

# Chapter 11

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## Production of Scotch and Irish whiskies: their history and evolution

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### Introduction

Whisky is the potable spirit obtained by distillation of an aqueous extract of an infusion of malted barley and other cereals that has been fermented with strains of *Saccharomyces cerevisiae* yeast. Various types of whisky are produced in a number of different countries in the world. They differ principally in the nature and proportion of the cereals used as raw materials along with malted barley, and also in the type of still used for distillation.

The principal types of whisky are also characteristic of particular geographical regions of the world. In Scotland, the characteristic product is manufactured using only malted barley as the raw material; and the fermented malt mash is distilled in relatively small pot stills.

The product, known as Scotch malt whisky, is produced in small distilleries, of which there are over 100 in Scotland. Scotch malt whisky is marketed both as a straight malt whisky, many brands of which have recently become extremely popular throughout the world, and also as a blend with another type of whisky produced in Scotland known as 'Scotch grain whisky' or (because it is distilled continuously in

Coffey-type patent stills) as 'patent-still whisky'. Most Scotch whiskies available on the international market consist of blends with 20-30% malt whisky and 70-80% grain whisky. Within the blend, there may be as many as 20-30 individual malt whiskies and grain whiskies. These blends are, by law, matured for at least three years but in practice this period is much longer. Unblended Scotch malt whiskies are usually matured for a minimum of eight years.

The cereals used in the manufacture of Scotch grain whisky are malted barley, together with a high proportion (up to 90%) of wheat or corn (maize). Currently wheat is the main cereal, chosen on the basis of cost and the attraction of using a Scottish-grown cereal. All whiskies are legally protected and defined, mainly because of the huge revenues that governments obtain from their sale. The Scotch Whisky Order (1990) and the Scotch Whisky Act (1988), in defining Scotch Whisky, state that to be called Scotch Whisky spirits must be:

1. produced at a distillery in Scotland
2. from water and malted barley to which only whole grains or other cereals may be added
3. processed at that distillery into a mash

4. converted to fermentable carbohydrate only by endogenous enzymes
5. fermented only by the addition of yeast
6. distilled to an alcohol strength less than 94.8% so that the distillate has the aroma and taste derived from the raw materials
7. matured in Scotland in oak casks of less than 700 litres for a minimum of three years
8. be a product that retains the color, aroma and taste derived from the raw materials
9. produced with no substance other than water and spirit caramel added.

The word 'Scotch' in this definition is of geographical and not generic significance.

Irish whiskey (spelled with an e) is a distinctive product of either the Republic of Ireland or of Northern Ireland. In the Republic of Ireland, definitions were enacted by the Parliament in the Irish Whiskey Acts of 1950 and 1980. The 1950 Act distinguished pot still whiskey from blends and stated that the title 'Irish Pot-Still Whiskey' was reserved solely for spirits distilled in pot stills in the Republic from a mash of cereal grains normally grown in that country and saccharified by a diastase of malted barley. The 1980 legislation specified that the term 'Irish Whiskey' only applied to 'spirits distilled in the Republic or Northern Ireland from a mash of cereals saccharified by the diastase of malt contained therein, with or without other natural diastases'. This meant that unlike Scotch, Irish whiskey may be produced with the use of microbial enzyme preparations in addition to malt. The 1980 Act also specified that the whiskey must be aged for at least three years in Ireland in wooden barrels. While not possessing the 'smoky' taste and aroma of Scotch, Irish whiskey is usually more flavorful and has a heavier body than Scotch. Moreover, the whiskey is distilled not twice, as in Scotland, but three times to give a very strong spirit of 86° GL compared with the 71° GL whisky distilled in Scotland.

The tremendous popularity of whiskies manufactured in Scotland, Ireland, the US and Canada has prompted several other countries to try manufacturing whiskies, usually ones

designed to resemble Scotch. Indeed, the number of countries with minor but nevertheless significant whisky-distilling industries must now be well over a dozen. Some countries, notably Australia and Japan, have whisky-distilling industries producing a sufficiently acceptable product for them to venture into the export industry. Other countries, including the Netherlands and Spain, have whisky-distilling industries that cater mainly, if not exclusively, for home consumption. The measures that some of these industries have taken to imitate Scotch become apparent when the spirits are sampled. One of the two whisky distilleries in Spain that located northwest of Madrid in the Guadarrama Hills near Segovia produces a very acceptable Scotch-type whisky. Its quality is attributed in part to the fact that the water used, which comes from the surrounding hills, closely resembles that used in highland Scotch whisky distilleries.

Recommended descriptions of Scotch whisky manufacture have come from Brander (1975), Daiches (1969), Dunnett (1953), Gunn (1935), Robb (1950), Ross (1970), Simpson (1968), Simpson *et al.* (1974), Ross Wilson (1959, 1970 and 1973) and Philip (1989). There are a few smaller books describing individual distilleries in Scotland. Recommended are those by McDowell (1975) and Wilson (1973; 1975). Recommended reading on Irish whiskey includes an account by McGuire (1973) and short review articles by Court and Bowers (1970) and Lyons (1974).

## History of whisky production

The origins of the art of distilling potable spirits are obscure, and probably date back to ancient China. However, the first treatise on distilling was written by the French chemist Arnold de Villeneuve around 1310. The potency of distilled spirits caused many to be known as the 'water of life', a description that survives today in such names as *eau de vie* for French brandy and *akvavit* and *aquavit* for spirits in Northern Europe. The name 'whisky' is a corruption of

‘uisgebaugh’, the Gaelic word for water of life. Uisge was corrupted first into ‘usky’, which finally became whisky after several centuries. Dr. Johnson sang the praises of this potable spirit, although in his Dictionary of 1755 it is listed under ‘u’ and not ‘w’.

Much to the chagrin of the Scotsman, it is likely that the first whisky was distilled not in Scotland but in Ireland. The spirit was known in Ireland when that land was invaded by the English in 1170. In all likelihood the art of distillation was imported into Scotland by missionary monks from Ireland. Two of today’s main centers of Scotch distilling, namely the island of Islay and the Speyside town of Dufftown, were the sites of early monastic communities.

Whisky, principally Scotch whisky, has for many years been one of the most popular distilled beverages in the world; and it was in Scotland rather than in Ireland that its qualities came to be extensively appreciated. This has continued to the present day and in the intervening period many Scotsmen have felt compelled to record for posterity their thoughts and inspirations on the potable spirit. There are numerous histories of whisky distilling in Scotland, some more comprehensive than others. For good general accounts, the reader is referred to the texts by Brander (1974), Daiches (1969), Ross (1970) and Ross Wilson (1970).

Whisky distilling flourished in Scotland not least because consuming the spirit helped the inhabitants to withstand the climatic rigors of this northern region of Britain. The first recorded evidence of whisky production in Scotland is an entry in the Exchequer Rolls for the year 1494. It reads “To Friar John Cor, by order of the King, to make aquavita, eight bolls of malt”. Production of whisky was therefore being controlled; and an Act of 1597 decreed that only earls, lords, barons and gentlemen could distill for their own use. To many Scots of this era whisky was a medicine, and in 1506 King James IV of Scotland had granted a monopoly for manufacture of ‘aqua vitae’ to the Guild of Barber Surgeons in the City of Edinburgh.

Taxation on whisky production first appeared in the 17th century. Breaches of the monopoly regulations and the need to raise money to send an army into England to help the English Parliament in its war against Charles I led to the Act of 1644, which fixed a duty of two shillings and eight pence Scot’s on a pint of whisky (the Scot’s pint was then about 1.5 litres). But the tax was short-lived and was replaced by a malt tax that later was also repealed.

At the time of the Treaty of Union between Scotland and England in 1707 there was a tax on malt in England, but not in Scotland. The English were irate; and in 1725 when Lord Walpole’s administration decided to enforce the tax in Scotland, the first of a series of Malt Tax riots occurred. The English, meanwhile, had cultivated a taste for French brandy, there being very little whisky consumed at that time outside of Scotland. However around 1690 William III began to wage commercial war against the French, and imposed punitive taxes on imports of French brandy into England. The English reacted by acquiring a taste for gin, which was distilled locally. The scale of drunkenness that developed with the popularity of gin had to be controlled by law; and the Acts of 1736 and 1713 levied high taxes on gin manufacturers. Both of these acts contained clauses exempting Scotland, but not for long. The Parliament in London saw the prospect of a rich harvest of taxes in the distilleries of Scotland; and in a series of acts starting in 1751, production of whisky in Scotland was increasingly subjected to taxation.

The outcome of these punitive measures was not surprising. An extensive and thriving business in illicit distillation of whisky grew up in Scotland as described by Sillett (1965). Curiously, illicit production of Scotch hardly extended over the border into England, although there are a few records of the operation of illicit stills in the Cheviot Hills west of Newcastle-upon-Tyne. Following the Act of 1823, which introduced much stiffer penalties for illicit distillation, and to some extent because of the increased standards of living in northern Scotland, illicit manufacture of whisky declined. Indeed, many erstwhile illicit

distillers emerged to become legal and registered distillers of Scotch whisky.

In 1826, Robert Stein of the Kilbagie distillery in Clackmannanshire, Scotland, patented a continuously operating still for whisky production. However, this invention was superseded in 1830 with the introduction by Aeneas Coffey of an improved version of this type of still. The appearance of continuous stills sparked off a period of turmoil in the Scotch whisky industry, it being claimed that the product from the continuous distillation of a mash that contained unmalted grain (described as neutral or 'silent spirit') could not be called whisky, since it had not been distilled in the traditional pot still. The battle was waged for about three quarters of a century; and in 1908 a Royal Commission decided that malt whisky and grain neutral spirit, when blended, could be labeled whisky.

The major factors which have affected the development of the whisky distilling industry in Scotland in this century have been economic. The industry has had to endure the privations of two world wars, the economic depression in

Great Britain during the 1920s and prohibition in the United States from 1920 to 1933, which greatly affected export of Scotch to North America. Since 1945, however, the industry in Scotland has consolidated and expanded. The 20th century has also witnessed a considerable improvement in the quality of whisky distilled and blended in Scotland as a result of the acceptance of blending malt whiskies with grain whisky and the amalgamation of several smaller distilleries into combines.

Scotch malt whiskies can be divided into 'highland', 'lowland', 'Islay' and 'Campbeltown whiskies' (Simpson *et al.*, 1974). The 'highland line', which separates the areas in Scotland in which the first two types of spirit are distilled, is a straight line which runs from Dundee in the east to Greenock in the west (Figure 1). It then extends southwards, below the Isle of Arran. Any whisky produced north of this line, including those from Campbeltown and Islay, is entitled to be called a highland malt whisky, while whiskies distilled in areas south of the line are designated as lowland whiskies. Of the 104 malt whisky



**Figure 1.** Highland and lowland whisky-producing regions of Scotland.

distilleries in Scotland, 95 are highland malt whisky distilleries. Of these, no fewer than 49 are situated in an area measuring 50 miles east to west and 20 miles southwards from the Moray Firth. This area of Speyside has been called the 'Kingdom of Malt Whisky' (Cameron Taylor, 1970). Classification of the four whiskies distilled on the islands of Jura, Orkney and Skye is disputed. Some authorities list them along with the Islay whiskies as 'island' whiskies, others as highland whiskies, which is geographically correct. There are also eight grain whisky distilleries in Scotland.

Whisky distilling in Ireland was, as has been noted, first recorded in the 12th century. By 1556, it had become sufficiently widespread to warrant legislation to control it. A statute proclaimed that year stated that a license was required to manufacture the spirit, but that peers, gentlemen owning property worth £10 or more and borough freemen were exempt (McGuire, 1973). Taxation of whisky distilling gradually became more excessive and collection of taxes became increasingly efficient. However, in 1779 there was an important change in the distillery laws. An attempt was made to limit the extent of evasion of spirit duty by prescribing a minimum revenue to be exacted from the owner of each still. The effect of this legislation was dramatic. In 1779, there was said to be 1,152 registered stills in Ireland. By 1790, this number had fallen to 216 and this inevitably fostered widespread illicit distilling (McGuire, 1973). This legislation lasted until 1823 when it was replaced by laws that taxed Irish whiskey on the volume of production, legislation that is essentially still in force today. Development of the Irish whiskey distilling industry in the present century has inevitably been influenced by economic circumstances and by the political division of Ireland into the Republic and Northern Ireland that occurred in 1922. Barnard (1887) described visits to 28 distilleries in Ireland, but closures and amalgamations followed such that when McGuire (1973) prepared his account, there were only two whiskey-distilling companies in Ireland, one with distilleries in Dublin and Cork in the Republic and the other with plants in Bushmills and Coleraine

in Northern Ireland. These two companies have since amalgamated and have concentrated their distillery operations in Cork and Bushmills. There has also been a move towards production of a lighter Scotch-type whisky in Ireland to replace the heavier traditional Irish whiskey.

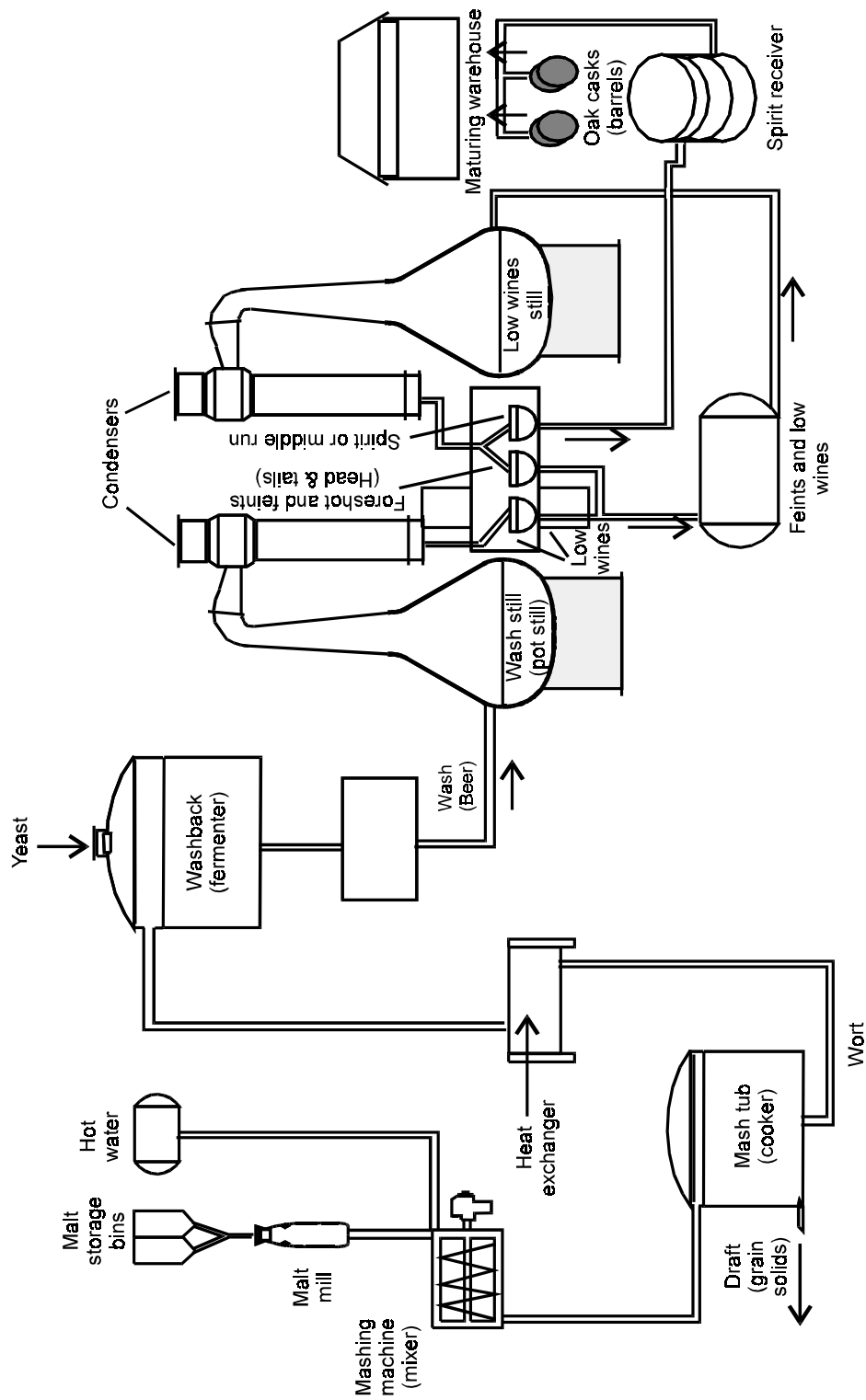
## **Outline of whisky production processes**

Whiskies differ basically in the nature and proportion of the cereals used as raw materials and on the type of still used in the distillation process. These differences in the production process are illustrated in the flow diagram in Figure 2 for production of Scotch malt whisky (production of Irish whiskey is very similar). Detailed accounts of each of the unit processes in whisky production are given in subsequent sections of this chapter.

A characteristic of Scotch malt whisky is that the only cereal used in its manufacture is malted barley (Table 1). After milling, the meal is mashed in a mash tun (Figure 2) similar to that used in breweries for beer production. During mashing or conversion, enzymes in the malt catalyze the hydrolysis of starch into fermentable sugars. In the manufacture of Scotch grain whisky and Irish whiskey, other cereals are used along with malted barley to provide additional starch in the mash tun (Table 1). Owing to the high gelatinization temperature of their starches, unmalted cereals must be precooked before they are incorporated into the mash.

The wort, or clear mash, leaving the mash tun is cooled and fed into a vessel where it is mixed with yeast. In Scotland and Ireland these fermentation vessels have a relatively small capacity and are known as 'washbacks' (Figure 2).

Fermentation is conducted with strains of the yeast *Saccharomyces cerevisiae* that are usually specially propagated for the purpose, although Scotch malt whisky distillers may use some surplus brewers yeast (Table 1). The process is allowed to proceed to a point at which the



**Figure 2.** Flow diagram showing the principal operations during production of Scotch malt whisky.

**Table 1.** Raw materials and unit processes used in the production of scotch and Irish whiskies.

|               | <i>Scotch malt</i>   | <i>Scotch grain</i>  | <i>Irish</i>  |
|---------------|--|--|---|
| Raw materials | Peated and unpeated malted barley  | Wheat or corn and a small proportion of malted barley  | Unmalted barley and unpeated, malted barley   |
| Conversion    | Infusion mash  | Mash cook followed by conversion stand   | Infusion mash   |
| Fermentation  | Distillers yeast and brewers yeast   | Distillers yeast   | Distillers yeast  |
| Distillation  | Two pot stills   | Continuous still   | Three pot stills  |
| Maturation    | At 62° GL in used charred oak bourbon whiskey barrels or sherry casks for at least three years | Up to 67° GL in used charred oak bourbon whisky barrels or sherry casks for at least three years | At 70° GL in used charred oak bourbon whisky barrels or sherry casks for at least three years |

specific gravity of the fermented mash has usually dropped to below 1.000. In pot still distilleries, the fermented mash or 'wash' (beer) is fed directly to a still known as the wash still, from which the distillates are redistilled in the second or low wines still. In Ireland, and in one Scotch malt whisky distillery, a third distillation is carried out. Finally, the freshly distilled whisky is stored in charred oak barrels for minimum periods of time that depend on the legislation in the producing country (Table 1). Scotch malt and Irish whiskies are customarily matured for much longer than the legal minimum period.

## Individual operations

### Raw materials

#### *Malted cereals*

Malted barley is the principal malted cereal used in whisky production. Like the brewer, the whisky distiller uses barley cultivars of the species *Hordeum vulgare* L. and *Hordeum distichon* (Hough *et al.*, 1971). Malted barley is employed as a source of enzymes (principally amylolytic) that catalyze the hydrolysis of starches and in

some instances serves as a source of starch that is converted ultimately into ethanol. These two demands must be finely balanced. In the manufacture of Scotch malt whisky, where only malted barley is used, care must be taken when the grain is sprouting during the malting process to ensure that only a limited amount of enzyme activity is produced. This is because enzyme is produced at the expense of the fermentable materials in the grain (referred to as 'extract'). However in the manufacture of other types of whisky, malted barley is often used as the only source of amylolytic enzyme in a mash bill that contains a high proportion of unmalted grain. In this type of whisky production the enzyme activity of the malted barley must be greater than that used in Scotch malt whisky manufacture.

Traditionally, barley used in the production of Scotch malt whisky was malted on the distillery premises using a floor malting system and dried over fires of coke and peat in the pagoda-shaped kilns which are still a feature of these distilleries. To a large extent, this system has been superseded by mechanical maltings, which produce malt for groups of distilleries. In order not to destroy the enzyme activity developed during malting, a balance must be achieved in the kiln between drying the green malt to a

suitably low moisture level for storage, curing to give it the appropriate flavor and retaining sufficient enzyme activity (Simpson, 1968). In maltings attached to the distillery, the kiln temperature is increased slowly over a 48 hr period to achieve an even rate of drying and the desired flavor. The latter character is achieved by fuelling the furnace with peat during the early part of the kilning period when the green malt is moist and readily absorbs the peat smoke or 'reek'. In mechanical maltings the green malt is dried at a faster rate with a forced-air draught, but a supplementary peat-fired kiln is often used to produce flavored malts. The amount of peat used varies with different maltings. Some of the distilleries on Islay in Scotland specialize in producing a whisky with a very pronounced peat flavor, and they therefore use heavily-peated malts. Malted barleys used in the manufacture of Scotch grain and Irish whiskies are not dried over a peat fire. They generally have a greater enzyme activity, so the relatively small proportion of malted barley used in the mash contains sufficient enzyme activity to convert all of the starch in the mash (principally supplied by unmalted cereals) into fermentable sugars. The greater enzyme activity in these malted barleys is reflected in their nitrogen content. Malts used in production of Scotch grain whisky have a nitrogen content of 1.8% or higher (compared with a brewer's malted barley with a nitrogen content in the region of 1.5%) (Hough *et al.*, 1971). Malting aids, such as gibberellic acid and bromate, are not normally used in production of malted barley for whisky manufacture.

Because of the high cost of malted cereals, considerable effort has been expended by the whisky distiller in attempting to devise methods that allow prediction of the yield of alcohol expected using different proportions of malted barley in the mash bill. Unfortunately, the methods customarily used by the brewer, such as those recommended in Great Britain by the Institute of Brewing Analysis Committee (1975), have proved of limited value. The brewer has used measurements of diastatic power, expressed as the Lintner value, which is a measure of the extent of saccharification of soluble starch

present in a cold water extract of the malt (Lloyd Hind, 1948) as an indicator of malt quality. Diastatic activity measured in this way includes contributions from both  $\alpha$ - and  $\beta$ -amylases. However, Preece and his colleague (Preece, 1947; 1948; Preece and Shadaksharaswamy, 1949 a,b,c) have shown that high  $\beta$ -amylase activity, as determined by the Lintner value, is not always accompanied by high  $\alpha$ -amylase activity. Pyke (1965) showed that the Lintner value of a malt is only useful for predicting the spirit yield in manufacture of Scotch grain whisky when the proportion of malted barley in the mashbill is low (Figure 3). Determination of  $\alpha$ -amylase activities of the malt gave a less satisfactory correlation than the Lintner value, an observation which agrees with that made earlier by Thorne *et al.* (1945). Further evidence for the unsuitability of employing traditional malt specifications for predicting performance in whisky manufacture has come from Griffin (1972).

Although the degree to which a malted barley has been peated can to some extent be assessed by smell, such is the importance of this character in malt that it must be determined in a more rigorous fashion. Peat smoke or 'reek' contains a wide range of compounds, but it is generally held that the peaty character is imparted to the malt largely as a result of absorption of phenols. For some years, Scotch distillers used a method based on a reaction of phenols with diazotized sulphanilic acid. A lack of specificity in this method, coupled with the instability of the diazonium salt, prompted MacFarlane (1968) to recommend an alternative method involving extraction of phenols from malt with diethylether under acid conditions with absorptiometric measurement of the color developed when the phenols are reacted with 4-aminophenazone. MacFarlane applied the method to a wide variety of malts, both peated and unpeated, and reported values ranging from zero (for an unpeated grain) to as high as 9.4 ppm for a malt produced on Islay in Scotland. (It has been calculated that to obtain a malt with 10 ppm phenols, one tonne of peat must be used for drying each tonne of malted barley).

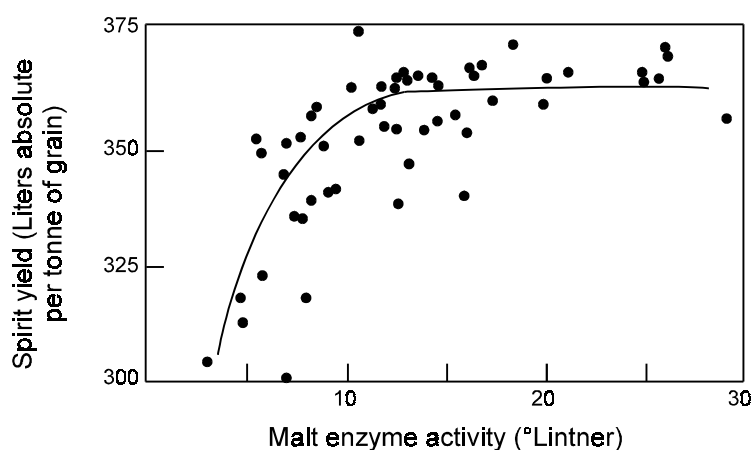


Scotch whisky distillers have been concerned by the possibility that colorimetric methods, such as those recommended by MacFarlane (1968) and Kleber and Hurns (1972), may not assay all of the organoleptically-important compounds that malt acquires as a result of peating. To examine this possibility, MacFarlane *et al.* (1973) produced peat smoke condensate on a laboratory scale and separated the oil and aqueous phases from the wax fraction, as only the two former would contain components that might appear in the distilled whisky. Six compounds, namely furfural, 5-methylfurfural, guaiacol, phenol, *p*-cresol and 5-xilenol, were detected in the aqueous fraction by gas liquid chromatography (GLC). The peat smoke oil was more complex, and no fewer than 30 peaks, some of them created by more than one compound, were obtained by GLC. The compounds included hydrocarbons, furfural derivatives, benzene derivatives and phenols. The authors stressed that using their GLC techniques, 3,5-xilenol is masked by the peaks of *m*-ethylphenol and *p*-ethylphenol, two compounds thought to make an important contribution to peat aroma and taste. Figure 4 shows gas liquid chromatograms of extracts of a peated and a unpeated malt.

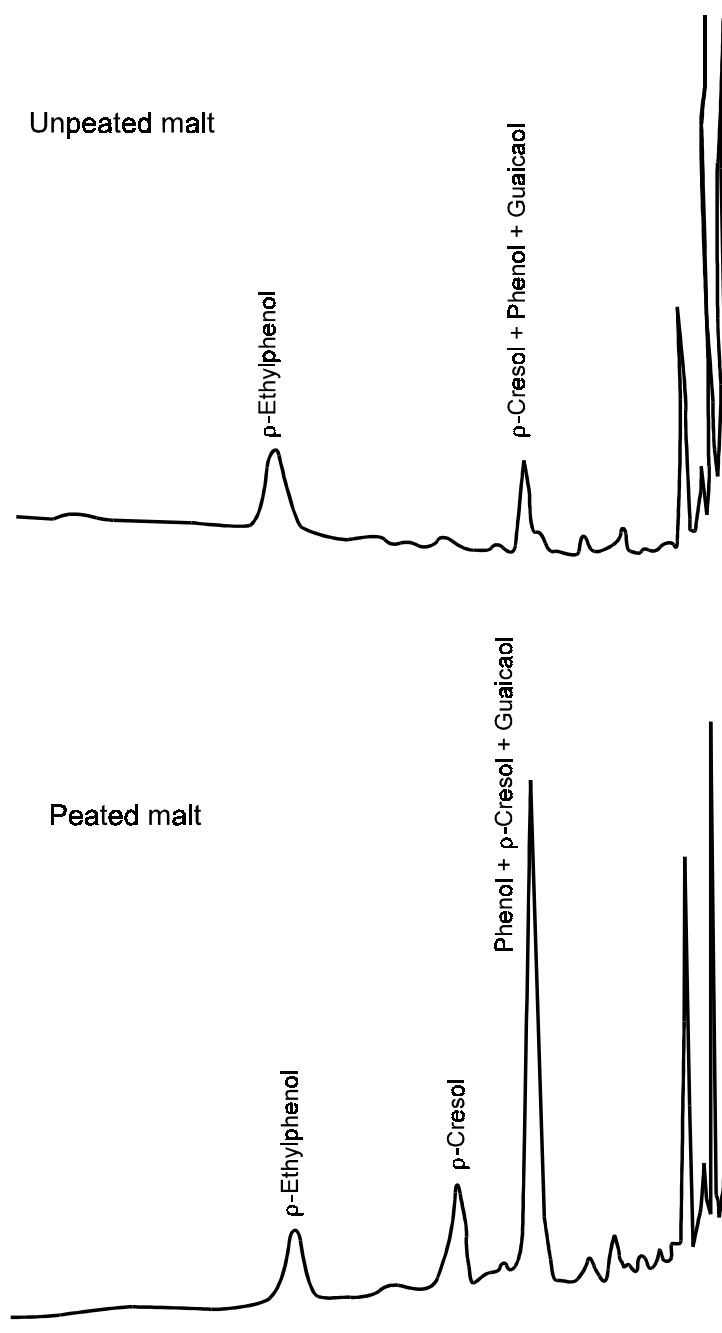
### Unmalted cereals

Fewer problems are encountered in arriving at specifications for the unmalted cereals used in whisky manufacture, namely wheat, corn, rye and barley. The corn (varieties of *Zea mays*) used in mash bills for manufacturing Scotch grain whiskies is usually of the yellow dent type, generally obtained from France. Occasionally white corn is used, and it is reputed to give a higher alcohol yield. Corn is a popular grain because it has a high content of starch that is readily gelatinized and converted into fermentable sugars. The US has imposed controls on the quality of corn used for whisky manufacture. In the US there are three grades of corn, with only grades 1 and 2 being approved for spirit manufacture. In Great Britain, on the other hand, the corn used is normally grade 3 on the US scale.

Unmalted barley used in manufacture of Irish whiskey has a quality intermediate to that used for malting and that used for cattle feed. In this way, the maltster can select the best barley available on the market at the time of purchase. For many years, a small percentage (about 5% of the total) of unmalted oats (*Avena spp.*) was



**Figure 3.** Relationship between the diastatic activity of a Scotch grain whisky mash bill and spirit yield. Laboratory fermentations were conducted using mash bills containing different proportions of malt mixed with corn, and therefore with different Lintner values. It can be seen that only when the proportion of malt is rate-limiting can spirit yield be correlated with the Lintner value (Pyke, 1965).



**Figure 4.** Gas-liquid chromatograms of extracts of unpeated and peated Scotch barley malts showing the contribution peating makes to the content of phenols in the malt. Peating leads to an increase in the size of the peak corresponding to phenol, *p*-cresol and guaiacol, and of the peaks which lie to the right of the mixed phenol peak (attributable to furfurals and hydrocarbons). *p*-cresol is also detectable on the chromatogram of extracts of peated malt. The ratio of the area of the total phenol peaks to that of *p*-ethylphenol is used as an indication of the peatiness of the malt.

included in mash bills for manufacturing Irish whiskey. It was contended that these grains, with their large husks, improved the texture of the grain bed in the mash tun (to assist in straining off the clear wort from the mash solids) and that oats influenced the flavor of Irish whiskey. Whether either or both of these effects were important will probably never be known, for oats are no longer used in the production of Irish whiskey (Court and Bowers, 1970).

## **Mashing and cooking**

Mash production in Scotch and Irish distilleries involves a process not unlike that used in breweries to prepare wort for beer manufacture. However, where cereals other than malted barley are used, the malt mashing is preceded by a high temperature cooking process.

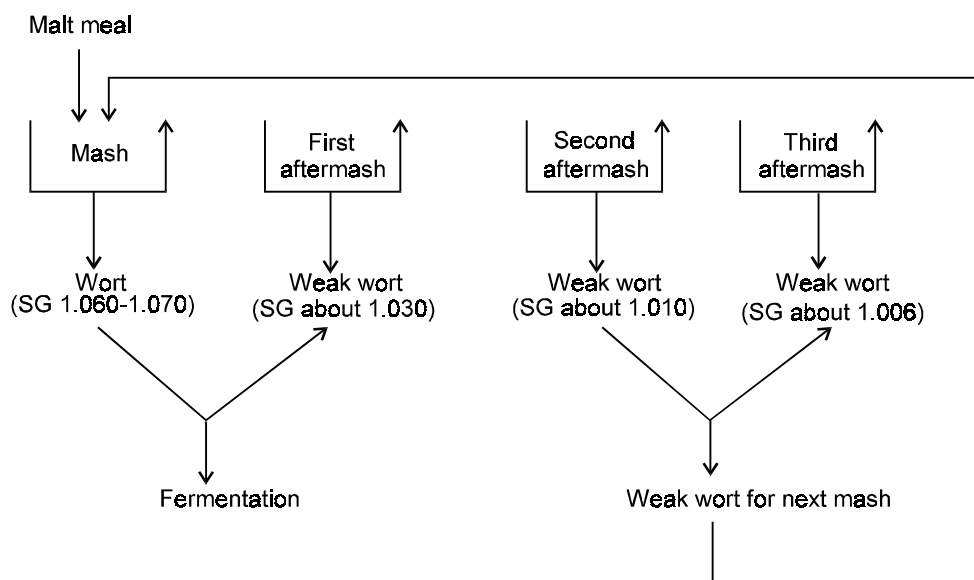
### ***Mashing***

Regardless of whether the mash bill contains cereals other than malted barley, the main biochemical changes that take place during mashing are hydrolysis of starch, protein and other biopolymers in the meal to produce water soluble low molecular weight compounds that form a fermentable substrate (or wort). The major starch-liquefying and saccharifying enzymes are  $\alpha$ - and  $\beta$ -amylases, while the limit dextrins formed by action of amylases on amylopectin are further hydrolyzed by limit dextrinases.

Barley malt used in the manufacture of Scotch malt whisky is coarsely ground in a roller mill adjusted to give a grind no finer than the malt warrants. Too fine a grind can give rise to a 'set mash' that settles on the bottom of the mash tun to block and impede drainage of the liquid (Simpson, 1968). The mash tun is usually preheated with water and the perforated bottom plates are flooded before mashing commences. The meal from overhead bins is mixed with hot water at 60-65°C in the proportion of one part meal to four parts water, and the mash is

homogenized by action of revolving rakes. In the traditional mashing process, the mash is usually loaded into the tun to a depth of about one meter and allowed to stand for about one hour, after which the wort is drained off from under the grain bed. This liquid extract, which has a specific gravity (SG) of 1.070-1.060 (Figure 5) is collected in an intermediate vessel known as an 'underback'. After being cooled to around 25°C in a heat exchanger, the wort is pumped into the fermentation vessel. The bed of grains in the tun is then re-suspended in water at 75°C and a second batch of wort is drawn off at a specific gravity of around 1.030 and passed into the underback. This process is known as the first aftermash and is repeated twice more; except that the dilute worts drawn off are not passed to the underback, but are returned to the hot water tank to be used in the next mash. The wort in the underback has a pH value of about 5.5, a specific gravity of 1.045-1.065, and an amino nitrogen content of about 150-180 mg/L. The spent grain residue or 'draff' is removed from the mash tun and sold as animal feed. Many distilleries have now reduced this process to just three water additions. Larger distilleries are now installing semi-lauter tuns in place of the traditional mash tuns. These have vertical knives, enabling the structure of the mash bed to be maintained whilst speeding wort draining, and sparging rings to allow simultaneous draining and sparging. The result is a faster more efficient extraction without loss of clarity or changing the composition of the wort.

Mash preparation in the manufacture of Irish whiskey is very similar to that used in Scotch production, but there are certain differences. Use of a high percentage of raw barley in the mash bill (up to 60%) has necessitated the use of stone mills or hammer mills to achieve the required grind. The unmalted barley is sprayed with water to give a moisture content of about 14% and then dried to around 4.5% moisture before grinding. The malted barley used is roller-milled. In recent years a plant has been installed in Ireland which uses a wet milling process that eliminates the need for watering and drying of



**Figure 5.** Flow diagram showing the mashing cycle in a Scotch malt whisky distillery.

the unmalted barley. Mash tuns used in Irish whiskey distilleries differ from those used to make Scotch in that they are larger, simply because these distilleries have stills with a greater capacity than those in Scotland. (This difference dates back to when there was a flat rate of tax per still in Ireland, rather than a tax per gallon of product.) Moreover, the mashing cycle differs in that the weak wort from the first aftermash is not passed to the underback, but is mixed with worts from the second and third aftermashes to be used in the subsequent mash (Lyons, 1974).

### ***Cooking followed by malt mashing***

The wheat, corn and rye used in production of Scotch grain whisky must be cooked before being added to the mash containing malted barley in order that the starch in these grains can become gelatinized and accessible to the malt enzymes. Traditionally, cooking was carried out as a batch process, but it has to some extent been superseded by continuous processes.

For batch cooking, the grain is freed from extraneous material by passage through screens and then ground either in a pin-type mill or a

hammer mill. It is then conveyed to the cooker and subjected to a cooking cycle involving high temperatures and pressures designed to bring about complete gelatinization of the starch. Inadequate cooking will sometimes leave starch granules intact in the mash, while excessive heating can cause caramelization and therefore loss of sugar and decreased spirit yield. Pyke (1965) has provided an account of the cooking of corn in its production of Scotch grain whisky and a similar process is still used today. The conventional cooker is a horizontal cylindrical vessel capable of working at pressures up to 90 psi ( $63 \times 10^4$  Pa), and fitted with stirring gear. A typical cycle in cooking corn might consist of 1-1.5 hrs of heating with live steam injection to reach a temperature of  $120^\circ\text{C}$  and a pressure of  $15 \times 10^4$  Pa. The mash is held at this temperature and pressure for a further 1.5 hrs. The liquid used for the mash is often that from the third aftermash mentioned earlier. At the end of the cooking time, the pressure is released and the hot cooked corn mash is blown directly into a saccharification vessel containing the malted barley suspended in hot water. Cold water is then added to bring the temperature in the mash tun to around  $63^\circ\text{C}$ . Good mixing is also essential at this stage; and

failure to achieve it can lead to the entire mash solidifying. The need for rapid cooling of the corn mash was emphasized by Murtagh (1970), who showed that with slow cooling or holding at a temperature around 82°C lipid-carbohydrate complexes could be formed that are not fermentable and can lead to a loss of 1-2% in spirit yield.

After a suitable mashing period, the mash may be filtered as described previously. More commonly, the entire mash may be pumped via a heat exchanger into a fermenter. Pyke (1965) has published the composition of a typical Scotch grain whisky mash (Table 2).

**Table 2.** Chemical composition of a typical scotch grain mash\*.

|  | %     |
|--|-------|
| Total soluble carbohydrate (as glucose)  | 9.00  |
| Insoluble solids                         | 2.20  |
| Fructose                                 | 0.13  |
| Glucose                                  | 0.29  |
| Sucrose                                  | 0.28  |
| Maltose                                  | 4.65  |
| Maltotriose                              | 0.96  |
| Maltotetraose                            | 0.45  |
| Dextrin                                  | 2.54  |
| Amino nitrogen (as leucine)              | 0.09  |
| Ash                                      | 0.27  |
| containing P <sub>2</sub> O <sub>5</sub> | 0.09  |
| K <sub>2</sub> O                         | 0.09  |
| M <sub>g</sub> O                         | 0.02  |
|  | µg/ml |
| Thiamin                                  | 0.46  |
| Pyridoxine                               | 0.61  |
| Biotin                                   | 0.01  |
| Inositol                                 | 236   |
| Niacin                                   | 11.1  |
| Pantothenate                             | 0.71  |

\*Pyke, 1965.

In the manufacture of Scotch grain whisky, the proportion of malted barley in the mash bill is usually around 10-15%, a proportion far greater than that required merely to provide a source of amylolytic enzymes. It is a practice of long

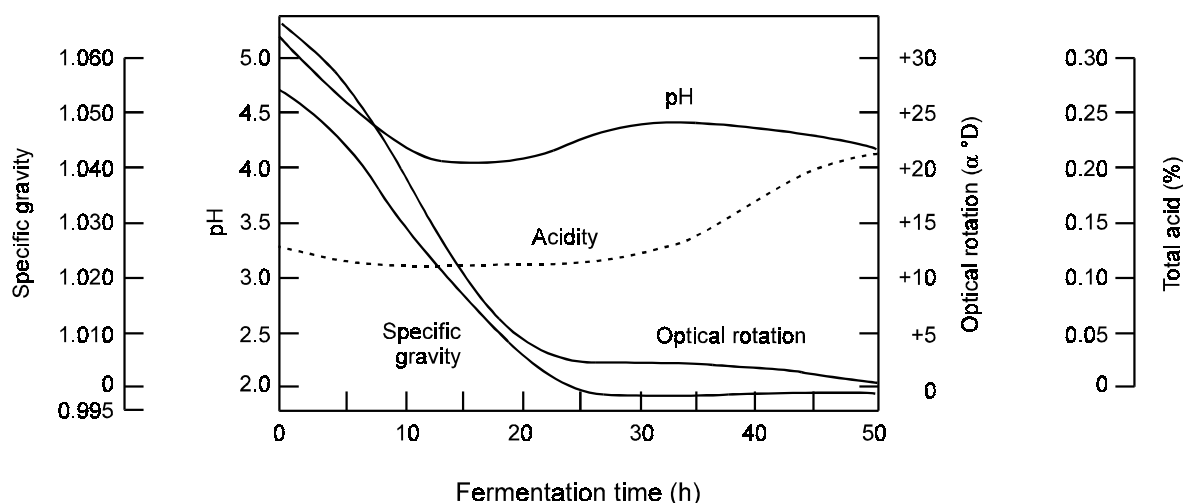
standing, and is done to obtain the required malt flavor in the grain whisky. This was noted as early as 1902 by Schidrowitz and prompted further comment from Valaer in 1940.

Continuous cooking has been adopted in recent years in Scotland. Stark (1954) listed the advantages of using continuous cooking. While practices vary to some extent in different distilleries, essentially the cereal is slurried at around 50°C and then pumped through a steam-jet heater into the cooking tubes where the residence time is normally 5-10 minutes. The continuous cooker has a narrow tube (6-16 cm in diameter) which reduces the incidence of charring and carbon deposition. The mash passes through the tube at about 20 meters/minute, with the temperature reaching near 65°C and the pressure 65 psi (40 x 10<sup>4</sup> Pa).

### Fermentation

The objectives in fermenting a whisky distillery mash with strains of *Saccharomyces cerevisiae* are to convert the mash sugars to ethanol and carbon dioxide while producing quantitatively minor amounts of other organic compounds which contribute to the organoleptic qualities of the final distilled product. Fermenting vessels vary considerably in volume, depending on the distillery. The small Scotch whisky distillery at Edradour near Pitlochry, for example, has fermenters with a capacity of only 4,500 liters. In contrast, other Scotch and Irish pot whiskey distillers have fermenters with a capacity in the range 50,000 to 150,000 liters. Much larger fermenters are found in Scotch grain whisky distilleries. Traditionally, the smaller pot distillery fermenters were made of wood, usually of larch or Oregon pine, but in recent years they have been constructed of steel or aluminum. In some Scottish distilleries, timber is still used as a covering material.

When the fermenter is partly filled, the mash is inoculated with a suspension of *Saccharomyces cerevisiae*. The source of yeast varies with the location and size of the distillery. Distilleries in



**Figure 6.** Changes in specific gravity, optical rotation, pH value and acidity during fermentation of a mash in the production of Scotch malt whisky (Dolan, 1976).

Scotland and Ireland, particularly the pot whisky distilleries, rarely have their own yeast propagation equipment. They rely on specially-propagated pressed yeast for use in fermentations. In Scotch malt whisky distilleries, this pressed yeast is augmented, usually on an equal weight basis, with surplus brewery yeast typically supplied in compressed form from breweries that may be as far as 500 km from the distillery.

The requirements for a strain of *Saccharomyces cerevisiae* yeast used in distillery practice have been described by Harrison and Graham (1970). Apart from the obvious need to select a strain that maintains very high viability in the pressed state (containing 25% dry weight), other very important properties of these strains are ability to tolerate concentrations of ethanol on the order of 12-15% (v/v) and the capacity to metabolize oligosaccharides such as maltotriose and maltotetraose in order to maximize the conversion of starch into ethanol and carbon dioxide.

Whisky worts usually have a specific gravity in the range 1.050-1.080, a pH value of around 5.0, a total acid content of 0.1% and an optical rotation of +30°. After inoculation, the yeast

content is 5-20 million cells/ml. The bacterial count varies with the cleanliness of the plant and the extent to which the raw materials were endowed with a microbial flora. Scotch grain whisky fermentations create little if any foam because of their large content of suspended solids. However, in most Scotch malt whisky fermentations only a small proportion of the suspended solids in the mash is retained in the fermentation vessel. These fermentations tend to foam and the distillers have resorted to the use of various types of antifoams.

Changes over the time course of a typical fermentation in a Scotch malt whisky distillery are depicted in Figure 6. Fermentation proceeds vigorously for the first 30 hrs, during which time the specific gravity falls to 1.000 or below and the optical rotation to around zero. The sugars in the wort are utilized in a particular sequence with glucose and fructose being fermented first, followed by maltose and then maltotriose. The removal of sugars during fermentation of a Scotch grain whisky mash is shown in Figure 7 (Pyke, 1965). Over the first 30 hrs the pH value, after declining to around 4.2, rises to about 4.5. During the first 30 hrs the specific gravity drops at a rate of about 0.5° per hour accompanied by

a massive evolution of heat. While many of the larger grain whisky distilleries have fermenters fitted with cooling coils, these are absent, or if fitted are relatively inefficient in most malt whisky distilleries where temperature can rise by the end of the fermentation to as high as 35-37°C. The distiller is concerned about the temperature rise during fermentation since this can cause the fermentation to stop or become 'stuck'. Temperature rise can be controlled by using a lower starting temperature or, because glycolysis of sugar is a heat-producing process, by using a lower initial concentration. Strains of *Saccharomyces cerevisiae* are well suited for malt whisky distillery fermentations since they can ferment efficiently over a wide temperature range. Fermentation is usually continued for at least 36 hrs and frequently longer, at which time the ethanol content of the wash is 7-11% (v/v). In larger distilleries, particularly those in the US, the carbon dioxide evolved is collected, liquefied and sold. Smaller distilleries, particularly the malt whisky distilleries in Scotland, usually do not have this facility.

It should be noted that mashes in malt whisky distilleries are not boiled, so any enzyme activity

manifested at the temperature of the mash and any microorganisms that can survive at that temperature will continue to be active during the fermentation. The continued activity of limit dextrinases in unboiled distillery mashes increases the concentration of sugars available for fermentation by the yeast. Hopkins and Wiener (1955) calculated that with amylases alone the yeast cannot metabolize the equivalent of the final 12-16% of the starch.

Another important consequence of using non-sterile conditions in distillery fermentations is the activity of bacteria that pass through in the mash, which are encouraged to some extent by the relatively high temperatures to which the fermentations can rise. In addition to lactic acid bacteria, the flora can include other Gram-positive as well as Gram-negative strains. The concentration of the flora depends on a number of factors including the extent to which the lactic acid bacteria grew during yeast propagation, the extent of the flora on the cereal raw materials and on the standard of hygiene in the distillery. There is no doubt, however, that the controlled activity of this bacterial flora, and particularly of the lactic acid bacteria, is accompanied by

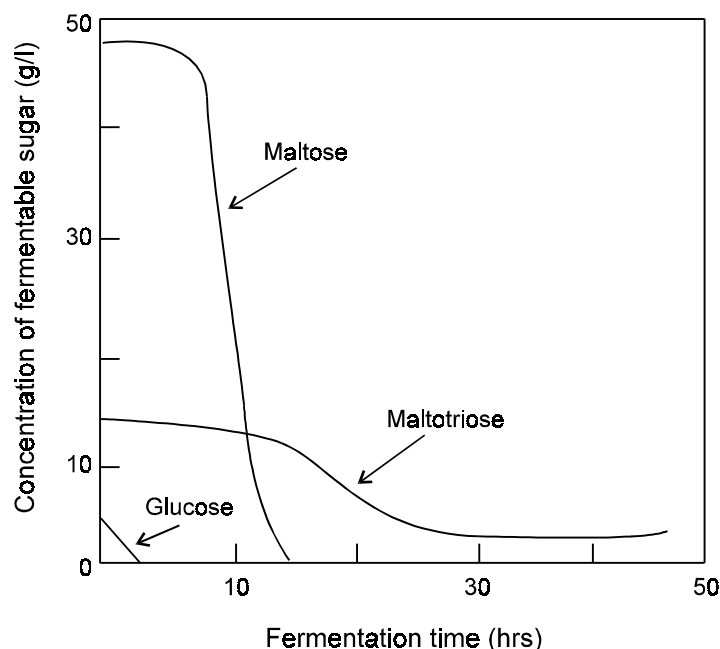


Figure 7. Time course removal of fermentable sugars from a Scotch grain whisky mash (Pyke, 1965).

excretion of compounds that contribute to the organoleptic qualities of the final whisky (Geddes and Riffkin, 1989).

During the first 30 hrs or so of malt whisky fermentation there is vigorous fermentation and the majority of the aerobic bacteria die. This, however, provides ideal conditions for growth of anaerobic or microaerophilic bacteria, principally lactic acid bacteria (mainly strains of *Lactobacillus brevis*, *L. fermenti* and *Streptococcus lactis*) with the result that the concentration of lactic acid in the fermented mash can be as high as 30 mg/L (MacKenzie and Kenny, 1965). A wide range of lactobacillus species have been identified in Scotch whisky fermentations including *L. fermentum*, *L. brevis*, *L. delbrueckii*, *L. plantarum*, *L. casei* and a bacterium resembling *L. collinoides*, in addition to *Leuconostoc spp.*, *Streptococcus lactis* and *Pediococcus cerevisiae* (Bryan-Jones, 1976). More recently Barbour (1983) isolated many species that did not conform to recognized species of lactic acid bacteria, a point emphasized by Walker *et al.* (1990) who used DNA hybridization techniques to classify distillery bacteria. Growth of lactic acid bacteria is probably enhanced by yeast excretion of nitrogenous nutrients at the end of a vigorous fermentation. Kulka (1953) demonstrated the ideal nature of yeast autolysate for growth of lactobacilli.

Bacterial activity in the fermenting wort also leads to removal of some acids. Actively growing yeasts secrete citric and malic acids, but MacKenzie and Kenny (1965) attribute the lower concentrations of these acids in malt distillery worts (as compared to brewery worts) to their partial removal by bacteria.

Occasionally, the extent of the bacterial flora in the fermenting wort can become too large. This causes problems due to sugar utilization by the bacteria that lead to an overall decrease in spirit yield. In addition, the bacteria may produce organoleptically-undesirable compounds and also release hydrogen ions causing the pH value of the wort to fall too low, thereby providing suboptimal conditions for action of certain enzymes. Examples of undesirable compounds that may be excreted by bacteria are hydrogen sulfide and other sulfur-containing compounds (Anderson *et al.*, 1972). Lactobacilli can also metabolize glycerol (excreted by the yeast during fermentation) to produce  $\beta$ -hydroxypropionaldehyde, which subsequently breaks down on distillation to give acrolein (Harrison and Graham, 1970). Acrolein imparts a pungent, burnt and often peppery odor to the whisky (Lyons, 1974). In a later paper, Dolan (1976) concentrated on the problems arising in malt whisky distilleries when there is an unacceptably high concentration of bacteria in

**Table 3.** Changes in the concentration of bacteria during fermentation of a minimally infected and a heavily infected Scotch malt whisky wort\*

| Age<br>(hours) | Minimally-infected wort |                     | Bacteria/ml        |                    | Heavily-infected wort |                    |
|----------------|-------------------------|---------------------|--------------------|--------------------|-----------------------|--------------------|
|                | Gram-negative rods      | Gram-negative cocci | Lactobacilli       | Gram-positive rods | Gram-positive cocci   | Lactobacilli       |
| At setting     | 2,000                   | 3,700               | n.d.               | 52,000             | 60                    | $0.6 \times 10^6$  |
| 10             | 150                     | n.d.                | n.d.               | n.d.               | n.d.                  | $2.3 \times 10^6$  |
| 20             | n.d.**                  | n.d.                | $1.53 \times 10^6$ | n.d.               | n.d.                  | $18.8 \times 10^6$ |
| 30             | n.d.                    | n.d.                | $10.2 \times 10^6$ | n.d.               | n.d.                  | $96 \times 10^6$   |
| 40             | n.d.                    | n.d.                | $10.2 \times 10^6$ | n.d.               | n.d.                  | $502 \times 10^6$  |
| 50             | n.d.                    | n.d.                | $50 \times 10^6$   | n.d.               | n.d.                  | $1 \times 10^9$    |

\*Dolan, 1976.

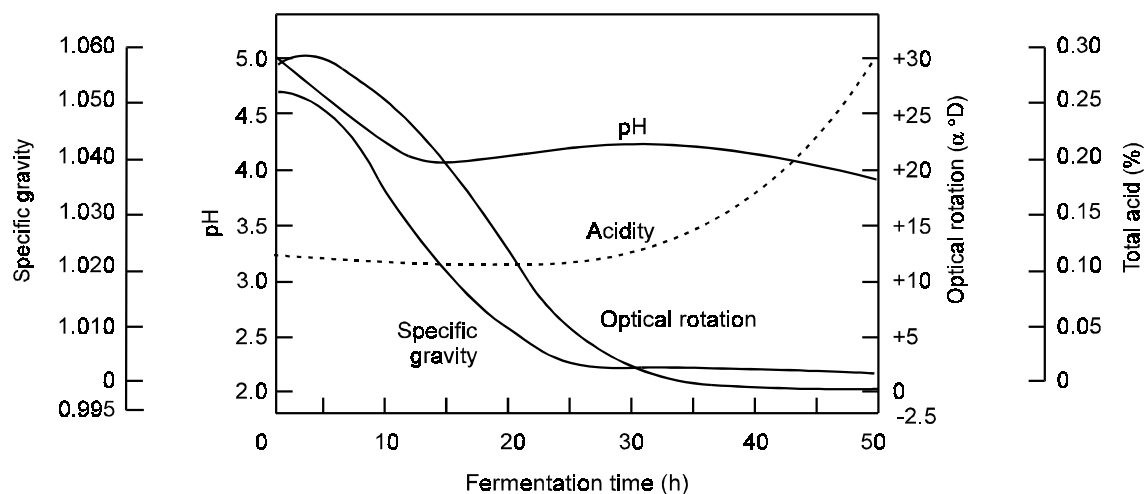
\*\*None detected.



the mash. Table 3 shows changes in the concentrations of Gram-negative and Gram-positive bacteria and (separately) of lactobacilli during fermentation of a minimally-infected mash and of a heavily-infected mash. The time course of fermentation of an unacceptably-infected malt distillery mash (Figure 8) shows, in comparison with similar data for fermentation of an acceptable mash (Figure 6), a greater rise in the acid content of the mash after about 35 hrs and a lower optical rotation of the mash after about 40 hrs. In the fermentations there is often a difference of up to 4 hrs from the time a rise in the acid content is detected to the point when the pH value of the fermentation begins to fall. Dolan (1976) attributes this to the buffering capacity of the mash. The data in Table 4 show the effect of different levels of infection after 30 hrs fermentation of a malt distillery mash on spirit yield and the associated financial losses to the distiller. Dolan (1976) recommends upper limits of 1,500 bacteria, 50 Gram-positive and 10 lactic acid-producing bacteria per million yeast cells in the mash at the start of fermentation.

Much less has been published on the effect of retaining solid material in the fermenting mash. However, marine microbiologists have long

known that the presence of solid particles in a liquid medium can affect bacterial growth, probably because of the concentration of nutrients at the solid-liquid interface (Heukelekian and Heller, 1940; Zobell, 1943). Moreover, Cromwell and Guymon (1963) found that formation of higher alcohols during fermentation of grape juice is stimulated by the presence of grape skins or inert solids. Beech (1972) made similar observations on cider fermentations. Merritt (1967), in the only detailed report on the role of solids in whisky distillery fermentations, states that a dry solid concentration of 50 mg/100 ml might typically be expected, although much will clearly depend on the design of the mash tuns used in individual distilleries. Merritt went on to report that a concentration of dry solids as low as 5 mg/100 ml causes an increase in yeast growth, and that solids also enhance the rate of production of ethanol and glycerol. There was also an effect on production of higher alcohols by the yeast (Table 5). With the possible exception of n-propanol, production of all of the major higher alcohols was increased in the presence of solids, the effect being particularly noticeable with isobutanol and 2-methylbutanol.



**Figure 8.** Changes in specific gravity, optical rotation, pH value and acidity during fermentation of a Scotch malt whisky mash containing an unacceptably high concentration of bacterial infection (Dolan, 1976).

**Table 4.** Effect of the level of bacterial infection on the loss of spirit incurred following fermentation of a scotch malt whisky mash.\*

| <i>Infection rating</i> | <i>Bacteria/ml in 30 hr old mash (million)</i> | <i>Approximate loss in spirit yield (%)</i> | <i>Approximate financial loss in US dollars (thousands)</i> |             |
|-------------------------|--|---|---|-------------|
|                         |  |   | At filling  | Duty paid   |
| a                       | 0 - 1  | <1  | <26   | <369        |
| b                       | 1 - 10   | 1-3   | 26 - 79   | 369 - 1106  |
| c                       | 10 - 100                                       | 3-5   | 79 - 131  | 1106 - 1844 |
| d                       | >100   | >5  | >131  | >1844       |

\*Dolan, 1976.

The effect of low insoluble solids content is a factor relevant to congener levels in malt whisky fermentations. In grain whisky production, where 'all-grains-in' fermentations are generally used, the degree of rectification during distillation is the principle determinant of higher alcohol levels in the spirit.

## Distillation

Whether the fermented beer is distilled in a pot still, as in production of Scotch malt and Irish whiskies, or in a continuous still based on the Coffey design as in the manufacture of Scotch grain whiskies, the objectives are the same: selective removal of the volatile compounds (particularly the flavor-producing congeners) from the non-volatile compounds and to create additional flavor-producing compounds as a result of chemical reactions that take place in the

still. Nevertheless, it is still most convenient to discuss whisky distillation under the separate headings of pot still and continuous distillation.

## Pot still distillation

The copper pot still, which is the feature dominating any Scotch malt or Irish whiskey distillery, has changed hardly at all over the centuries, except of course in size. Traditionally, the onion-shaped stills were fired from beneath and had the vapor pipe or 'lyne arm' from the still projecting through the distillery wall to connect with a condenser in the form of a coil immersed in a water tank fed from a local stream (Figure 9). Internal steam-heated calandria are now preferred to direct firing because this decreases the extent of pyrolysis of the still contents and results, for example, in a lower concentration of furfural in the whisky. Variations

**Table 5.** Production of higher alcohols (mg/100 ml).

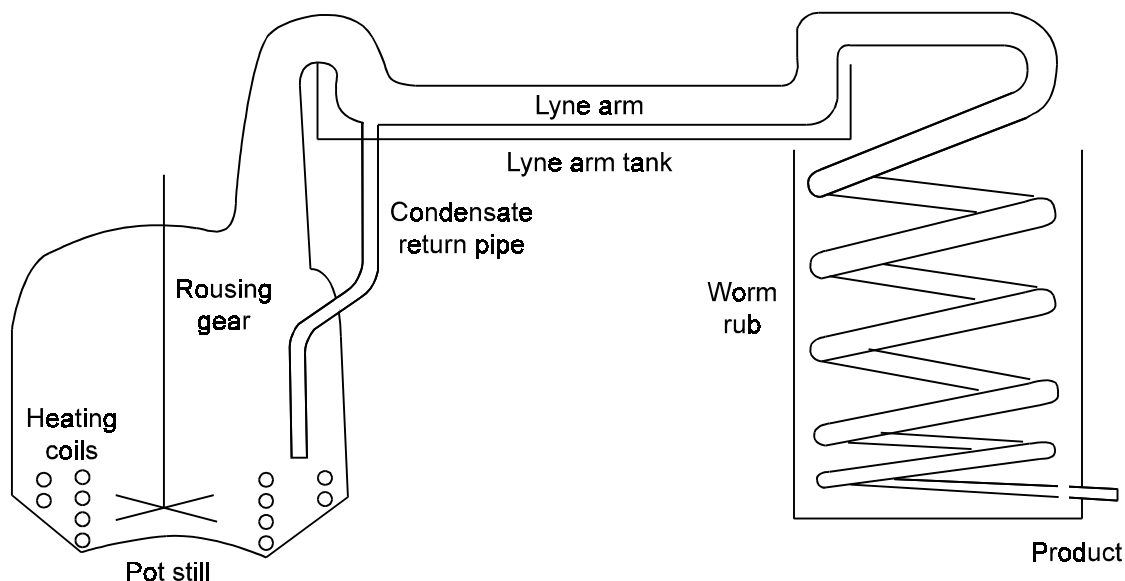
| <i>Mash</i> | <i>Insoluble solids content (mg/100 ml)</i> | <i>n-Propanol</i> | <i>Isobutanol</i> | <i>2-methyl butanol (amyl alcohol)</i> | <i>3-Methyl butanol (isoamyl alcohol)</i> | <i>Total</i> |
|-------------|---|-------------------|-------------------|--|---|--------------|
| 1           | 0   | 1.5               | 4.2               | 3.0                                    | 8.0                                       | 16.7         |
|             | 25  | 1.5               | 6.5               | 4.4                                    | 8.8                                       | 21.2         |
| 2           | 0   | 2.8               | 4.5               | 2.7                                    | 9.5                                       | 19.5         |
|             | 35  | 3.3               | 6.6               | 3.7                                    | 10.1                                      | 23.7         |
| 3           | 0   | 1.7               | 4.8               | 3.1                                    | 8.0                                       | 17.6         |
|             | 50  | 1.7               | 6.8               | 4.6                                    | 9.5                                       | 22.6         |

in still design include expansion of the surface area of the column into a bulbous shape, water jacketing and return loops from the first stage of the condensation. (Nettleton (1913) provided a valuable account of early still design). In many distilleries, condenser coils have been replaced by tubular condensers that have an advantage in that they are designed to conserve the heat extracted from the distillates. Yet other pot stills are fitted with 'purifiers,' which consist of a circular vessel cooled by running water interposed between the neck of the still and the condenser. In Irish pot stills this purifier function is effected by a trough fitted around the lyne arm through which running water is circulated. Pot stills in Scotch and Irish distilleries are traditionally constructed of copper. The reason for this adherence to copper is more than tradition. It has been established that copper fixes some volatile sulfur-containing compounds that are produced during fermentation but undesirable in the distilled spirit.

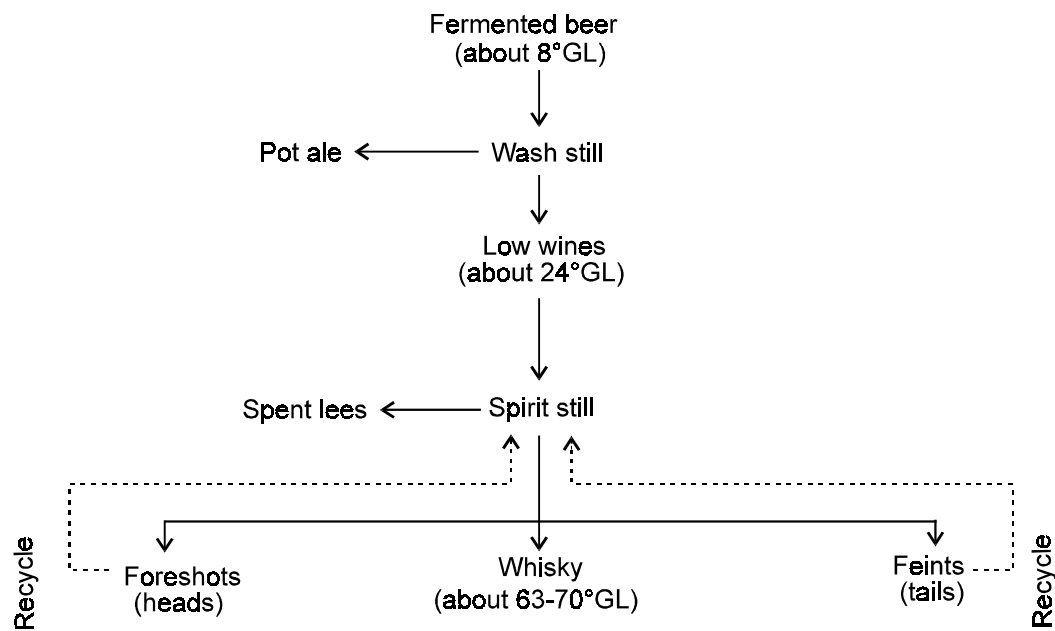
Early whisky distillers realized that although the objective of distilling was to separate volatile constituents from the beer, collecting the distillate not as a whole but in several fractions

and combining certain of these fractions gave a much more acceptable product. Pot still distillation in Scotland and Ireland differ not only in the size of the stills (25,000-50,000 liters in Scotland vs 100,000-150,000 liters in Ireland), but also in the different ways in which fractions are collected from the stills.

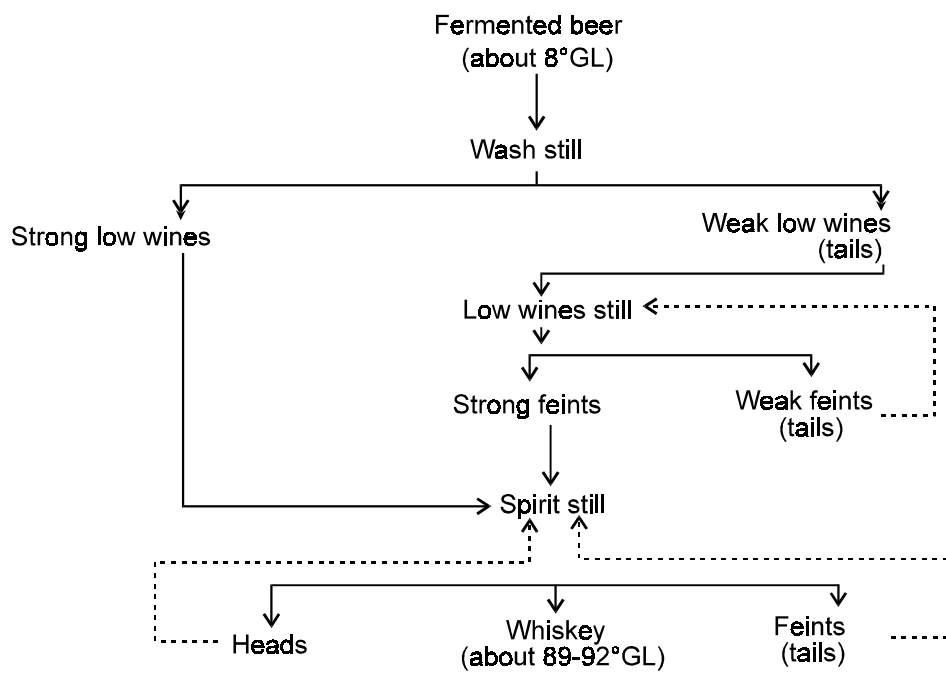
In Scotland, the beer is subjected to two distillations. In the first, carried out in the beer still, the beer is brought to a boil over a period of 5-6 hrs and the distillate is referred to as low wines. This distillation effects a three-fold concentration of the alcohol in the beer (Figure 10). The residue in the wash still, known as 'pot ale', is either discharged to waste or evaporated to produce an animal feed (Rae, 1967). Distillation of the low wines in the spirit still is still more selective. The first fraction, which contains low boiling point compounds, is rejected as 'fore-shot heads'. At a stage determined by continued hydrometric monitoring, which usually occurs when the distillate has an alcohol content of approximately 70-73° GL, the distillate is switched from the fore shots tank to the whisky receiver tank. This switch has traditionally been made at the discretion of the distiller; and he has been



**Figure 9.** Diagram of an Irish distillery pot still. Designs used in Scotch malt whisky distilleries are similar except that the pot is onion-shaped and the still usually has a shorter lyne arm not surrounded by a lyne-arm tank (Lyons, 1974).



**Figure 10.** Flow diagram showing the stages in distillation of Scotch malt whisky.



**Figure 11.** Flow diagram showing the stages in distillation of Irish whiskey.

aided by the disappearance of a bluish tinge when water is added to the distillate. The monitoring process takes place in a spirit safe secured with a Government Excise lock. Collection of the distillate is terminated when the alcohol content has fallen to a specified value, although distillation of the feints or tails is continued until all of the alcohol has been removed from the low wines. The residue remaining in the spirit still is known as 'spent lees', and like pot ale is either run to waste or evaporated to manufacture animal feed. The whisky distilled over in the middle fraction has an alcohol content of 63-70° GL.

The manufacture of Irish whiskey involves three rather than two distillations (Figure 11). The fermented beer is heated in the wash still, and the first distillation ('strong low wines') is collected until the distillate reaches a predetermined specific gravity. The distillate, then known as 'weak low wines', is switched into a separate vessel. The weak low wines are pumped into the low wines still and are re-distilled to produce two fractions termed 'strong feints' and 'weak feints'. Strong feints are mixed with the strong low wines in a spirit still. Distillates from this are collected in the same fashion as in production of Scotch. The whiskey collected usually is about 89-92° GL.

### ***Continuous distillation***

No fundamental changes have been introduced into the design of the patent or Coffey still over the past century. Automation, particularly of the beer feed, is now commonplace, as is continuous monitoring of other stages in the distillation process. Nevertheless, many Scotch and Irish producers of grain whiskies continue to use a still which, like the original Coffey still, has just two columns: a beer stripper (or analyzer) and a rectifier.

A description of the operation of two column continuous stills in the manufacture of Scotch grain whisky has come from Pyke (1965). In order to obtain whisky of high quality from these stills, they must be operated such that the alcohol

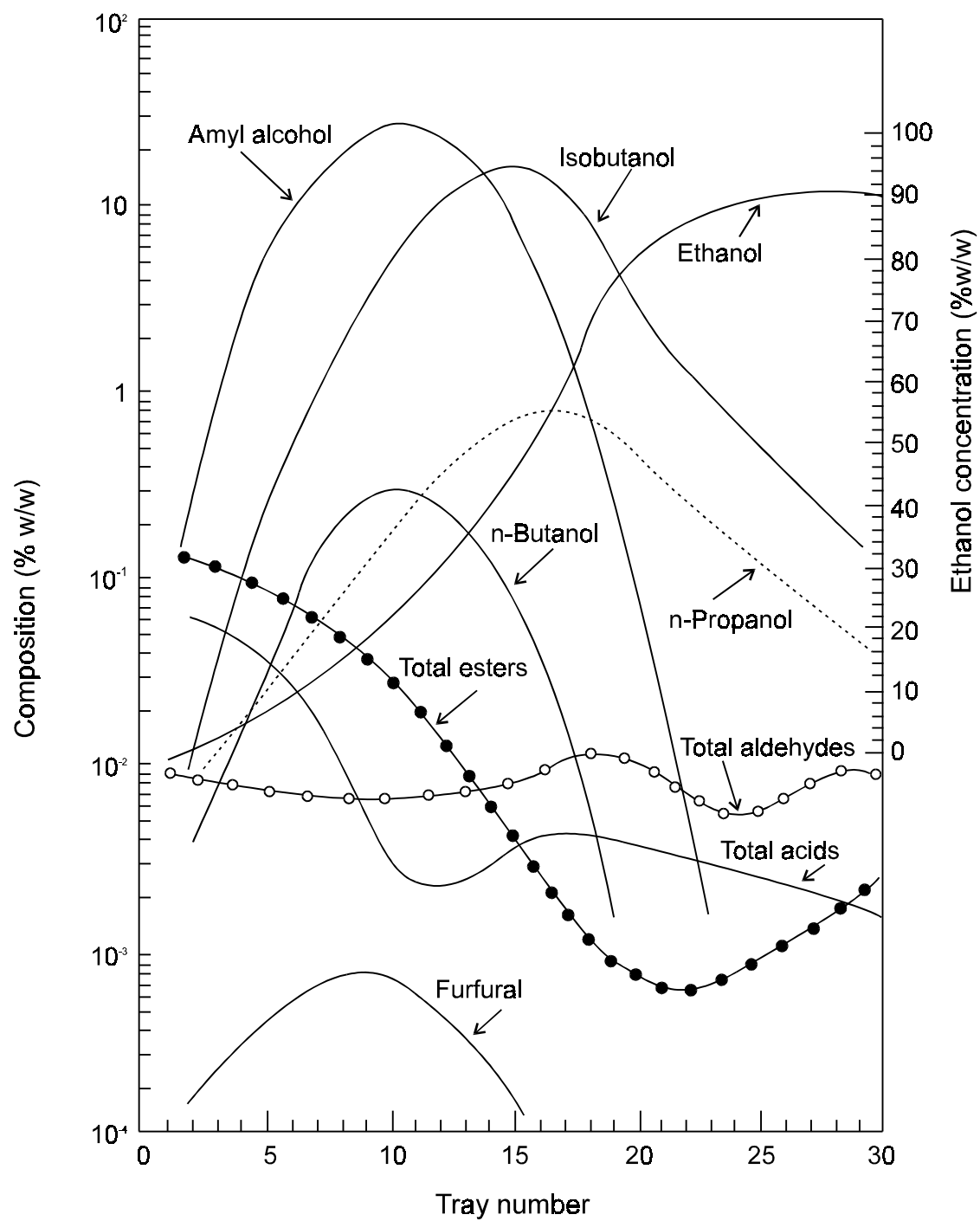
concentration of the spirit at the spirit draw tray in the rectifier is not less than 94.17° GL. The manner in which the precise control of still operation can affect the composition of the whisky is shown in Figure 12. As illustrated, if conditions are changed in either direction on the abscissa, the concentration of congeners will alter with a possible adverse effect on final product quality.

### **Maturation and aging**

Freshly distilled whisky of any type is very different from the spirit that is later bottled, either singly or blended. The transformation is brought about by storing the whisky in oak barrels for periods of time that depend on traditional practice and legal requirements. In general, whiskies are matured for far longer than the legally-required period of time. The raw spirit is taken by pipeline from the distillation plant to the tank house where it is diluted with water to the required strength and then transferred into barrels.

Maturation in barrels is accompanied by a loss of liquid by evaporation, and the relative rates of loss of water and of alcohol determine whether the aged whisky has a higher or lower alcoholic strength than that at filling. In Scotland, where the barrels of whisky are stored in cool, unheated, but humid warehouses, the alcoholic strength decreases (Valaer, 1940). In contrast Valaer and Frazier (1936) reported that in the US storage conditions cause an increase in alcoholic strength. Maturation in barrels is also accompanied by changes in the chemical composition of the whisky. These changes are attributable to extraction of wood constituents from the barrel, oxidation of components present in the original whisky as well as those extracted from the wood, reactions between components in the whisky and removal and oxidation of highly volatile sulfur components by the carbon char on the inner surface of the barrel.

Some of the earlier investigators reported on changes in the composition of the major classes



**Figure 12.** Changes in composition of the vapor at different trays in a Coffey still rectifier used in the manufacture of Scotch grain whisky (Pyke, 1965).

of organoleptically-important compounds during maturation in barrels. Thus, Schidrowitz and Kaye (1905) found increased concentrations of volatile acids in aged whiskies, a trend also described in the report of the Royal Commission on Whisky and Other Potable Spirits (1909). Several reports followed. Liebmann and Scherl (1949), for example, reported increased concentrations of acids, furfural, tannins and color with maturation in barrels. The arrival of the gas liquid chromatograph and HPLC greatly accelerated research on this topic; and more recent data on chemical changes that take place during maturation are described later in this chapter.

Maturation of whisky in oak barrels is an expensive process; and it is hardly surprising that consideration has been given to methods for acceleration. Jacobs (1947) described several of these methods including pretreatment of the beer with activated carbon, chemical treatment of the whisky to convert aldehydes into esters and use of oxidation treatments. Such techniques are not used in the industry, which adheres to the traditional nature of the whisky-producing process.

## **Blending and coloring**

Whisky manufacturers take great pride in the quality of their straight or blended whiskies and in their ability to maintain the quality of a particular product over the years. Blending is conducted by experts who advise manufacturers on mixing or blending large volumes of whisky after appropriate sniffing and tasting sessions on experimental blends. Blended whiskies are filled into re-used barrels after dilution with water (or not) and stored for a further period of time referred to as 'marrying'. The water used for dilution is softened or demineralized, since water containing an appreciable concentration of salts can cause hazes in the whisky (Warricker, 1960). After marrying, and dilution if necessary, the color of the whisky may be adjusted to the desired value by adding caramel. Some brownish

pigment is extracted from the casks, but this may not be sufficient to provide the desired color. Finally, the whisky is clarified for bottling by filtration through sheets of cellulose (Simpson, 1968). Chill filtration may also be practiced, since it removes tannin material from the whisky and prevents subsequent appearance of haze.

## **Effluent disposal and spent grains recovery**

Traditionally effluents from whisky distilleries were disposed of in the most convenient manner. Spent grains were retailed, often quite cheaply, to local farmers as animal fodder while pot ale and spent lees were simply discharged into the local sewer, stream or river. This is no longer the case, mainly because of the distiller's awareness of the nutritional value (largely the protein content) of some of these effluents, and public awareness of environmental problems arising from uncontrolled disposal of effluents into waterways. Simpson has described the production of 'distillers dark grains'. These processes are widely used to dispose of effluents from whisky distilleries, especially where the traditional methods of disposal are forbidden or uneconomic.

## **Organoleptically important components of whisky**

Modern analytical techniques have enabled major advances in the understanding of the compounds responsible for the organoleptic properties of whiskies. However, the first reports on the nature of flavor-producing compounds in whiskies antedate the era of gas liquid chromatography by nearly half a century. Two publications by Schidrowitz (1902) and Schidrowitz and Kaye (1905), dealing exclusively with Scotch whiskies, reported on the higher alcohol, acid and ester contents of some 50 different brands. They reported analyses of several Campbeltown Scotch malt whiskies. A

report by Mann (1911) published a few years later also quoted values for acidity and levels of furfural, aldehydes, esters and alcohols in Scotch whiskies imported into Australia.

### ***Concentrations of organoleptically important compounds***

Since the introduction of GLC into distillery laboratories, several reviews have been published on the composition of the major flavor-producing compounds in whiskies (namely higher alcohols, esters, carbonyl compounds, organic acids, aromatic compounds) and on the identification of individual compounds that make up these fractions. Higher alcohols, which are still routinely determined with GLC using a polar stationary phase (Aylott *et al.*, 1994), are quantitatively the most important. Scotch malt whiskies are richest in higher alcohols, with contents often well over 2 g/L. Free fatty acids are relatively volatile and make a major contribution to the organoleptic qualities of whiskies. Concentrations of acids in some Scotch malt whiskies can be as high as 0.4-1.0 g/L absolute alcohol (Duncan and Philip, 1966). Roughly comparable concentrations of esters are found in whiskies, although those produced with pot stills generally have higher concentrations than those from continuous stills. Ester concentrations in Scotch and Irish pot still whiskies have been reported in the range of 0.27-0.87 g/L absolute alcohol (Valaer, 1940). Lighter whiskies contain lower concentrations of carbonyl compounds, although the concentration varies with the brand. Grain whisky may have as little as 20 mg/L of aldehydes, while in a mature Scotch malt whisky the concentration may be as high as 80 mg/L (Duncan and Philip, 1966).

### ***Chemical nature of organoleptically-important compounds***

Duncan and Philip (1966) reviewed chromatographic and other methods used to separate

the various organoleptically important compounds from whiskies. Even though analytical methods such as capillary column gas chromatography linked to a mass selective detector (GC-MS) (Aylott *et al.*, 1994) have developed in terms of sensitivity and discrimination, a major problem in analyzing whisky by GLC is the overwhelming preponderance of ethanol and water. Only one volatile compound, namely isoamyl alcohol, is likely to be present in a concentration exceeding 0.01%; while most of the others are present in concentrations that rarely exceed 50 ppm. Indeed many compounds now understood to have an important impact on whisky flavor are present at ppb levels. Carter-Tijmstra (1986) described a technique for measuring dimethyl trisulfide, a compound with a threshold of only 0.1 ppb present in whisky at concentrations below 50 ppb. Analyses are most conveniently conducted on extracted fractions of the different classes of compounds. When direct analysis of whiskies has been employed, only a limited number of components have been determined (Morrison, 1962; Bober and Haddaway, 1963; Singer and Stiles, 1965).

Recent developments in headspace analysis using trapping and thermal desorption techniques (Pert and Woolfendon, 1986) have enabled analysis of the more volatile components of whisky flavor whilst avoiding interference from high concentrations of other congeners. Headspace analysis using trap and purge techniques would now be the method of choice for measuring highly volatile sulfur compounds such as dimethyl trisulfide.

Some idea of the variety of compounds detected in whiskies came from a compilation of both published and unpublished sources (Kahn 1969, Kahn *et al.*, 1969). Of the some 200 compounds listed there are 25 higher alcohols, 32 acids, 69 esters and 22 phenolic compounds. Undoubtedly, this list could now be extended quite considerably. Of the higher alcohols, isoamyl alcohol and optically-active amyl alcohol predominate, accompanied by lower concentrations of isobutanol and n-propanol. Characteristically, there are usually only low



concentrations of n-butanol and secbutanol. The principal organic acid in whiskies is acetic acid, which can account for between 50 and 95% of the total content of volatile acids determined by titration. Of the remaining acids, caprylic, capric and lauric are quantitatively the most important (Suomalainen and Nykänen 1970a). Some of the characteristic flavor and aroma of Irish whiskies may be attributed to somewhat higher concentrations of the odoriferous butyric acid. Compared with other types of whisky, Scotch whisky characteristically contains more palmitoleic acid (and its ethyl ester) than palmitic acid. Suomalainen (1971) has suggested that the typical stearin-like smell of Scotch malt whisky may be attributed to long chain fatty acid ethyl esters. It is not surprising to find that ethyl acetate is the major whisky ester in view of the prevalence of acetic acid in the distillate. Concentrations of 95 mg/L have been detected in a blended Scotch whisky (de Becze, 1967). Other esters such as ethyl caprate are present in much lower concentrations, on the order of 2-10 mg/L. Of the carbonyl compounds, acetaldehyde is the principal component together with a range of other short chain aldehydes. Furfural, with an aroma resembling that of grain, also occurs with as much as 20-30 mg/L in Scotch malt whiskies (Valaer, 1940). Acrolein, a pungent and lachrymatory compound, is also present; and it has been suggested that it may contribute to the 'peppery' smell of whisky.

A variety of other organoleptically important compounds has also been detected in different whiskies, many of which result from maturing the whisky in charred oak barrels. Scopoletin and other aromatic aldehydes, including vanillin, were detected in bourbon by Baldwin and his colleagues (1967). These compounds were previously identified by Black *et al.* (1953) in ethanolic extracts of plain and charred American white oak from which the bourbon whiskey barrels subsequently used for the maturation of Scotch and Irish whiskies are constructed. (It should be noted that the US regulations for bourbon whiskey production require that new

charred oak barrels be used for maturation; so there is a continuous supply of once-used bourbon barrels that are shipped to Ireland and Scotland for re-use.) A lactone dubbed 'whisky lactone', also appears in whisky following storage in oak barrels. This compound,  $\beta$ -methyl-octalactone, was first isolated from Scotch whisky by Suomalainen and Nykänen (1970b). It has since been reported that both *cis* and *trans* diastereomers of the compound occur in whisky (Nishimura and Masuda, 1971). Other compounds detected in whisky include phenols (Salo *et al.*, 1976), glycerol and erthritol (Black and Andreasen, 1974), pyridine,  $\alpha$ -picoline and various pyrazines (Wobben *et al.*, 1971).

### ***Contribution of compounds to organoleptic properties***

Published information on compounds responsible for the organoleptic qualities of whiskies is meagre and very largely confined to reports by Suomalainen and his colleagues from the State Alcohol Monopoly in Helsinki, Finland. In order to assess the contributions made by whisky components to the odor of these spirits, Salo *et al.* (1972) concocted a synthetic whisky with components that chromatographic analysis had revealed were present in a light-flavored Scotch whisky. The synthetic whisky was made using 576 g of a mixture of higher alcohols, 90 mg of acids, 129 mg esters and 17.4 mg carbonyl compounds in highly-rectified grain spirit diluted to 34° GL in water that had been ion-exchanged and treated with activated charcoal. This imitation whisky contained 13 alcohols in addition to ethanol, 21 acids, 24 esters and 9 carbonyl compounds. Caramel coloring was used to give it the color of a distilled and matured whisky. Odor thresholds of the individual compounds and groups of compounds were determined as described by Salo (1970). Experienced taste panel participants were easily able to distinguish the imitation whisky from a blended Scotch whisky; but when the concoction was mixed with an equal amount of the Scotch, only 6% correct

judgments above chance were made. This suggested that the concentrations of, and interactions between, components of the synthetic whisky were not greatly dissimilar from that in the Scotch used for comparison.

Odor threshold determinations of individual components in the imitation whisky revealed that the contributions made by the mixture of alcohols and acids accounted for only about 10% of the total odor intensity, despite the fact that the alcohols themselves accounted for over 70% of the total concentration of organoleptically-important compounds in the concoction. Esters and carbonyl compounds had a much greater influence, particularly butyraldehyde, isobutyraldehyde, isovaleraldehyde, diacetyl, and the ethyl esters of acetic, caproic, caprylic, capric and lauric acids. Since just three of the most important carbonyl compounds could substitute for the whole carbonyl fraction, there would seem to be considerable homogeneity in the odor contributions made by these compounds. Interestingly, the relative contributions made by the different classes of compounds are not very different from the contributions Harrison (1970) reported that they make towards the taste of beers.

Threshold values can be assessed not only for individual components in whisky, but for the total aroma of the beverage. Salo (1975) examined this by diluting different whiskies with water until the characteristic whisky aroma could only just be recognized. Values for the threshold dilution of several commercial whiskies shown

in Table 6 reflect the differences in aroma strength for several different commercial whiskies.

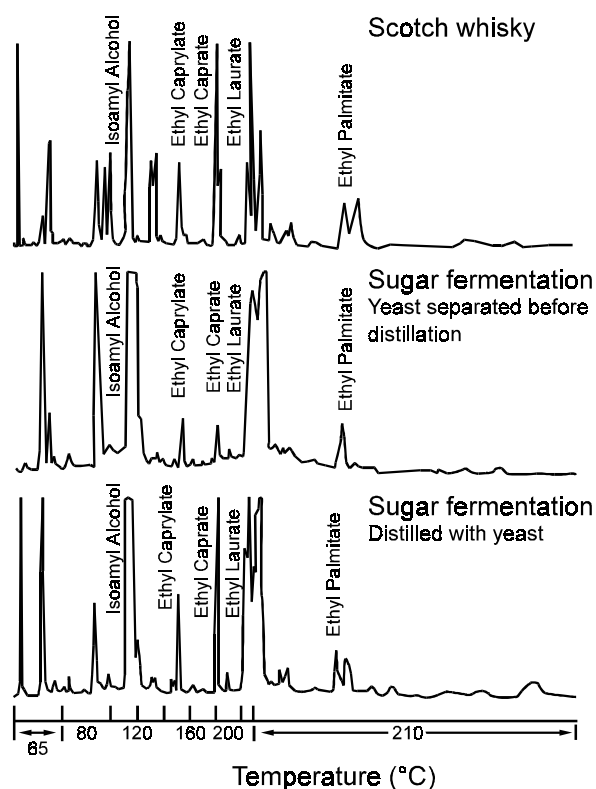
### ***Origin of organoleptically-important compounds***

The two main sources of the organoleptically-important compounds in whisky are the yeast used to ferment the wort and the charred oak barrels in which the whisky is matured. Suomalainen and Nykänen (1966) fermented a nitrogen-free medium containing sucrose with a strain of *Saccharomyces cerevisiae* and distilled the fermented medium either after removing the yeast by centrifugation or with the yeast remaining in the medium. Gas chromatographic analyses of these distillates are reproduced in Figure 13, which also shows a chromatogram of Scotch whisky for comparison. There is clearly a similarity among the three analyses; although differences, such as the higher proportion of isoamyl alcohol in the distillate from the fermented medium, can be detected. Also worth noting is the greater concentration of ethyl caprate in the distillate from the spent medium containing yeast as compared with that obtained by distilling the medium from which yeast had been removed. It would be interesting to learn of the importance of yeast strain in production of organoleptically-important compounds in whisky. Unfortunately, there is a lack of published data on this matter.

**Table 6.** Threshold dilution levels for nine different types of whisky\*.

| <i>Whisky</i>     | <i>Threshold dilution (x 10<sup>-4</sup>)</i> | <i>One standard deviation range (x 10<sup>-4</sup>)</i> |
|-------------------|---|---|
| Scotch malt       | 0.56  | 0.2 - 1.3   |
| Scotch, old blend | 0.87  | 0.5 - 1.5   |
| Scotch, blend     | 1.20  | 0.3 - 4.2   |
| Irish             | 1.30  | 0.4 - 2.0   |
| Bourbon           | 2.40  | 0.6 - 11.5  |
| Irish             | 4.50  | 2.7 - 7.5   |
| Canadian          | 10.40   | 3.0 - 37.0  |

\*Salo, 1975.



**Figure 13.** Gas liquid chromatograms of aroma compounds produced by yeast in a nitrogen-free sugar fermentation, with a trace for comparison of the aroma compounds detected in a sample of Scotch whisky (Suomalainen and Nykänen, 1966).

The complexity of the processes for determining the presence and concentration of compounds in whisky may be illustrated by looking at one compound for which this process has been elucidated in detail. In the 1980s ethyl carbamate, a naturally-occurring component of many alcoholic drinks, was identified as being undesirable. Canada specified by regulation a maximum limit for ethyl carbamate in whisky spirit of 150 ppb and the US set guidelines of 120 ppb. Control of this compound was only possible after the processes resulting in its presence in spirit were fully understood. Extensive research in Scotland revealed the mechanism of ethyl carbamate production and facilitated the introduction of very effective control measures. These measures maintain levels close to zero

and always less than 30 ppb in distilled whisky spirit.

Cook (1990) reviewed the outcome of this research and the resulting control procedures. When barley sprouts during malting, a glycoside, epiheterodendrin (EPH) is present in the acrospire. This glycoside, which survives kilning and mashing, is extracted into the wort and converted by yeast enzymes to glucose and isobutyraldehyde cyanohydrin (IBAC). The IBAC is stable during fermentation, but when heated above 50°C at distillation it breaks down to form volatile nitriles. There are a multiplicity of potential routes nitriles can follow including reaction with other beer components or complex reactions often mediated indirectly by copper. Some of the volatile nitriles may escape these reactions and pass into the distillate. A number of reactions can remove the nitriles from the spirit, one being the reaction with ethanol to form ethyl carbamate.

Two control strategies may be used to prevent ethyl carbamate reaching the final spirit. Firstly, some varieties of barley produce low levels of the glycoside EPH (Cook, 1990), and plant breeders are now concentrating on incorporating this character in all varieties for the distilling industry. The second strategy relies on the low volatility of ethyl carbamate. Control of distillation conditions eliminates all the volatile precursor formed by copper mediated reactions. Non-volatile substances are formed prior to the final distillation in malt distillation or before rectification in grain distillation. Thus, ethyl carbamate can be minimized in the final spirit.

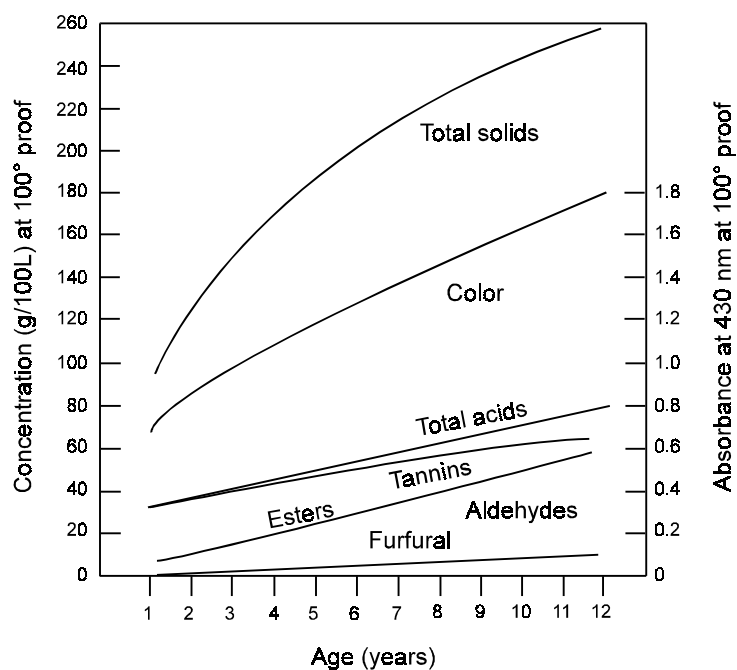
A considerable number of studies have been published on those organoleptically-important compounds arising either directly or indirectly from the oak barrels in which whisky is matured. The increase in coloring, tannin, dissolved solids and acid concentrations are not observed when whisky is stored in glass, which is proof of the importance of the oak barrels in the maturation process. An analysis of heartwood of the American oak (*Quercus albus*) gave cellulose (49-52%), lignin (31-33%), pentosans (or hemicelluloses, 22%) and compounds extracted

with hot water and ether (7-11%; Ritter and Fleck, 1923). However, when charred oak sawdust was directly extracted with water or 96° GL ethanol, the extracts obtained differed markedly in odor from aged whisky. Moreover, none of the various fractions of ethanol-soluble oak extractives contained flavors that resemble mature whisky (Baldwin *et al.*, 1967). As a result, it is now generally held that the maturation process involves not only extraction of compounds from the oak but also chemical modifications of at least some of the compounds extracted from the wood.

For a long time, most of the work reported on this aspect of maturation of whisky came from the laboratories of Joseph E. Seagram and Sons in the US. More recently, accounts of the mechanisms of Scotch whisky maturation have been given by Philip (1989) and Perry (1986) and on the maturation of whisky generally, by Nishimura and Matsuyama (1989). A theme from all this work has been the identification of a number of mechanisms of maturation common to all whiskies. These divide into addition,

subtraction and modification by reaction. There is addition of components from the oak wood, including those derived from lignin, tannins and oak lactones. There is the subtraction of volatile compounds from the maturing whisky by evaporation and adsorption on the charred surface of the barrel (Perry, 1986). Lastly there are reaction processes including establishment of equilibria among acetaldehyde, ethanol and acetal (Perry, 1986), polymerization reactions (Nishimura and Matsuyama, 1989) and oxidation-reduction reactions (Perry, 1986; Connor *et al.*, 1990). Many of the reactions involve, and are indeed dependent on, the components extracted from the wood.

Looking at several of these reactions in more detail will serve to illustrate the complexity of the maturation process. The work at the Seagram laboratories, whilst focused on bourbon, is directly relevant to Scotch and Irish whiskies for which once-used bourbon barrels are extensively utilized for maturation. Changes in the concentrations of organoleptically-important compounds during a 12 year storage of a 109°



**Figure 14.** Changes in composition of the vapor at different trays in a Coffey-still rectifier used in the manufacture of Scotch grain whisky (from Pyke, 1965).

US proof (54.5° GL) bourbon, on a 100° proof (50° GL) basis, are shown in Figure 14. The nature and origin of some of these compounds have been examined in some detail. Among the aldehydes, scopoletin and the aromatic aldehydes syringaldehyde, sinapaldehyde, coniferaldehyde and vanillin are important. According to Baldwin *et al.* (1967), these compounds could be formed by ethanol reacting with lignin in the oak wood to produce coniferyl alcohol and sinapic alcohol. Under mildly oxidizing conditions, these alcohols could be converted into coniferaldehyde and sinapaldehyde, respectively. Vanillin could then arise from coniferaldehyde, and syringaldehyde from sinapaldehyde. The increase in aldehyde content during maturation is also attributable in part to formation of acetaldehyde by oxidation of ethanol. Formation of ethyl acetate probably accounts for the steady rise in the ester content of whisky during maturation.

Several other groups of compounds not described in Figure 14 are also important in the maturation process. Monosaccharide sugars are found in mature whisky, and probably arise from the pentosans and other polysaccharides in the oak wood. Otsuka *et al.* (1963) reported that a mature Japanese whisky contained xylose, arabinose, glucose and fructose, while Black and Andreasen (1974) added rhamnose to this list when they analyzed a mature bourbon. The latter workers found that the concentrations of arabinose and glucose increased at a faster rate than those of xylose and rhamnose over a 12 year maturation period. Salo *et al.* (1976) also detected low concentrations of mannose and galactose in a matured Scotch malt whisky in addition to the sugars already noted. The concentrations of sugars in mature whiskies (of the order of 100 mg/L) are too low to suggest any sweetening effect on the beverage. Phenols are also detectable in mature whisky, although some of these probably arise during mashing (Steinke and Paulson, 1964) or from malt produced using peat-fired kilns (MacFarlane, 1968). However, Salo *et al.* (1976) reported an increase during a one year maturation of a Scotch malt whisky in the concentration of

eugenol, which is a major phenol extracted from oak chips by ethanol (Suomalainen and Lehtonen, 1976). Also present in mature whiskies are sterols, which may precipitate in bottled whisky stored at room temperature. Black and Andreasen (1973) found campesterol, stigmasterol and sitosterol in mature bourbon, in addition to sitosterol-D-glucoside, although the possibility that some of these were formed during mashing cannot be excluded. Finally, reference has already been made to the whisky lactone,  $\beta$ -methyl-octalactone, and its origin.

Not surprisingly, the nature and amounts of compounds extracted from charred oak wood depend on the ethanol concentration of the whisky. It is to some extent an advantage to mature whisky at a high proof, since this requires fewer barrels and saves on storage space. Until 1962 the US Treasury Department limited the barrelling proof of whisky to a maximum of 110° US proof (55° GL). In anticipation of this limit being raised to 125° US proof (62.5° GL), Baldwin and Andreasen initiated a series of experiments in 1962 to establish the importance of barrelling proof on changes in color and concentrations of organoleptically-important compounds during maturation of bourbon whiskies. Their report in 1973 indicated that color intensity and congener concentration of whiskies matured for 12 years decreased as the barrelling proof was raised from 109° US proof (54.5° GL) to 155° US proof (77.5° GL). The one exception was the higher alcohol content, which remained approximately constant.

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