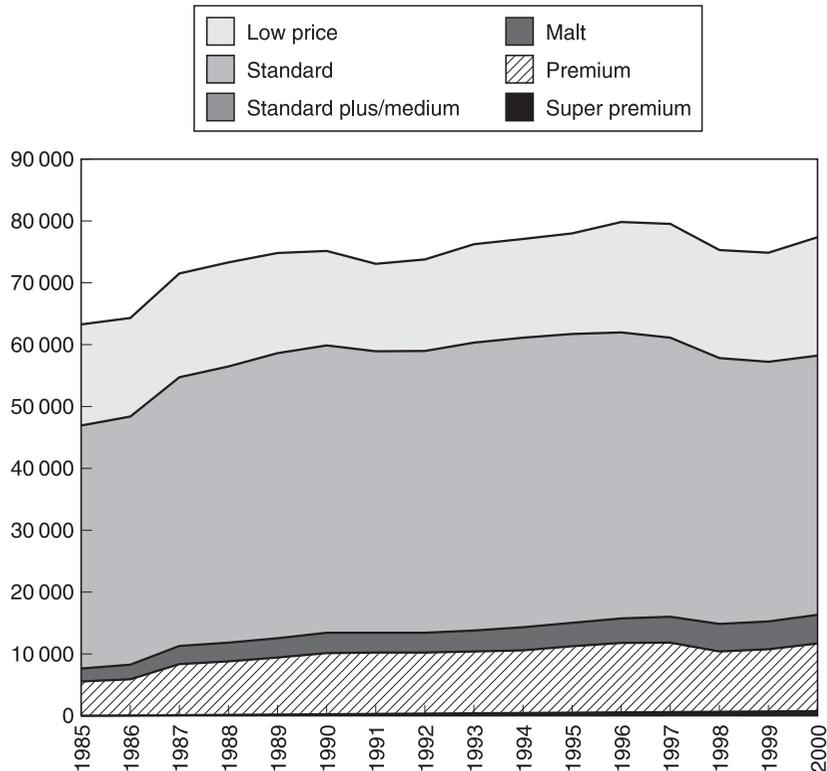
Figure 10.2
Worldwide whisky sales (in '000s 9-l cases) – domestic and duty free

The leading brands that today dominate the category have developed strong loyalty from their consumers; the result of years of building their reputations. Alongside the major brands there are many niche products that cater for the demands of the connoisseurs. Today consumers enjoy a diverse choice of brands catering for all tastes and budgets. However, making choices may not be easy for consumers who cannot always differentiate between brands. The power of persuasion through promotion can therefore exert a strong influence on choices made, and a complex and sophisticated consumer marketing industry has built up. The modern trade that is the Scotch whisky industry is forever tuning into the consumer and trying to satisfy their every demand profitably.

Current industry dynamics

This section discusses some of the tools that are used to build strong consumer franchises, explores the main building blocks in making a marketing plan, and describes the various approaches of companies in carving a niche for their

particular brands. Marketing theory continues to evolve, but at the heart of any campaign to conquer the loyalty of consumers there lie some basic elements that will never change; an attempt is made here to pinpoint some of these elements and focus on successful strategies to increase consumer demand.

It is vital when looking at any market to be able to view the marketplace through the eyes of the consumer. Consumers considering their choices in alcoholic beverages must first narrow down the selection to three broad categories: beer, wines and spirits. In more recent times the choice has blurred, as there are products that successfully transcend these traditional product categories. Some brands have re-segmented the market and proposed new expressions of their products that are trying to cater for new modes of consumption – for example, there is now a new class of ready-to-drink (RTD) or new-age beverages that can be beer-, wine- or spirit-based, and sometimes are mixes of all three basic forms of alcoholic drink. RTD products have been fuelling a large part of the growth in consumption in alcoholic drinks in the last few years, especially in the western world. Their popularity with younger drinkers is high – the consumer can chose a trusted brand in a format that fits well with popular drinking modes. The success stories here have managed to offer an alternative to the popular traditional offerings, such as draft beer or chilled wine. The consumer is usually comforted by a well-known brand that brings a guarantee of quality, with an image to boot.

The consumer framework for choosing a drink for a particular occasion usually starts with the category of choice, and then narrows down to a brand. In terms of category choice, the decision will be partly related to the setting and type of occasion. On the other hand, for some consumers category choice may be of secondary importance – particularly where there is a social drinking occasion, when choosing the right 'badge' or brand may be more important. When it comes to brand selection there is no end of choice available to consumers today. Indeed the choice is bewilderingly large, and consumers can select from scores of different products. A visit to any bar, restaurant or retail outlet will confirm the plethora of brands available. Probably the most promiscuous**55 of the three categories of alcoholic beverage is wine. Here the choice is highly fragmented, with wineries remaining generally small in size, notwithstanding some recent moves by major multinational drinks corporations to consolidate the industry and build a portfolio of wine brands. Compared to many other fast-moving consumer good categories, the drinks industry arguably offers more consumer choice than most – as can be seen from a visit to any major supermarket, whose shelves are abundant with alcoholic drinks of all tastes and variety.

A framework for consumer choice when selecting drinks is set out in Figure 10.3.**56 The three driving factors are need, occasion and personal state. 'Need' relates to the person's mood, and 'occasion' to when, where and with whom the consumer is going to enjoy a drink. 'Personal state', on the other hand, answers the question: 'Who am I at that moment?'. The way in which these three factors intercept help determine what choice of product and brand the consumer makes.

```
                    ┌─────────────────────┐
                    │         Why         │
                    │                     │
                    │ • What mood I'm in   │
                    │ • What needs I have  │
                    │ • What benefits I seek│
                    └─────────────────────┘
                               │
   ┌──────────────────────────┐│          ┌──────────────────────────┐
   │      When/where          ││          │          Who             │
   │                          ││          │                          │
   │ • Type of occasion/situation│          │ • Show who I really am     │
   │   (e.g. pleasure, business)│         │ • Show who I'd like to be  │
   │ • Where I am             ││          │   (e.g. vs peers)         │
   │ • With whom              ││          │ • Show who I ought to be   │
   │ • Day of the week        ││          │   (e.g. business)         │
   │ • Time of day            ││          │                          │
   └──────────────────────────┘│          └──────────────────────────┘
              │                 │                      │
              │      ┌──────────▼──────────┐           │
              └─────▶│        What         │◀──────────┘
                     │                     │
                     │ • Brand I buy/drink │
                     └─ ─ ─ ─ ─ ─ ─ ─ ─ ─ ┘
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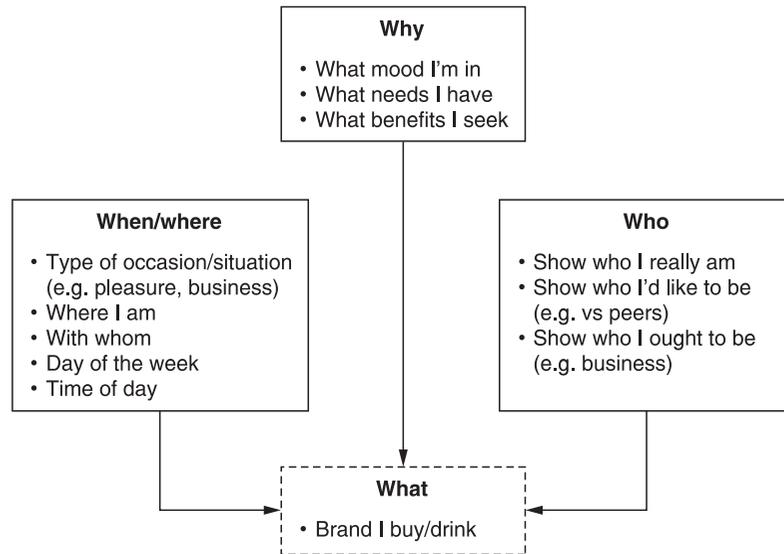
Figure 10.3

The consumer selecting a brand of spirits and inclined to make a popular choice will naturally often chose one of the most popular brands. At present the five most demanded international brands are Bacardi, Smirnoff, Johnnie Walker, J&B and Absolut. These five names cover three distinct categories: vodka, rum and whisky. When confronted with such an array of products, the existence of well-known brands acts as a powerful means of assisting consumers in making choices. The brand is a point of reference for consumers, allowing them easily to identify their brand of choice by its values in terms of taste, image and other attributes. Of course choices can be made without reference to a particular brand – for example by choosing simply a vodka or a whisky. However, today the vast majority of consumers rely on brands when making choices, as this gives them reassurance as to the provenance and quality, and ultimately a guarantee of satisfaction.

With the vast array of spirits that are on offer to consumers around the world, Scotch whisky has to compete with some other very popular alternatives. The choices have tended to grow over the years as consumers have developed a taste for many alternative spirits, ranging in the past from gin to more contemporary drinks such as vodka and tequila. However, this is a dynamic process, and new categories emerge as others lose appeal. For example, few people today know that the bitters that are popular in some European markets used to be a desired tipple in Great Britain. Whisky has developed almost universal appeal, and holds a strong position as the category of choice of many of the world's drinkers. The whisky category also includes some brands that are of international repute and have the strength to compete effectively with strong competitors from other categories.

In terms of category share, Scotch whisky at present enjoys approximately 9 per cent of the global spirits market. The total share of whiskies goes up a fair amount when all other types of whisky from around the world are added, including bourbon, Canadian, Japanese and other locally produced forms of whisky. Indeed, contrary to common perception, 'brown spirits' are today overall a very large and vibrant part of the total world spirits market. In addition to whisky, this category includes such drinks as cognac, other brandies, and dark rums. To put matters in context, the leading category continues to be vodka, with a commanding position of approximately one bottle out of every four bottles of spirit consumed globally, confirming the huge popularity of white spirits with consumers.

Economics of the whisky industry – the value chain

Cost of goods and the impact of maturing spirit charges

A detailed understanding of the industry and its operations is helped by a full understanding of the way in which costs are built up in the supply chain, starting from production and continuing all the way through to marketing. The most important element that differentiates Scotch whisky in terms of cost from other beverages is the consideration of time as a factor. Whereas a normal industrial product will be ready for sale the day it leaves the factory door, in the case of whisky, law stipulates that the product must be matured for no less than three years. There is a delay between the moment of production and the time of sale, and during this period the funds that have been set aside for the production process are effectively dormant, waiting for the product to become ready for market. This in itself is not a problem in theory, providing that there are sufficient funds available to finance the business pending sale of the stock that has been produced. In practice most players in the industry have a strong and steady cash flow from their activities, and therefore the financial strength and stability to afford the burden of holding some of their products for very long periods of maturation before bottling and shipping to market.

This position can only be reached once a company is producing healthy profits. Profit margins simply have to be calculated to take the time/cost factor into consideration. However, achieving profitability is much more complex, and results from the marketing action that producers take. How they manage to achieve this is discussed later in this chapter.

Looking at the cost of goods in more detail, the following aspects need to be considered:

- Spirit: the cost of cereals, fuel and energy, direct labour and production overheads
- Financing: the cost of funds required to finance the inventory from the time of production until a sale takes place

- Wood: the depreciation of the cask (over its useful life) and the cost of repairs to the stock of wood
- Warehousing: the cost of storing casks during the maturation period
- Transport: the cost of moving whisky from the point of distillation to the point of bottling
- Bottling: the cost of packaging a case of whisky, including labour, bottling overheads and depreciation
- Packaging materials: the variable cost of materials, including an allowance for wastage.

Bringing all these costs together gives a total product cost, and provides a key input variable in the value-chain equation. The gross margin on the product can be calculated by deducting the net revenue derived from the product cost. As in all consumer product industries, emphasis must be placed on minimizing the cost of goods in order to help maximize gross margin. The cost of goods is generally being reduced through production efficiencies while maintaining quality standards, and this process helps allow room for an adequate marketing effort to take place and for a profit to be earned in the final count. The nature of competitive markets is such that marketing costs have if anything grown over the years, as producers compete harder for the consumer's loyalty. Therefore, driving gross margin generation helps to free-up cash for marketing expenditures that will have to be committed in order for the brands to flourish.

Marketing costs

The main areas to be considered under the broad heading of marketing costs can be broken down into two key components. First there are the brand development costs, which include product marketing (advertising, promotion, public relations and any other paid-for activities designed to influence demand in whatever form). Secondly, there are costs related to channel management, or selling and distribution. This category refers to the entire infrastructure that is required to drive product through the distribution chain to reach consumers. Another way to view cost is that there are the costs of getting the product to market (channel costs) and, once it is there, of pulling it through to the consumer (advertising and promotion; A&P). The sum of these two major categories of cost is often the largest single cost line on the P&L.**57 The careful management of these resources is arguably the most difficult area of the management decision-making process in a consumer branded goods company.

The direct output can be measured as profit after marketing (PAM), representing the net revenues minus the cost of goods and the selling/A&P expenses. PAM measures the economic contribution of the product to the company's finances after allowing for all costs related to its production and marketing. Adding value at this level is a key variable in terms of ultimately driving corporate profitability. Success in building long-term value, measured

in PAM terms, will drive shareholder value over the long run, and is the goal of modern consumer marketing companies.

Marketing costs vary significantly by brand, as there is generally a distinction between brands that are being built, maintained or milked. In the build phase the brand may have a level of spend that is high in relation to revenues, incurring losses. Maintenance applies to brands that are in strong existing market share positions that must be defended against competitive attack. The brand should be making a 'normal' profit at this stage in its lifecycle. Finally there are brands that are being milked, where the expenditure in marketing is kept low and profitability is maximized in the short term. This strategy is applied to brands that are deemed to have weak prospects. Common to many consumer goods industries, Scotch whisky producers have somewhat narrowed their portfolios and allowed some brands that were historically of importance gradually to die away, focusing their attention on strengthening their leading brands.

Branding

> Companies *sell* products; companies *market* brands.
>
> (Bobby Calder and Steven Reagan)**58

Making products meaningful

Branding is at the centre of building any business towards sustainable profitability over time. The power of a brand with the consumer will ultimately translate into profits for the producer if this process is managed skilfully. The alcohol beverage industry is no exception, and includes some of the most successful examples of profitable global branded consumer goods companies. Large global players enjoy advantages in the development of portfolios of brands covering key segments of the market, even if the recent trend is for concentration by the key players on a selected number of reputable brands. Valuable marketing resources are channelled into these brands, which in turn are able to reinforce their leadership, gaining consumer trust and loyalty.

The role that brands play in the alcohol beverage market mirrors many other categories. By creating unique imagery and perceived benefits around a product, marketers are able to attract consumers again and again to their leading brands. The brand itself often becomes the main basis today for consumers choosing one product over another. This is the process by which sustained economic value is created, and the product becomes the profit driver for the company. Without strong brand credentials, the status of the product risks being ill defined and consumers will struggle to fit the product into their pattern of consumption. On the other hand, a well-defined positioning for the brand that is well articulated to the consumer has the chance of yielding the best overall result. Scotch whisky marketers have to address these very

issues, and the strategies they implement determine the long-term success of both the category and their respective brands.

In simple terms, branding is the discipline whereby marketers tell people what it is about their products that make them meaningful. Charles Revlon, of the eponymous cosmetics company, used to say 'I sell hope'. Giving meaning to the product will let the consumer know how it is to be understood and what makes it unique as a brand. The brand becomes a symbol or mark that is associated with a product, and to which buyers attach psychological meaning. Brands can be a form of currency for consumers that can enhance their experience through offering increased certainty and performance. The question is, what are the means by which companies can systematically approach this issue in order to give their products unique personalities that will make them into brands? For the brand to have value for consumers, the associations it offers should ideally become a part of their lives. The brand's equity is created through product design, advertising, distribution, and all the other ways that the company contacts the consumer. The total result of all this effort in some way must in the end reside in the buyer's mind. Unless this is the case, a brand is merely a product with a meaningless name attached to it. Stephen King, of the WPP group in London, said: 'A product can be copied by a competitor whereas a brand is unique. A product can be quickly outdated whereas a successful brand is timeless'.

Successful positioning involves first affiliating a brand with some category that consumers can readily grasp, and then differentiating the brand from other products in that same category. This is referred to as competition-based positioning. For sustained success, it is also helpful to link a brand to the consumer's needs and objectives. This is called goal-based positioning. Brand positioning is an essential discipline to any marketer because it forms a basis for the setting of product attributes and pricing strategy, and selecting the promotional and channel mix to fit the demands of a particular targeted segment of users best. In other words, once the positioning is clear the producer can structure the marketing mix in the context of one overall strategic plan for the brand.

In trying to understand positioning further, it is useful to analyse how people represent information in their memory. This can provide a starting point for developing a competition-based positioning strategy. One way information about brands is stored in memory is in terms of natural categories. Taking beer as an example, Heineken is represented in memory as part of the subcategory of lager beer. In turn, lager is part of the category beer, which is part of the category of alcoholic beverages. A level could be added to the hierarchy under Heineken that subdivides the brand form into draft or bottled. For most analytical purposes in consumer behaviour, only two elements of the hierarchy – the brand and the category in which it has membership, or its frame of reference – will suffice.

At each level in the hierarchy objects can have three types of association: attributes, people and occasions. Attributes are physical characteristics of a product, such as its colour, size and taste. People and occasions are together regarded as the image they associate with the brand. Most positions involve

some combination of attributes and image. For alcoholic beverages in general, image is normally the dominating attribute, even though many people will try and rationalize their brand choice in terms of the product's physical attributes. It is not surprising, however, to note that consumers do not make their choices based upon attributes and image alone. Rather, they use attributes and image to infer some benefit. The benefit is usually an abstract concept based on appeal to the consumer's emotions, and it answers a need.

The preferred approach to positioning is to inform consumers of the brand's category membership before starting to put forward points of difference in relation to other category members. The rationale is that first consumers need to know what type of needs the brand can fulfil. To this end, the marketer generally first informs consumers of category membership, and then follows by developing awareness of the points of difference. Campaigns that try to do both these tasks at once often fail because the message insufficiently develops either claim. Naturally, the more resources available to the marketer, the easier it is for the brand to push these messages home.

While it is important to establish the brand's point of difference, this is not usually sufficient for effective brand positioning. At best, this may simply help to grow the category. If many firms engage in category building, the effect may be consumer confusion. In alcoholic beverages this is arguably the case in the table wines category, and may also have occurred in single malts, where in both cases consumers are confronted with a plethora of products making similar claims and thus it is 'harder' to choose between brands. Developing compelling points of difference is normally critical to effective brand positioning, and the message should generally be consistent over time. A sound positioning strategy therefore requires specification of the category in which the brand holds membership, allied to a clear reason as to why the brand stands out from others in the category.

Points of difference

Establishing points of difference, even if only perceived, requires an understanding of the consumer's beliefs. Which consumer beliefs about the category can be used to promote a benefit? The strongest positions are usually ones where a brand enjoys a clear point of difference on a benefit that is a key driver of category use. Category leaders supported by large budgets with which to outspend the competition may simply claim the benefit that motivates category consumption for themselves, using this as their unique selling point (USP). This is referred to as trying to own the category generic benefit.

Smaller brands, on the other hand, typically attempt to establish a niche benefit as their point of difference. Niches are achieved by using the primary category benefits to establish category membership, and then by selecting some benefit other than the focal one for the category to establish brand dominance. In normal practice, limiting the number of benefits that are made focal pays off by creating clarity. In fact, processing brand benefits requires substantial attention from consumers, and they are likely to become

confused or switch off if too much information is put forward. Also, one benefit can easily undermine another – for example, if a brand claims to offer high quality and at the same time be cheap, the consumer may question the real benefit of consuming this brand, as low price and quality don't always go together.

Brand positioning

With competition-based positioning, membership of the category is developed by highlighting the points of similarity with the category, or relating the brand to a category exemplar. Once the brand has acquired category membership status, its advantage is presented over other category members by representing the point of difference. The consumer rationale to trust the brand is thereby built step by step. So, for example, Laphroaig whisky sets out clearly that it belongs to the single malt category, and differentiates itself from other brands by emphasizing unique product attributes in terms of its special taste and flavour.

Once the consumer has established a basic understanding of the brand's category attachment and any point(s) of difference, there exists an opportunity for growth by deepening the meanings associated with the brand's position. This entails demonstrating how the brand relates to the consumer's goals, and having insight into what motivates the consumer to use a brand. The process employed will start with defining key attributes and image, moving through functional benefits and eventually touching on emotional and assumed benefits. By the end of the process a point can be reached where the benefit defines the essence of the brand. Brand essence thus becomes shorthand for what is at the heart of what makes a particular brand unique in the eyes of the consumer.

The same process that can be undertaken at brand level can be undertaken at category level. The assumption with category essence is that if consumers perceive a brand to be positioned in a manner that is sensitive to their problems, the brand is viewed as a solution to these problems. Appropriating category essence can be a useful way to compete for a brand that does not have a meaningful point of difference.

The challenge that marketers face is to find a viable basis for differentiating their brands. One frequent trap that brands fall into is where they are marketed based on a point of difference with their competition, but one that has no relevance to consumers' reasons for choosing the brand. Claiming multiple benefits is the other frequent error; trying to imply that there is something in the brand for everyone. This approach very often ends up confusing the consumer, leaving it unclear what the brand is really about. Once a position is developed, most of the activity behind the brand should be focused on and aimed at sustaining the point of difference. Sustaining benefit over time provides a barrier to entry by competitors.

After the key choices have been made regarding the targeting and positioning of a product, it is useful to summarize these decisions in a positioning statement. This statement is a summary of the key aspects of the marketing

strategy, and will serve as the foundation for decisions about all elements of the marketing mix. The positioning statement should attempt to answer the following questions:

1. Who should be targeted to use the brand?
2. What is the key reason for buying? (the proposition)
3. What are the emotional and functional benefits?
4. What are the reasons to believe the proposition? (substantiators).

A good positioning statement will clearly set the brand apart from its competitors. It should also give a clear understanding of when and why the consumer should use the brand, and what would motivate the purchase. Succinct reasons should be available, justifying why the brand is a compelling proposition. Avoiding vagueness is important, as well as making sure there is a clear linkage between the brand's point of difference and the target consumer's needs and requirements. The distillation of all of above into the brand-positioning statement should produce a clear and unequivocal view of 'brand essence'. This statement is a form of genetic code defining the personality and values of the brand.

Creating brands

To be successful, all brands must have product, price and channel strategies to support the image communicated through advertising or other promotional means. Advertising, while a central part of the marketing mix for any brand, is by no means the only vehicle that should be considered for communicating with consumers. Whatever the means chosen, the brand-building process should be guided in all its detail by the desired positioning of the brand. This is particularly true with brands of international repute sold across many different markets. The overall positioning of the brand must be rigorously upheld and implemented at local level in a manner consistent with the agreed positioning. Implementation is by way of all the decisions related to the marketing mix. Depending on the type of brand being developed, the elements in the marketing mix will be varied. Further discussion on each of the variables in the marketing mix follows.

   When it comes to alcoholic beverages, brands tend to be valued primarily based on imagery. Scotch whiskies are no exception, and the vast majority of the best-known brands are reliant on their imagery for their position in the market and their consumer following. Image brands distinguish themselves by offering unique sets of associations, and these associations are usually based on an emotional (non-natural) appeal to the consumer. For example, Johnnie Walker wants to associate its brand with 'celebrating male success', and its current 'Keep Walking' campaign features male celebrities who have succeeded in their own terms and often against odds. The principle behind an image brand is that consumers are more likely to engage with the brand if it can tap into these types of powerful associations, rather than if the product is

portrayed as tasting better or is smoother. This can be a powerful route to success, but can require large financial resources to develop and sustain.

Managing image brands

Image brands will succeed if and when they make an emotional connection with the consumer. Advertising and the other forms of promotion play a prominent role in developing the image of any brand. The process of building the brand takes a lot of time and effort and considerable resources, because the image has to be created in the mind of the consumer. Caution should also be exercised, and the limitations of the brand-building process understood – for example, products that rely heavily on user imagery may have limited appeal across generations. Young people in many countries may see Scotch whisky as the drink of the older generation. Where this situation is faced it provides a challenge for Scotch marketers who are seeking to discover ways to regain support for their brands with a lost generation of drinkers. There is no ready and easy solution to this challenge, although much effort continues to be made to conquer new and younger groups of consumers.

Determining the optimal branding strategy will, at the early stage, mean making choices between stand-alone branding and family branding. In the former case each of a company's brands are unrelated, whereas with family branding multiple brands are linked under a common house name. The familial association can provide an assurance of quality up to a certain level. On the other hand, product branding can enable the firm to address distinct market segments of consumers who may not want to be associated with each other. Also, buyers seeking relatively exclusive products may disdain brands that are ubiquitous, seeking more obscure and esoteric choices in place of the bestsellers. An issue with multiple branding is the cost of sustaining a broad portfolio of unrelated products when the brand-building process is by definition a costly and long haul process. In the end, success rides upon consumer acceptance, with the winning brand's resonating strongest in the consumer's mind. A brand that manages, through a carefully chosen and properly executed positioning strategy, to carve out a unique and relevant position in the eye of the consumer, will be in the best position to succeed.

Routes to market

Channels of distribution – introduction

In terms of the four key marketing variables ('four Ps'), 'place' refers to all choices related to the design, selection and management of the channels of distribution. Coughlan and Stern define a marketing channel structure as a set of pathways a product or service follows after production, culminating in

purchase and use by the final end-user.**59 Some of the key questions that arise are:

- What role do consumer characteristics and demands play in the appropriate channel design?
- Why do marketing channels change in structure over time?
- How should a manufacturer decide what types of intermediaries to use in the channel?
- What problems can arise in the ongoing management of complex marketing channels?

A marketing channel is normally a set of interdependent organizations involved in the process of making a product available. A key point is that the company is not going about its business in isolation. Indeed, channels rely on assembling sometimes complex networks of independent parties – manufacturers, wholesalers, retailers etc. – to serve the needs of the end user. Each one of the parties involved depends on the performance of all the other parties in the chain, and orchestrating this complexity requires discipline from all parties. The interaction of the key players and their ability to work together is a key success factor.

In the chain, the focus should be on the end user irrespective or where any party stands in the distribution chain. Satisfying the ultimate consumer is the goal that all parties in the chain need to address. Indeed, simply passing the product down the line does not guarantee success. The marketing channel structure is a strategic asset for the producer. Managed optimally, it not only allows the product to be physically brought to market but also it helps implement and support the brand's positioning strategy in a consistent manner right through to the point of consumption. Many acquisitions are helped by the ability of manufacturers to obtain distribution synergies and increase their muscle with the trade. In the drinks industry, many recent mergers have been predicated on obtaining large synergistic benefits by channelling previously competing products through a common distribution system. Successful international drinks marketers have channel capabilities that are designed to bring to market a broad portfolio of brands, each with a distinct positioning, and effectively execute the brand message at point of sale.

Reaching the drinks consumer

In the case of alcoholic drinks, it helps to understand both where the product is purchased and where the actual consumption takes place. These two may places may or may not be the same. There are broadly two locations of end usage: on licensed premises (also known as on-premise), or at home. Commonly in the drinks trade the two channels are referred to as the on and off trades. The on-premise trade relates to consumption in bars, hotels and restaurants, whereas the off-trade is defined by channels (including supermarkets and other specialist outlets) that supply the market for retail pur-

chases to satisfy personal consumption at home and with friends, as well as gifting. Internet retail is a new form of specialist retailing often tied with established bricks and mortar retailers. It is also an area where a few producers are offering their brands directly to consumers by way of their own websites. The travel market is an important category that includes elements of both on and off consumption. For example, airlines serve passengers beverages in flight as well as running their own retail shops on board.

Channel design

The orthodox approach to channel design is to start by examining the target audience in terms of end users, and to work back from there. The market needs to be segmented into groups of end users that have similarities in terms of their needs and lifestyles. Defining groups based on their demands should be related not just to the product itself, but also to the service needs of the group. The same product can be in demand from channels with widely varying needs in terms of level of service. We should recognize the value-added element that must be provided to meet the needs of specific groups of customers.

With a clear idea of who the marketer is targeting, an important step is to understand what are the main occasions on which consumption will take place. A decision can follow regarding the most likely type of outlet to focus on. Early on in the decision-making process there is the question of whether consumption will be mainly on or off premises, and the relevant importance of each sector. The answer to this basic but sometimes difficult question will help to determine which channels need to be included in order to get the brand in front of the potential user at the right time and place. The alcoholic drinks trade has always relied heavily upon the on-trade for the purpose of brand building.

The marketing channel challenge involves two major steps: first, selecting the right channels, and secondly, ensuring successful implementation of that design. As discussed previously, the design step involves segmenting the market and understanding the demands of end users. The channel design will focus on utilizing the routes to market that are the most appropriate to reach the main points of usage for the brand. Whoever is the ultimate consumer being targeted, the marketer should be able to tailor the channel strategy accordingly. Until such time as the channel strategy is established, other marketing efforts (for example in advertising and promotion) will likely be wasted resource and energy as long as the consumer can't effectively connect with the product.

The optimum channel is usually selected based on the channel that is the most efficient at meeting the needs and servicing the demand of the specific consumer segment being targeted. There are two main building blocks in channel design: first an understanding of the types of intermediaries that are going to be involved in the particular channel, and then of who they are specifically. The choice has implications not only on efficiency but also on the image of the product, depending on the final selection of retail outlets. It is also necessary to determine the level of intensity with which the channel will be

used. This could range from a high level through to only selective or exclusive arrangements. The company must distinguish between the channels that the company considers necessary and those that should not be used. Some channels will just not be viable because the support required cannot be met by the company's current resources. Knowing what segments to ignore is very important, because it is keeps the channel effort focused on the key segments from which it plans to reap profitable sales.

Invariably the planning will involve the use of internal and external resources in the company. Most channel activities rely upon the interplay between a number of entities – for example, the brand owner may or may not have an own sales force or distribution division to conduct the selling-in of the product to the relevant intermediaries, and the function may be outsourced to a wholesaler of some description. Wholesalers include companies that can either be generalists or are specialized in servicing a limited segment of the market. The critical point to have in mind at all times is the ultimate end-user, and the relative abilities of the chosen intermediaries to execute effective merchandizing strategies and bring the product in front of the target audience in the most appropriate way must be considered. In the case of markets that are managed by exclusive distributors, the choice can have significant consequences on the success or failure of the brands. The level and quality of the relationships of the distributor in their particular country or area of operation can be a key success factor. In the drinks industry, as elsewhere in commerce, fortunes of brands have risen and fallen based on the performance of local market distributors and their abilities to meet their consumer's needs. The trend of global retail consolidation is now confining the role of the exclusive distributor to supporting the overall marketing effort in different ways. The distributor needs to complement the efforts of the producer, who is seeking to capitalize upon relationships with global retail giants to expose own brands more widely to consumers regionally and around the globe. The distributor's task is to have the knowledge to be able to implement international positioning strategies for brands in a way that is relevant and effective in their marketplace. To summarize, marketers have to think global and act local.

The costs and efficiencies of the channel system also need constant monitoring. The challenge that the marketer faces is reducing costs without endangering the service needs of intermediaries. This exercise is constantly being revisited by producers, as such a large amount of cost is tied up in channel management and there is a danger of wasting these resources. Failed channel design and implementation can condemn brands to poor performance and cause profit targets not to be met.

Channel power and conflict

Once intermediaries have been identified and the terms of reference have been agreed with respect to the objectives to be achieved, the discussion on strategy can commence. It is important that a good dialogue exists between all key players in the channel system, especially at the planning stage. At this time it

should be clear to all participants what their roles are and the part they will play in moving the product into the ultimate consumer's hands. Certain players fulfil specialized roles in the channel process, and they need, with all other channel players, to be coordinated so that the entire chain works in harmony. Any weakness in the system will cause the overall performance of the system to suffer.

It may be that a choice is made to opt for a parallel approach, with a number of parties competing within the same channel. If this is the case rivalry will be the norm, and this needs to be managed in a way that it is constructive, not destructive. The manager needs to be able to identify the source of the conflict and differentiate between poor channel design and poor distributor performance. Once the problem is identified, swift action must be taken to reduce the conflict and restore normal operations of the distribution system. Controlling rivalry is often achieved by use of channel power. Power in this context is the ability of one player to control the decisions taken by another member at a different level in the distribution chain. When the disparate members of the channel system are brought together to advance overall performance in line with the overall brand goals, then the channel system is well coordinated. Coordination is an overriding objective of the entire channel management process.

Multiple marketing channels

More and more attention has been focused on multi-channel approaches to marketing. The advent of the Internet has been a catalyst in this debate, because consumers can now be reached through a variety of brick and mortar retail outlets, direct mail catalogues and on-line web stores. The producer needs to recognize the needs of consumers and to adapt the channel strategy to give them the choice and convenience of accessing the product in the manner that suits them. New channels do not replace old ones but tend to be complementary, and may simply allow buyers more flexibility in how they make their choices. With multiple channels, the potential for channel conflict increases. In this context marketers must also manage the issue of unplanned movement of goods between markets referred to as 'parallel' or 'grey'. Intermediaries and dealers are always ready to gain profits from price arbitrage, given that product can easily be moved from one market to another, and prices will naturally be driven down to the level established by the most efficient channel. Even with increasing channel choices, the objective of channel management is still to maintain viable channels to reach the chosen target market segments the company has identified.

Conclusions

Constantly educating the consumer can be very costly, although the payback can be rewarding in terms of market share, sustained uniqueness, a price premium and, ultimately, greater profits. To support this, creating, maintain-

ing and managing the channel system is an important strategic undertaking for the company. The high fixed-cost nature of channel systems necessitates careful consideration of how channels should be structured and managed. Accessing performance information, keeping constant vigilance and having the willingness and ability to respond are some of the keys to successful ongoing channel management.

The product

Historical development

The development of the marketing strategy for any brand requires a clear framework for understanding what the consumer wants in order to know what product to offer. However, his has never stopped entrepreneurs from using their instinctive feel for what consumers want in coming up with new products.

Those who pioneered the development of the large-scale whisky business a century or so ago were confronted with these very choices. Whereas historically the type of product on offer was traditional malt whisky made by the old-fashioned methods pioneered in Scotland and Ireland, a real breakthrough took place in the 1830s with the advent of the Coffey still. The invention of the Patent still by the Irish Engineer Aeneas Coffey allowed producers to offer drinkers a product with a lighter aroma than the traditional malt whisky. From this time forward whisky drinking grew as the taste became popularized and it was developed into a product with mass appeal. In these early days of the industry there was no minimum maturation period, and a large part of the market was represented by consumption of 'on-tap' whisky in pubs and bars around the country.

A campaign ensued at the beginning of the twentieth century to raise the quality standards of the industry. The means chosen was the imposition of minimum ageing rules. Those who proposed this new legislation understood that through the imposition of tighter rules on the manufacturing of the product they would be able to improve the appeal of the category overall and their own brands. This move proved extremely effective, and led to an overall improvement in the Scotch whisky consumer franchise. Producers working together in such cross-industry initiatives are able to help improve quality for consumers across the board, and thereby the category's competitiveness overall.

Premium whiskies

Marketers of Scotch whisky over the years have made many efforts to seek to promote their higher quality special whiskies. The best example of successfully seizing this opportunity to appeal to the consumer's need for more aspirational brands was the development of the aged twelve-year-old blend category. The market was led from an early stage by two leading brands,

Chivas Regal and Johnnie Walker Black. Consumers flocked to these products because they offered prestigious imagery combined with product quality of a high order. The brands became accessories of drinkers whose lifestyle associated positively with the upscale imagery of these brands. The age of the product also became an important criterion for product differentiation, especially with less knowledgeable consumers.

Single malts

Companies had detected a growing demand for products that would cater for a more particular clientele. The scene was set for the development of a new category, single malts, that emerged as a product with appeal to more discerning users. These brands had been closeted for years as hidden treasures and kept away from the consumer limelight except in the North of Scotland, where they historically enjoyed a good following. However, noticing that consumers were looking for a greater choice, some marketers seized the chance and started to make serious efforts to promote these whiskies. One brand, Glenfiddich, acted the locomotive role for the category, and has retained its leadership ever since, spotting early on the first shoots of demand for these products. What this category has done collectively is to take consumers back to the original roots of the industry and the malt distilleries of the Highlands and Islands of Scotland, where they can explore the pleasures that come from the complexity and variety that are the hallmark of the single malt whisky. In recent times there has been a trend towards producing new and interesting product variations, principally by using maturation methods as a variable. Special wood finishes in particular have added new interest to the whisky category, appealing especially to connoisseurs.

Product differentiation

Seeing scope for product differentiation within the Scotch category itself opened up opportunity for growth, and allowed those brands that were able to see the trend to emerge and gain market share. More product differentiation ensued, and today the market for single malts is fragmented as producers have offered consumers more choices to satisfy their curiosity and desire to experiment in seeking new taste experiences. This evolution has helped to alter overall perceptions of whisky, heightening consumer interest and creating new appeal for the category overall.

Taking a broader view, there have been other successful attempts at achieving consumer success through clever product differentiation. A good example is the creation of whisky liqueurs. By marrying the finest whiskies with fine herbs, spices and sugar, whisky liqueurs were able to address demand for a product that would have an appeal to a different user. One brand, Drambuie, stands out above all others. This company managed to create a niche to satisfy drinkers who preferred a sweeter-tasting product. The success of this brand and others that followed was product-driven marketing in action.

Deluxe blends, single malts, whisky liqueurs: these are three manifestations in product terms of whisky made to meet differing demands of consumers. Each of these categories has played a role in adding complexity and value to Scotch whisky in the mind of the consumer. The search for new and better products will continue unabated, and marketers will always be trying to find new niches to exploit. One area marketers of Scotch whisky are wrestling with is the RTD product category, where they have yet to find a formula for success in the way that Smirnoff and Bacardi have with their 'Ice' and 'Breezer' products. The search for innovative ideas will go on as brand owners relentlessly pursue the creation of new streams of income to leverage the equity of their existing brands. Experimentation with new forms of distillation, maturation and other aspects of the product will also continue as part of a concerted effort to find new ways to bring greater satisfaction to consumers.

Packaging – being best dressed

As with human beings, whose clothes send out strong signals of their individual style and character, the same applies to any packaged consumer product. The design and form of the container in which the product is sold sends out messages that the consumer will assimilate, and plays an important role in determining the purchasing decision.

The greatest whisky in the world incorrectly packaged will lack perceived value for the consumer by being wrongly dressed. Having decided on the liquid, the process of preparing all aspects of the product in terms of packaging form one of the cornerstones of successfully grounding the marketing strategy for the brand. No product strategy is complete without bringing together the contents and packaging strategy into a unified plan. Consumers are not attracted by either liquid or packaging alone; it is achieving the right overall mix of both liquid and pack that is the starting point for any serious effort to market the brand. Putting in place the appropriate packaging strategy usually involves a number of key steps, described below.

Forming the packaging strategy starts with defining some parameters that will help frame the choices to be made by all involved in the process. The strategy needs to cover all elements, including the design of the bottle, the labels and any gift and outer packaging. There are a number of participants in the process, ranging from engineers to design specialists. At its most simple, packaging must carry out a physical role in acting as a vessel to deliver the liquid to consumers. The container needs to meet criteria, including size, and the ability to be transported and to be leak-proof. The container will normally also be filled using machines of various types, and consideration must be given to the practicalities and cost of differing forms of bottle.

More important, for the marketer, is a clear understanding of the intended positioning of the brand. Once consumers know a brand's category membership, the focus is usually on establishing a point of difference. Finding a product differentiator is important to consumers, and distinguishes the brand from alternative offerings. The over-riding goal is to establish an understanding of

how the product is intended to be targeted, and what the unique messages are that the marketer wants delivered. By translating this into a succinct brief, the marketer can start to involve other parties in the process of developing suitable packaging. The end result should be in harmony with the intended essence of the brand, its position within the category and, most importantly of all, the consumer's perception of what the product should look like.

Another vital consideration is the trademark. If the brand has already existed for some time, most likely it will have acquired a reputation of its own. This reputation is embodied both in the taste that the consumer is used to and in the element of imagery. This imagery will have been formed through contact with the brand through various means. Users of the brand will have experienced the brand first-hand, and will recognize the trademark and the design and packaging of the product. All these elements come together to create an image for the brand. Non-users may also be aware of the brand through advertising and other means of communication, and may equally hold an image of the brand, albeit possibly without ever having consumed a drop. Brands with history have historical baggage, and these elements, including the brand roots, can be factored into the brand positioning and, ultimately, the design brief. The designer will create an end result in terms of logos, typeface, symbols and overall design, keeping all elements in harmony while at the same time giving the brand some differentiating features that help it stand out of the pack. Conformity is a double-edged sword in the world of packaging; it can send out strong category clues, but may be a poor means of achieving differentiation. Creating highly individual presentations can be successful (if kept within the boundaries of what consumers will accept), or can fail completely because they do not conform to category norms. To illustrate, one company seeking to differentiate its main brand from the pack but wishing to remain firmly within category norms decided to adopt a triangular bottle as its trademark design. From that day forward the brand in question, Grant's, developed a personality of its own while continuing to conform overall to category requirements.

The importance of making correct packaging decisions is a reason why there are advantages in conducting exhaustive technical and consumer research in advance of putting products to market. The objective of most research of this type is very often to counter the risk of taking incorrect decisions that could jeopardize any efforts and result in wastage of scarce resources. However, care must be taken not to put one's entire faith in such studies, and to trust one's natural instincts to take the right decisions. Many a marketing decision in the field of branding generally, and packaging specifically, has been left to the instinct of the marketer. Some people develop a strong instinct for what works and what will appeal to consumers. Left to their own devices, these people are responsible for many of the best-dressed and presented brands.

Another key issue with packaging is the need regularly to adapt the presentation of the brand. This adaptation will have two goals: keeping the positioning in line with the desired strategy for the brand, and ensuring that the brand is appropriately dressed for the times. Just as in the car industry, yesterday's model with time looks dated, and drivers want to be seen behind the

wheel of the car that reflects their image. Likewise, drinkers are making a statement about themselves every time they order a particular brand. So the brand needs to outwardly reflect that image, and the look and feel of the bottle and label play major roles in sending our visual signals about the brand's personality. Many marketers follow the Unilever adage of using the 'bacon slicer' approach, and make regular and gradual adjustments to the packaging of their brands. Only if a brand is encountering a serious problem in terms of market share, volume sales or other key performance indicators should radical changes to the packaging be contemplated, remembering that product packaging is only one element of the overall marketing mix. Radical change risks confusing current users and losing their loyalty and custom. If such change is not heavily outweighed by new customer acquisition, to replace lapsed users, the game can be lost completely.

Managing price

The importance of price

Price is one of the 'four Ps' in the marketing mix, and in terms of the decision-making process it is important that finance and accounting work closely with marketing to reach the right policy. Decisions relating to price management are key drivers for any company in achieving profitability and long-term shareholder value. Today's consumers are even more determined to search for the very best value for their hard-earned cash, and are increasingly willing to change their buying habits in their search for better value propositions. Value is an aggregate of the functional and emotional benefits that the product creates – for example, while relatively expensive, Dom Perignon offers good value for money to many buyers of champagne. Effective price management is, however, key to developing and building our brands in both the short and long term.

Of all the components of the marketing mix, price is the only one that directly generates income. It has an immediate and direct impact on a brand's ability to generate revenue. All the other components (promotion, product, packaging, and distribution) generate costs and represent investments.

If the strategic objective is profitability more than market share, the marketer will price less aggressively and seek those target segments that are willing to pay the set price, rather than cut prices to appeal to a larger segment. It is not very realistic to expect to maximize both profits and market share in highly competitive markets.

In overall terms, the price a marketer asks consumers to pay for a brand:

- Drives the perceived value of the total proposition offered by the brand
- Provides a key signal of product quality and competitive positioning of the brand
- Dictates the level of funds available for brand building investments.

Price directly influences consumer brand choice, and has a critical role to play in creating the equity of the brand. If the overall influence is positive, the brand can develop successfully to achieve its maximum profit potential. Pricing, in combination with the benefits associated with the product, its packaging and such promotion as is carried out, is central to the development of a brand's perceived value and its ultimate success in the marketplace. Financial contribution generated through pricing also enables further investment in marketing activities of all forms, and is the foundation for maximizing long-term profitability. Thus, achieving the optimal price/volume/equity equation will improve the company's chances of maximizing long-term shareholder value.

There are a number of formulae that can be used for setting price, but very few can be used in total isolation. Some of the major determinants of the pricing decision for marketers are set out below.

Cost of goods-related pricing

The product's variable cost is generally the pricing floor. This section examines each of the major components that are relevant to building the cost structure of a Scotch whisky brand.

One of the main cost factors that plays a large role in pricing within Scotch whisky (and alcoholic beverages in general) is the taxation element. It is commonplace in most countries to impose some form of excise duty on alcoholic beverages, and a relatively high rate has tended to be imposed on spirits compared to other forms of alcohol, including beer and wines. Whatever the specific level of tax, all marketers must factor duties and other taxes into their cost equations in order to calculate the cost of goods. In some countries the tax component may represent well over 50 per cent of the final consumer price.

Given the heavy tax burden, marketers are obliged to some degree to use cost-plus calculations when determining the price at which they intend to sell. Another element that complicates the equation when considering cost-plus pricing is the level of overheads allocated to a particular product. Generally allocations are made on an arbitrary basis, based on the expected operating level of the company and factors or keys that are determined by the accounting department. Depending on how these keys are set, each product ends up picking up a share of the company's overhead and this is in turn factored into the calculation of cost. The third component of cost is of course the direct costs of the product, and there should be little doubt as to the accuracy of these.

The three main cost elements – tax and duties, direct costs and overhead allocation – can then be added together and the marketer can consider what mark-up or margin the product can bear. Obviously this decision can't be taken in isolation of other factors, such as the price of competing brands, but the benefit of this type of analysis is to allow companies to determine, out of their portfolio of products, which are those that can generate the highest economic profit.

A variant on cost-plus pricing is using pricing to fill the profit gap to meet the plan, or where a price increase is dictated by the planned profit needs of the business. This approach could be seen as short-term thinking, and may have the potential to damage the long-term health of the brand. It is also based on the premise that price increases always result in an increase in profit, which may or may not be the case depending on the price elasticity factor.

Competitive set pricing

Companies can also set pricing based on what the competition is charging. This is referred to as competition-oriented pricing. In this scenario the marketer keeps a watch on competitive pricing and sets the company's prices relative to one or more competing brands. In this situation the company is not maintaining a strict relationship between demand for the product and cost.

The most popular type of competition-oriented pricing is where a firm tries to keep its price at the average level charged by the industry, also called going-rate or imitative pricing. Product differences, whether packaging, quality, or other product features, serve to desensitize the buyer to existing price differentials.

Matching competitor price moves is often the chosen strategy on the grounds that this represents a least risk approach. This assumes that competitors know what they are doing, and ignores potential competitive advantage from alternative pricing strategies. In other words, pricing a brand solely in relation to a competitive set is usually not advisable, although for brands that are followers in the market this form of benchmarking is unavoidable, and failure to heed the impact of changes in competitor brand prices could have serious negative consequences for the marketer. In this context, price elasticity (which is discussed below) is important because it determines the way in which the volumes of brands fluctuate when price adjustments are made both in absolute and in relative terms.

Market-oriented pricing

This refers to pricing a product according to what the market will bear, which tries to exploit perceived brand strength and competitive weakness. Pricing to what the market will bear risks pushing the price too far, thus damaging its perceived value and the long-term potential of the brand. Pricing a brand too highly may create opportunities for lower-priced competition to develop, which may change the category dynamics altogether, gradually cannibalizing market share.

The product, including its features, brand name, mode of distribution and of the communication, creates the value. The price captures the value. Pricing is a complicated decision, because value can be manipulated. It is therefore hard to establish objectively the market price that any product can bear in isolation of other considerations, such as the competitive set of products.

It is sensible to consider the value of a particular brand to a targeted group of consumers, but equally to be able to ascribe pricing levels in a vacuum or without reference to other products is almost impossible. This stresses the complication of the pricing decision-making process, and the degree of skill and understanding that is required by marketers to take the correct decisions.

Pricing management principles

International Distillers and Vintners (IDV), now part of Diageo, identified for their brands three basic principles that need to be carefully considered before taking any important pricing decisions for a brand:

1. Price elasticity
2. Price positioning
3. Perceived value.

Price elasticity

Price elasticity refers to the ratio of percentage change in demand (quantity sold per period) caused by a percentage change in price. It is defined as the percentage increase/decrease in sales volume that would be produced by a 1 per cent decrease/increase in its relative price within the competitive set of the category. Price elasticity is always negative. The higher the price elasticity value, the bigger is the response of sales to each 1 per cent change in price. If a brand has a price elasticity of between 0 and −1, then demand for the product is said to be 'inelastic' because the percentage change in sales is less than the percentage change in price. If the price elasticity is above −1, however, then the demand for product is said to be 'elastic' because the percentage change in sales is greater than the percentage change in price.

Determining a product's price elasticity helps to answer the question regarding what happens to category share if the company changes retail price relative to its competitors. In practice price elasticity is extremely difficult to measure, and is dependent upon a high level of data and relatively sophisticated analysis techniques. However, the price elasticity of a brand is a good indicator of its relative strength within the category. Brands with low price elasticity will, by definition, have a high proportion of loyalists amongst consumers and/or have fewer competitors. Brand strength can be monitored by comparing category share and price elasticity. The effectiveness of cumulative marketing effort can be measured by its effect on reducing price elasticity. Price elasticity can also vary according to how the competitive set is defined. This makes it important to define the most appropriate category and competitive set for the brand in any particular market, to ensure that a meaningful price elasticity value can be derived.

In general, higher-priced brands and brands that have been developed through consumer marketing-led strategies tend to have lower price elasticity than lower-priced brands and brands that have been developed through sales-led strategies. Successful brands usually have more powerful 'value

added' reasons to buy other than just price – image, taste and convenience, for example.

For most mature brands, elasticity changes slowly over time because brand equity takes time to build and relative prices tend to move slowly within a narrow range. Generally in mature categories, the price elasticity factor is only disturbed by major external factors such as large duty increases or severe competitive price-cutting. It should also be borne in mind that price elasticity operates at many levels: markets, categories, brands, different product lines and sizes within a brand all have price elasticities, which may vary considerably.

Price elasticity helps determine whether a price promotion will generate a sufficient increase in sales and profit for the promotion to pay back. If a marketer doesn't know the price elasticity of the brand, it is unwise to run a price promotion or change its price. It is also important to consider competitors' sensitivity to changes in price differentials, and their likely action, before making price change decisions. Price elasticity analysis can help identify when a brand moves outside its 'acceptable price' band – when it changes its price position to such a degree that consumers re-evaluate their brand choice. The edges of these price bands are sometimes referred to as price thresholds. Even unique brands that may dominate a category have thresholds beyond which consumers stop buying, switch to a competitor, or move outside the category.

Price positioning

This principle relates to pricing a brand at the optimal long-term level in relation to its competitors. Consumer markets tend to segment into distinct price bands, often some distance apart, and each price band is associated with a level of product performance or quality. Depending on the market structure and the price gap between segments, there is scope for positioning products at a different price point within a segment to try and create a competitive advantage.

Taxation, referred to above as a key cost driver, can also create significant distortion on both the definition and spread of price segments. The low price segment often accounts for a larger proportion of the category in higher taxation markets. To retain market share, brands in the standard and premium price segments must manage price relative not only to other brands within their own price segment but also to the cheaper alternatives on offer.

The emergence of own-label brands and low-priced commodity labels has meant that the floor level pricing in the spirits market generally, and Scotch whisky in particular, has dropped significantly in real terms. This phenomenon, sometimes referred to as commoditization, has an adverse effect on category dynamics, puts prices of standard and premium brands under severe pressure, and destroys economic value within the categories overall. No simple remedy exists for this situation, which any single brand owner is generally powerless to stop. In the long run the trend towards commoditization will polarize markets in two directions. Many consumers will seek the reassurance brands with trusted values and continue to support those brands that are

perceived to offer sufficient added value. On the other hand, large groups of consumers will move in the direction of cheaper brands and will not display the same level of loyalty. They will be more inclined to switch preference based upon small reductions in price.

Once a brand has a well-established positioning image, it becomes difficult fundamentally to change this positioning and adjust relative price pricing over time. A consumer's predisposition towards a standard category brand generally creates barriers to trading up to a higher price, even after improving brand benefits. It is also difficult for a brand to trade down into a lower price segment, as short-term volume gains may not generate sufficient incremental profit for this to pay back. There is also the danger that any short-term gain will be offset by long-term erosion in brand image, which would reduce the brand's capacity to sustain its volume in the long term.

However, a brand may have sufficient scope to move price within its price segment, subject of course to its price elasticity. In general, leading brands within the segment may have the ability to move their relative price up or down within the segment; weaker secondary brands more usually only move price down. Leading brands tend to have lower price elasticities, whereas secondary brands usually do not and the only option that is available to them is to price-down relative to brands in the same price segment. Another common strategy for brands wanting to appeal to a wide target audience is to offer a range of products covering a variety of price points.

Perceived value

It is useful to understand how consumers link the perceived benefits of a brand to its price. Perceived value is a trade-off between perceived benefits and the price the consumer must pay to acquire these benefits. This is a qualitative measure in the sense that it refers to the assessment the consumer makes in the appraisal of the total benefits that are being offered by a brand in relation to the price being asked for. The higher level of price must be balanced in the consumer's mind by a higher level of benefits. Products that offer more benefits will be perceived to deliver more value, and thus improving the benefits can support a higher price, or a higher share at a lower price. Moving to a higher price without offering more benefits (whether functional or emotional) to compensate will very likely lead to share erosion.

Advertising and promotion

The brand-building process

The marketing mix is rounded out by the fourth 'P', which plays a key role in developing demand. The marketing of alcoholic beverages is no different in this regard to that of other consumable products. Manufacturing a good product and bringing it to market at a fair price is usually not sufficient to max-

imize the potential of any brand. Normally, every time a brand or category is being developed the product is being promoted in some way or another. In the case of a Scotch whisky it may range from a barman recommending the product to customers to a broad-reaching advertising campaign designed to build awareness and product trial. Whatever the means of promotion chosen, the ultimate goal of all brand builders is to maximize the value of their products in the eye of consumers in order to achieve a share of the market and, ultimately, make a profit.

Marketers use a variety of tools to promote their brands. The overall aim of promotion is to build bonds with consumers by means of communicating messages to them. With consumers, for relationships to be lasting and of value they generally need to be emotional in nature. The message also needs to convey real values and be communicated in an effective manner. Being sure that the message is targeted at the right audience is part of ensuring the wise use of resources and gaining a positive effect. In summary, with promotion the key issues that need to be answered are how much to spend and in what way.

So how can promotion be defined? Kotler's definition is that promotion encompasses all the tools in the marketing mix whose major role is persuasive communication (Kotler, 1999). There is a whole raft of tools that fit this definition, but in overall terms they can be broken down into four broad categories:

1. Advertising
2. Personal selling
3. Public relations and publicity
4. Sales promotion and merchandising.

Each of these basic four components offers a wide panoply of alternative means to communicate with the consumer. 'Above and below the line' is another form of referring to marketing activity; the former refers to advertising, and the latter to all other activities. This section gives only ageneral overview of each of the elements of the promotional mix.

Promotional activity can cover a very wide range of actions, but the common denominator is that it is normally targeted in nature and is objective-led – i.e. it is aimed at achieving set consumer goals. In other words, the purpose is to achieve a behavioural outcome on the part of the targeted audience. Planning activity is an important discipline that all marketers attempt to adhere to, although *ad hoc* activity can, with luck, yield good results. Having clear consumer goals is vital in order to know what aspect of consumer behaviour the marketer is trying to influence – for example, is it to try and encourage trial, to persuade consumers to adapt the product as one of their main brands, or simply to get the consumer to become aware of the brand? A structured approach to advertising and promotion that has measurable targets and is properly tracked for effectiveness will help ensure that the expenditure impacts on the consumer. But it is wise to heed the comment by Lord Lever, the soaps and detergent 'king', who mused that only half the money spent on advertising was effective, and then went on to complain that no one could tell him which half it was?!

Advertising

Advertising consists of paid-for messages by an identified party using a wide array of media to reach the target audience. The purposes of advertising are manifold, but can usually be broken down into two underlying objectives: to convey awareness of the product and to establish a clear image of it in the minds of the target audience. In other words, advertising plays a dual role: informing consumers and getting the brand more 'top of mind', and developing an image of the product. Advertising very often plays a critical role in establishing a brand's equity. It can be particularly effective when a brand is differentiated from its competitors on dimensions that are important to consumers. Consistently advertising a brand's position also serves as a barrier to competitive entry.

The company's approach to planning any communications will be guided by the brand positioning statement and a definition of who is being targeted. Developing this statement will of course depend on a discussion on consumer insights, these being the foundation for effective advertising. What consumers believe about a brand and the category is one important element of the consumer's beliefs; the other is how consumers use advertising information to make brand decisions. With effective consumer insights leading to a clear statement of the brand positioning strategy, the marketer can commence developing communication objectives. Leading up to this phase there will usually be some market research carried out to reveal consumer insights – usually using focus groups, in-depth interviews and surveys. These lay the foundations for designing creative and media strategies. The company also has commercial goals for the brand in terms of targets for recruitment of new users, on top of the important job of retaining the brand's existing consumer franchise. With a clear idea of what needs to be communicated, to whom, and with what intensity, an agency can be given a detailed brief and can start the process of recommending a communications strategy to fit the overall brand objectives. At this stage, close cooperation is often needed between all the actors involved in the nascent marketing campaign. The company marketing team, advertising agency and research company team all need to be put to work in a coordinated manner to plan and execute the development of the communications campaign.

The alcoholic drinks industry spends relatively large amounts on advertising. This is not necessarily the case for Scotch whisky, where absolute levels of expenditure have not generally been high and relative amounts have not been great when compared with expenditure on other drinks categories. With few exceptions, drinks advertisers use the media in order to provide messages to consumers about their brands in order to sustain and grow their share of the market. The motive can often be defensive, particularly for brands that are in leadership positions and where consumers need constant reminding of their benefits. Advertising may also be playing a role in the drinks industry in promoting the much talked about trend of consuming 'less but better'. Whatever the case, much emphasis is placed on developing strong brands and on creating the foundations for a product's unique image.

Advertising based upon accepted consumer beliefs is generally more effective than advertising requiring consumers to change beliefs. Most often, therefore, advertisers will develop messages that conform to accepted beliefs. If this is not possible because the brand equity is out of line with the consumer's beliefs, the knowledge of the beliefs can be used to develop arguments to change the consumer's disposition. It is also useful to understand how consumers' process the advertising messages received. It is commonly realized that they process the information of what is said about the brand in relation to their current repertoire of information about the advertised brand and its competitors.

Brand linkage

The elaboration of a brand's benefits is obviously effective only if it is linked to the brand name. It is clearly useful to introduce information about a brand that is consistent with what people already know about it. An example of this approach was a campaign for Cutty Sark whisky, a brand recognized by drinkers for its tall ship sailing logo. The brand was losing share at one point, and launched a campaign: 'don't give up the ship'. Subway advertisements in New York said: 'When you have had it with graffiti, don't give up the ship'. The slogan was memorable because it was the cry during the Revolutionary War, and also reminded consumers of the symbol of the brand. Sales of the brand were seen to recover at that time.

However, brand linkage is difficult to achieve. The reason is that a persuasive campaign has more associations with a person's life than with a brand name. The small number of associations that people generally have in relation to brands makes them hard to remember. On the other hand, brands that are rich with associations in people's minds are easier to recall – for example, Coca Cola™ for soft drinks and Bacardi for spirits. It is less difficult with these well-known brands for consumers to link the message to the brand, than for products that are less prominent in their daily lives. Brand linkage is even less likely to work when the brand does not have a strong point of differentiation. When several producers use the same messages, the linkage between brand and message is weakened further.

Creative strategies

The aim is obviously to find the message that will induce the desired action by the consumer. There are a number of creative strategies integral to the delivery of the message that can help consumers evaluate an advertising message. For example, using message discrepancy between the position advocated in a message and people's present beliefs is one potentially strong motivational device. **60Persuasion is usually only minimal when the communication argues a position people currently hold maybe only reinforcing people's current beliefs. On the other hand, a message that is very discrepant with the consumer's current views may just prompt disbelief. An approach somewhere in the middle is therefore usually preferable.

Threat appeals are another strategy employed, although not all threats will result in a persuasive response. Focusing too hard on dire consequences may just bring about an emotional response. If this approach is to be used, it is important to focus on helping people to recognize danger and be better able to cope with it. More common is to use humour as the means to persuade the consumer. An advantage is that this usually succeeds in getting the consumers' attention and generally also motivates them to process the message. However, there are serious drawbacks, and humour is not always effectively used in advertising. To work to good effect, the humour should generally be related to conveying the brand benefit and ideally be focused on the product itself. If the focus can't be on the product, it may be better to focus on non-users than on the actual consumers.

Media strategies

The resources available to run any campaign are always limited, and therefore there must be wise use of media in planning any advertising. Media strategy starts with gaining an understanding of who the target consumer is. In order to establish how to target the consumer, a view also needs to be taken regarding the target's media habits. In overall terms, media selection is a trade-off between reach and frequency. Reach relates to the number of people in the target who have been exposed to a message during the period of the campaign; frequency is the average number of times the target see the message during the campaign period. For a given advertising budget increasing one means reducing the other, and this trade-off is a key decision that advertisers must take. There is no evidence pointing to any one correct way to manage this trade-off. More relevant, perhaps, than the amount of times an audience sees one message is the type of exposure.

Krugman (1977) has advocated three types or stages of exposure to advertising:

1. Establishing that a brand belongs to a category
2. Focusing on the brand's point of difference
3. Prompting a decision by the consumer.

The overall focus is not so much on the number of exposures that an advertisement is given, but rather on the fact that consumers respond to mounting exposures in different ways. In overall terms, persuasive impact increases with every impact, but substantial repetition prompts a decline in message impact. This is commonly referred to as campaign or message wear-out. To overcome this phenomenon, advertisers commonly use the technique of changing the context but maintaining the central theme. The problem with this approach is that unless the change in context offers consumers new insights it is hard to sustain their interest, and the message is simply not processed. Wear-out can be forestalled by presenting new information to stimulate the audience to process the message.

Another dimension of the media strategy is when to advertise. Continuity throughout the year is an expensive proposition and is affordable only to the

biggest brands. On the other hand, brands can use a concentration strategy, focusing the campaign on periods where consumption is historically highest for the category. The danger with this approach is that it can become self-fulfilling in terms of concentrating brand sales even further and exacerbating the seasonality in the brand's sales. Hybrid approaches try and seek the middle ground. Another media strategy is 'flighting', where a campaign is run in short bursts interspersed with periods of silence. The idea with this approach and other variations on the same theme is to get the impact of concentration along with the sustaining value of continuity.

When dealing with the issue of tight budgets, the advertiser has the choice between narrowing the target or reducing the media used to target the audience. Reducing the geographical scope of the campaign and the times during the year when there is an advertising presence are other simple means of restricting the reach and frequency of the campaign. The normal objective is to have a strong presence among as large a proportion of the target audience as is affordable.

Integrated marketing communication

The principle of integrated marketing communications is not a novelty for drinks marketers. This refers to the broad range of communication tools that are available in the armoury of the company to spread their messages to consumers. Apart from advertising, these include public relations, direct marketing, event marketing and Internet marketing, and each are explored in a little detail below. Of all these tools, while advertising is the largest in terms of the total amount spent, it does not always play the leading role. Its role depends on the brand objectives and the audience being targeted. Indeed, there are some well-known and not unsuccessful brands that choose to ignore paid-for media and concentrate on other communications means to get their message over. In spirits the majority of brands tend to attach importance to advertising because it allows the brand to be brought to the attention of a wide audience, but there are many niche brands, particularly at the premium end of the spectrum in terms of quality, that eschew mass media and concentrate on word of mouth recommendation, perhaps supported by some public relations and event marketing. The over-riding principle normally applied is that the message about the brand needs to have a consistency across all channels of communication. If an integrated campaign is properly planned and executed, the synergy of the same message appearing in different manners can be highly effective and lead to building intimacy with the customer.

Measuring effectiveness

The last step in the communications process is to measure the effectiveness of the campaign. There is a range of standard measures to be considered, but all ultimately point at the key question of whether the target audience has awareness of the brand resulting from the campaign. Then, more importantly, what

does the consumer know and believe about the brand and its personality? This is usually captured by measuring the attitudes of consumers towards the brand, including their likes and dislikes. This is usually in the form of quantitative market research, and through repeated tracking of such variables the marketer starts building a picture of how the attitudes of consumers are evolving through time. The research findings can be useful in picking up warning signals and allowing the messages and appeal to be modified to attempt to improve the position of the brand in the consumer's eye.

Direct marketing

One of the key tools employed by marketers, direct marketing has many similar attributes to advertising. It is paid for, and allows the marketer to enter into a direct dialogue with the consumer and to transmit a message. Direct marketing has become a very widely used medium for communication with consumers, and one of the main reasons is that the brand owner can generally target messages with a fair degree of accuracy. Its impact is also often easier to measure. Indeed, the principle of much of direct marketing is that companies identify their target audience and then seek to establish a mailing list to match. Another group that marketers often try to target by way of direct marketing is their existing customer base. Names of a company's customers can be a valuable source of continuing revenues, and customer loyalty programmes can be conducted to keep interest in the brand high by way of an ongoing dialogue with the consumer.

Direct marketing does have limitations. Its effectiveness has been undermined by the constant barrage of unsolicited materials that consumers receive through their letterboxes and that fall out of their newspapers and magazines. However, the growing use of the medium is testimony to the fact that response rates in the majority of cases more than cover the direct costs of the campaigns, thus yielding profits. In more recent times, a new dimension to direct marketing has emerged in the form of telemarketing. In this case consumers receive unsolicited calls from brands offering their goods and services.

In the context of drinks, direct marketing can be a useful tool, particularly in the form of targeted direct mail. Premium brands can derive good value from this using this medium, sending promotional literature, perhaps augmented by a special promotional trial offer, on the brand.

Public relations

In the view of some, this is one of the most potent means for transmitting messages about brands. In this case the objective is to transmit the message to the audience in an indirect manner. Once again, starting with careful consumer targeting allied to achieving clarity with the message, the strategy is to have the message relayed through other organs. A key point of difference with advertising is that the featuring or transmission of the message is not paid for directly. The conduit for relaying the message in general tends to be the media,

where the journalists, writers and other contributors to the media write and/or talk about the product. Of course, a major challenge with all PR is to ensure that the message stays intact and close to the intended line. Getting the message through to the intended audience can be a challenge, given that the transmission of the message is not under the company's direct control.

Public relations is a must for most marketers of drinks. One reason is the interest that the sector's products attract from the media. In this context, relationships with the writers need to be well maintained so they are kept abreast of the developments of the company and its brands. Consumers of drinks, and particularly connoisseurs, are avid readers of the newspaper columns, and a brand should aim to get a good share of voice in this area and to have the right things said. Negative PR is generally avoidable, providing the brand owner is careful to foster good relationships with the key writers and experts in the field.

Event marketing and sponsorship

This tool can be one of those with the highest impact. The principle involved is to relate the brand to an event that is appropriate in terms of the audience and message that the marketer wishes to convey. For example, to promote new-age ready-to-drink spirits, brands may look for events that are attended by a younger audience and that have a fun and social aspect. At the heart of a good event and sponsorship campaign is the skill of carefully matching the brand personality to the event. A failure to do this can lead to sending misleading messages about the brand to the consumer, whereas if the association is strong an element of synergy can be built up and such promotions can run effectively over long periods.

The sporting world has always been an area targeted by drinks brands. Obviously the brands are attracted to the audiences, particularly those who offer a good match with the consumer profile of the brand. An example of this is the case of the long-standing sponsorship of the Scottish Rugby Team by Famous Grouse. In this case the relationship has been prosperous for the brand by association. The effect transmits a clear message in terms of the Scottish credentials of the brand, as if it had been chosen on its merits. The message has reached audiences outside of the home country and assisted in building the awareness and credentials of the brand more widely. The leading Scotch brand worldwide, Johnnie Walker, has in a similar fashion stamped its mark on the sport of golf in a consistent manner over the years, thereby using the sport to transmit a constant message about the prestige and quality of the brand to a global audience.

Internet marketing

A newcomer to the suite of marketing tools, Internet marketing is still evolving as brands experiment to find the optimum uses afforded by the on-line medium. In the most simple terms, there are opportunities to feature banner adver-

tising on websites. Most observers doubt that Internet advertising is going to replace the traditional forms of media; indeed, it is already apparent that this form of advertising suffers from a low rates of 'click through'. Once the consumer has clicked though to the brand's web page the message will normally be transmitted, and for this to take place the brand needs to have an effective home page and site. The medium obviously lends itself to advertising products where the consumer's need for information is relatively high. Consumer non-durable goods are not generally in this category, and alcoholic beverages are no exception. So while most producers have established elaborate websites as homes for their brands on the Internet, many of these sites have low visit rates. The Internet can also be used in a direct marketing context. By collecting email addresses of interested consumers and entering into a dialogue with them, companies have found they can build a relationship much more quickly and for less cost than with conventional direct marketing.

Conclusion

Advertising is an inexact science, as is marketing is overall. Intuition plays a large role in finding the messages that build consumer appeal and create brand equity over the long run. Planning tools are also there to help make rational choices and approach decision-making in a disciplined and structured manner. In the final analysis, the impact of advertising depends to a large extent on whether it resonates with the consumer. Indeed, much of the impact of advertising works through self-persuasion. Highly effective advertising will tend to build a strong link between the brand and the perceived benefit to the consumer, and this is often achieved by clearly distinguishing the brand from its competitors using a unique style of creative execution. With alcoholic beverages there are limitations on the mediums for advertising, and care needs to be exercised to respect both the legal framework and any voluntary codes of conduct. Common sense is required to ensure that the messages are responsible and do not mislead in any manner. The question of how to publicize the message, or of the choice of media, is another important step in planning the communications strategy for the brand. There are a myriad of ways and means for conveying the message, and this represents a vast specialized discipline in itself.

Market research

In today's world, the combination of strong competition and scarce resources means that advertising and promotion needs to be approached with a high degree of professionalism. This has led to the emergence of the market research industry, where the positioning of products is exhaustively explored to find optimum strategies. All the elements of the promotional mix are also put through tests to ascertain whether they are likely to produce the intended

results. This might, for example, include establishing what image an advertisement for a particular brand creates in the minds of all targeted consumers. Communications are often pre-tested in 'rough' before large budgets are spent on producing the final advertisement. The answer to these questions and others will indicate to marketers if the message is in line with the positioning statement, and give them the confidence to commit expenditure of scarce financial resources. On the other hand, a poor diagnosis sends out warning signals and allows the marketer to go back to the drawing board and amend any aspect of the marketing mix. In all of this it is worth stressing that market research should not be a substitute for common sense. Sometimes the solution is obvious and it is not necessary to incur valuable resources in terms of time and money to conduct long pieces of research. It is particularly important to interpret any data coming from market research studies very carefully, to avoid falling into misinterpretation traps.

Used properly, market research can be a very powerful tool with which to explore all aspects of the brand development process. The usefulness of research can range from the basic diagnosis of consumer needs to mapping out potential new product development strategies, and more specific testing of campaign material for a well-established brand. It is usual to distinguish between two general types of research: exploratory and evaluative. Assembling the right team of experts to conduct any form of research and interpret the results is a good starting point. Also, it is vital to give the team a carefully prepared and thorough written brief. Without a clear understanding of the background and the objectives of the research, money can easily be wasted and, in extreme cases, misleading findings can emerge that give spurious information leading to wrong decisions. If research indicates that consumers prefer brand X over brand Y, it doesn't always lead to the former being more successful, because perhaps there may be other deeper factors motivating brand selection that the research has overlooked and have not been volunteered by the research sample. Another classical trap in market research is to have an ill-defined target that results in sampling consumer groups who are not representative of the actual target audience.

General conclusions

The Scotch marketer of today must at all times remain focused upon the needs, desires and preferences of the consumer. It also helps to be aware of the broader alcoholic beverage market in which producers are competing, and to recognize that Scotch whisky is just one of the many choices that the consumer has to select from. With the eye firmly upon the consumer during all stages of the marketing process, the chances of success and continuing prosperity are greatly increased. The customer is said to be 'king, queen and master', and being on the lookout for consumer trends and meeting consumers' needs is the task of every marketer. Therein lies the route to long-term profitability and success.

Acknowledgments

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