CHAPTER

9 Batch distillation

Denis A. Nicol

INTRODUCTION

Scotch malt whisky production is derived by the double or triple distillation of a fermented mash derived from pure malt, water, and yeast as the only permissible raw materials defined by law. This chapter reviews the history of stills, differences in still design, the construction of stills and their ancillary equipment, the operation of wash and spirit stills, product quality and efficiency, potential problems to be avoided during distillation, and the role of copper in the construction of pot stills and quality of the final product.

HISTORY

Distillation was carried out from earliest times using pot stills, which were initially made of ceramic or glass and eventually of copper. These early stills were directly heated by open fires in a furnace or hearth. Heat-sensitive materials to be distilled could be heated by means of a water- or sand-filled bath, called a *bain marie*, invented by a first-century alchemist known as Mary the Jewess. Distillates were originally air-cooled condensers, which had tapering lye pipes delivering the product to glass or clay vessels. Worm tubs and eventually the shell-and-tube condenser superseded the primitive condensers. Alcohol was not recovered by distillation in any quantity until the 12th century, when stills of a crude design, caulked with clay and straw, were improved upon by using a close-fitting pot and head and lye pipe to improve the recovery of alcohol from inferior, unpalatable beers and wines.

The production of alcohol was the preserve of monks in monasteries, within whose hallowed cloisters alchemy was practised in the vain search for the philosopher's stone, which was believed to be key to the transmutation of base metals into gold. An "elixir of life" was also sought but to no avail; instead, alcohol filled this niche, apparently being prescribed for all manner of ills.

The Reformation of the Church saw the dissolution of the monasteries in England and Scotland. The knowledge accumulated by the monks was dispersed throughout the land, being acquired by individuals seeking to learn a trade or profession: brewers, distillers, alchemists, apothecaries, or barber–surgeons. The monks were instrumental in establishing the medical sciences and the early brewing and distilling

Whisky: Technology, Production and Marketing. DOI: 10.1016/B978-0-12-401735-1.00009-X

industries. Thus, whisky (*uisge beatha*, or “water of life”) was first noted by Henry II in 1170, when he and his army invaded Ireland and witnessed the natives of that land engaged in making such a drink from a mash of cereals called *usquebaugh*. The first written account of the making of whisky dates back to the Scottish Exchequer Rolls of 1494, which refer to *aqua vitae*, the tantalising and unattainable elixir of life.

DISTILLERY DESIGN

All malt whiskies, derived from low-nitrogen barleys, are produced in copper pot stills (Nicol, 1989) using a time-honoured traditional design (Figure 9.1). The following principal elements comprise a simple pot still, whether for distilling wash or low wines and feints:

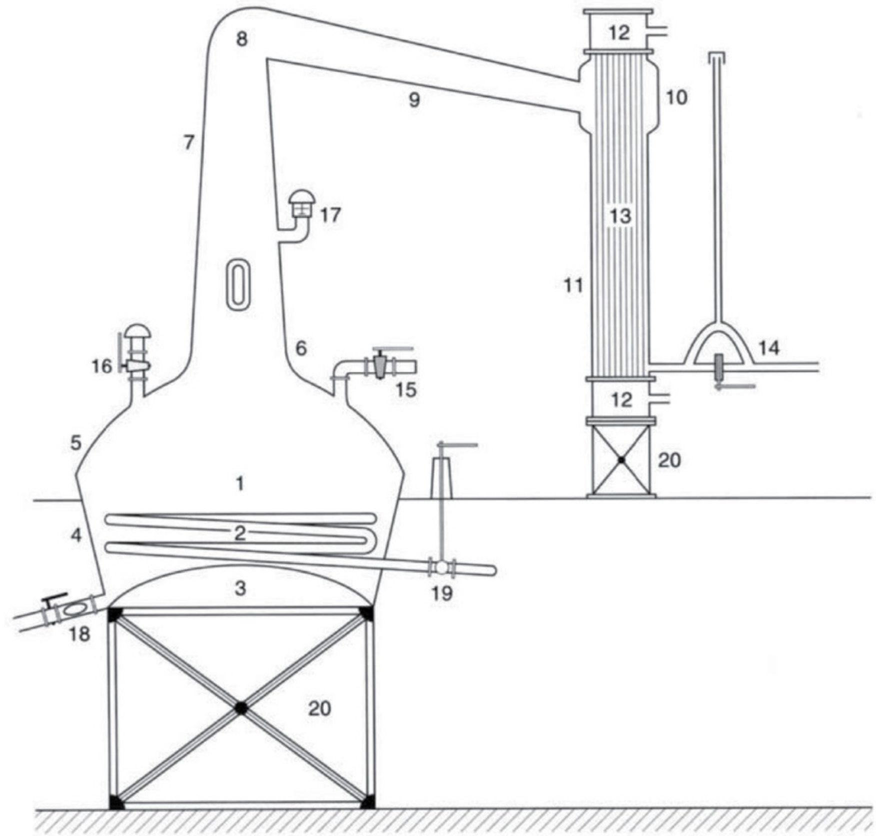
- *Heating source*—Direct fire (coal or liquid petroleum gas; see Figure 9.2) or indirect fire (steam coils, kettles, pans, or external heat exchanger, where the steam is raised by an oil- or a gas-fired boiler; see Figures 9.3, 9.4, and 9.5)
- *Pot*, which contains charge to be distilled
- *Shoulder*
- *Swan neck*
- *Head*
- *Lyne arm*, *lye pipe*, or *vapour pipe*
- *Worm tub* or *condenser*
- *Tail pipe*
- *Spirit safe*

Figure 9.6 illustrates the overall layout of a typical malt distillery.

HEATING SOURCE

Several fuels are available to heat the stills (Watson, 1989). With direct-fired stills, the pot must be designed to withstand the rigours of direct firing, and the copper crown and flue plates must be made of a sufficient gauge (16-mm) copper to withstand the intense local heating. Where copper is not expected to be exposed to intense heat, the gauge can be reduced (10 mm). The base of the direct-fired still is convex, resembling an inverted saucer, the rim facilitating discharge. The hearth or furnace upon which the still rests is of brick or steel 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 being individually controlled by dampers. Where coal is used as the heat source, 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. In Scotland today, coal is no longer, or rarely, used as a primary heat source for distilling.



Key

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

FIGURE 9.1

Pot still design.

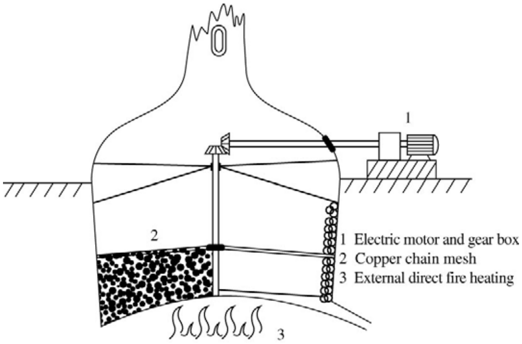


FIGURE 9.2

Direct-fired still with rummager.

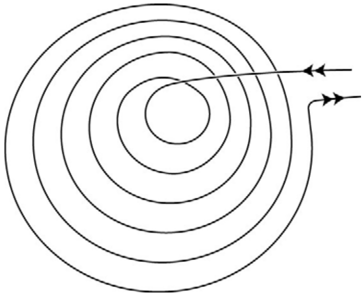


FIGURE 9.3

Plan view of steam coil.

For indirect heating by coil, pan, or kettle, steam is supplied by an oil- or gas-fired boiler and is transferred by a steam manifold from the central boiler at the crown pressure. The pressure is reduced to the desired operating pressure for the individual still 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 feints still can be fitted with an extra coil to be used to distil the middle cut gently, thus ensuring good reflux. Steam traps are strategically positioned to remove steam condensate that might otherwise waterlog the steam lines. Condensate returns, post heating, are fed back to the boiler feedwater tank via a condensate manifold as an energy-saving step. The steam demand is calculated to provide sufficient heat to bring the still to a boil and into the safe within an hour or less. The calculation should include the maximum demand for steam when heating hot liquor tanks and several stills simultaneously. The boiler output is then designed based on this demand and sized accordingly, building in extra capacity.

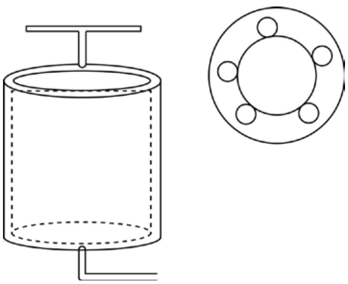


FIGURE 9.4

Steam pan.

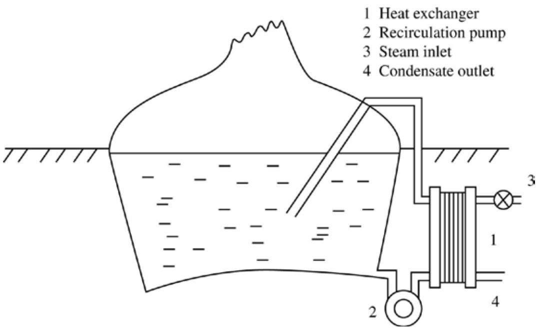
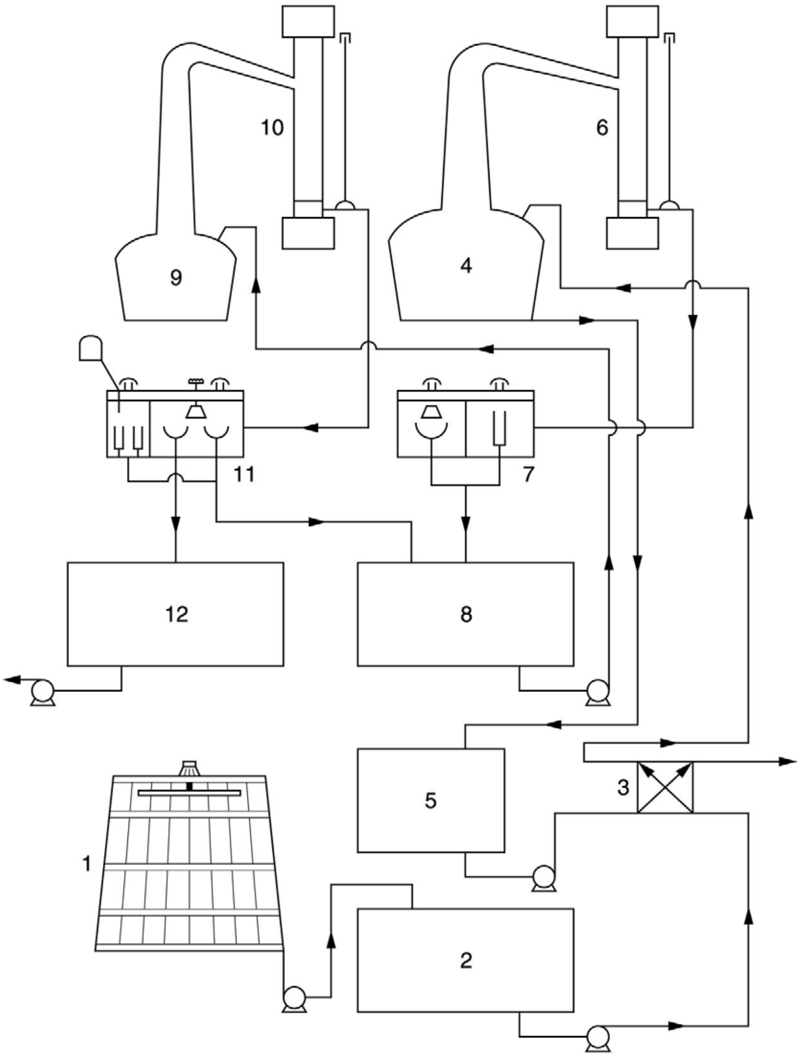


FIGURE 9.5

External steam heating using plate heat exchanger.



Legend

1. Washback
2. Wash charger
3. Wash preheater
4. Wash still
5. Spent wash receiver
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)

FIGURE 9.6

Typical malt distillery layout.

Direct firing of wash stills requires a rummager (a flail resembling chain mail) made of copper or brass, suspended from a rotating geared shaft, which as it rotates scours the flue plate and base of the wash still. This reduces the amount of charring, thus maintaining heat transfer.

To conserve energy, the external flue plates of an indirectly fired still can be insulated to prevent radiant heat loss through the sides of the pot. The shoulder and all the other parts of the still should not be insulated as this would adversely impact on the reflux, which is essential for spirit character. Wash preheating, when used, can considerably reduce total distillation times, thus saving energy. A treatise on energy-saving techniques can be found in a government report titled *Drying, Evaporation and Distillation* (Energy Technology Support Unit, 1985).

Condensers can now be run hot (>85 °C), but this can only be accomplished by using a subcooler prior to distillates entering the spirit safe to protect the delicate safe instruments. The hot water thus recovered can be incorporated in the first water for mashing purposes.

POT

The pot (Figure 9.7) can assume many shapes (e.g., onion, plain, straight, ball), provided that sufficient volume and surface area are maintained to ensure that the heating elements remain totally immersed at the end of distillation (Whitby, 1992). Such a

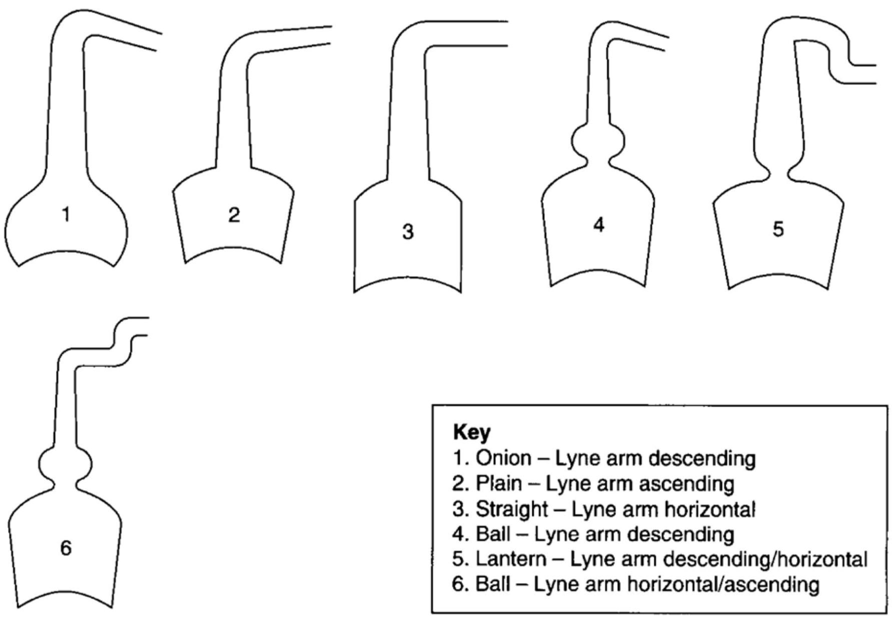


FIGURE 9.7

Shapes of stills.

problem does not exist with direct-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 among 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 the collapse or accidental discharge of the still. The reverse is applicable on charging, with sequential closing of the discharge valve followed by opening of the charging valve and finally closure of the air valve on the completion of charging and application of heat. Such requirements are redundant with today's use of automatic remote controls through programmable logic controllers (PLCs) with sequencing operations. It is now possible to operate distillation plants with a minimum of personnel. 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 swan neck has the greatest influence on the final character of new-make spirit (Plain British Spirit, or PBS). A good example of the importance of the swan neck is Hiram Walker's Lomond still, developed by Alastair Cunningham, which incorporated sieve plates in the neck of the Inverleven malt still in Dumbarton, Scotland. This type of still is still in use in certain distilling plants in the industry today. It enables "tunes" to be played with the distillates, providing distillates of different congener ratios. The neck of the still can vary from short to long. It can be tapered, straight sided, or severely swept in to the head. At the base of the neck, it can assume the shape of a lantern glass, be ball shaped, or just be directly connected to the pot, thus resembling an onion. The neck is provided with two oppositely placed sight glasses, so when the still comes in, any foaming can be seen, especially in a wash still, thus demanding a reduction in heat. A light can be attached to the rear sight glass to illuminate the still internally. A cold finger can be installed at the top of the neck. Cold water can be used to help prevent overfoaming of the still into the safe (i.e., foul distillation). A vacuum relief valve or seal pot (Figure 9.8) is fitted well above the boiling and foaming line in a wash still to prevent seizure of the valve by dextrins and solids.

HEAD

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

LYNE ARM OR LYE PIPE

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

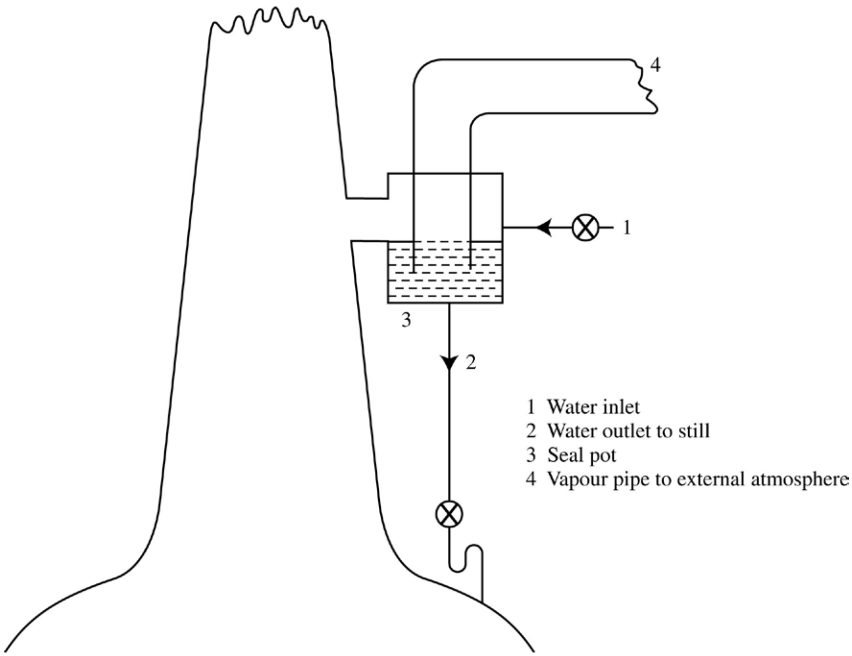


FIGURE 9.8

Anti-collapse seal pot.

has an important bearing on the spirit character. It can be designed to be horizontal, ascending, or descending to the condenser or worm tub. The angle of ascent or descent is computed to be shallow. Such permutations as there are will affect the organoleptic nature of the new spirit. The lyne arm can be interrupted by a purifier (Figure 9.9), a device fitted with baffles and cooled by an external water jacket or internal coil. Its use is to encourage heavy oils (higher fatty acid esters, C15+) to return to the body of the still during distillation. The purifier returns the heavy oils to the still via a U-bend.

WORM TUB OR CONDENSER

The worm tub, of ancient origin and design, is a large coopered, water-filled wooden vessel containing the worm (a long coiled, tapering copper tube), which is an extension of the lyne arm, beginning with a diameter equivalent to that of the lyne arm and reducing to about 76 mm, before being led to the spirit safe. Cold water is fed to the base of the tub and exits through an overflow pipe at the top. At certain times of the year, when water is in short supply, the exiting heated cooling water can be chilled by means of a cooling tower or a seawater-cooled plate heat-exchanger at coastal distilleries, before returning to the cooling system. The successor to the worm tub is the shell-and-tube condenser or even a plate heat-exchanger with copper plates

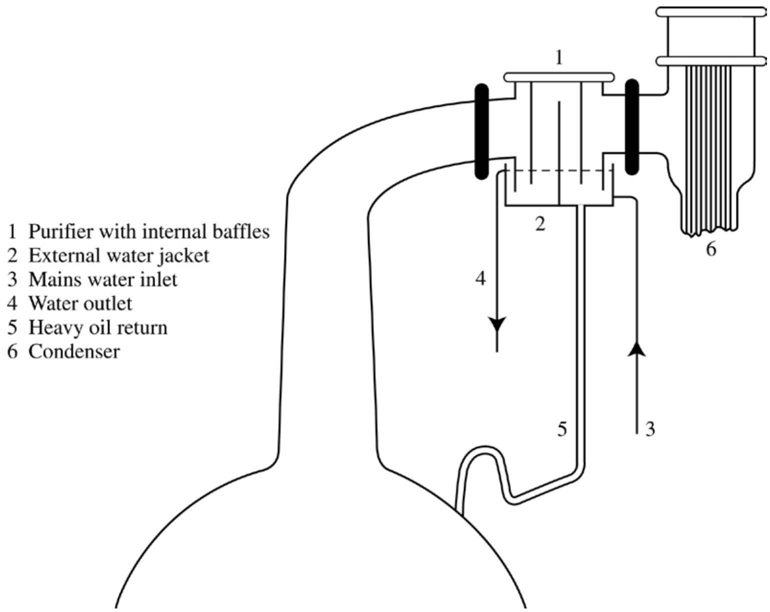


FIGURE 9.9

Purifier on low wines and spirits still.

to condense the vapours. If a condenser is used in a system that recovers sensible heat from the outflowing water at an elevated temperature (>80 °C), a subcooler post-condenser will be required to chill the spirit to less than 20 °C to protect and enable spirit-safe instruments to record reliable readings. The cooling technique can impinge on spirit quality, as worms can produce a product imbued with the aroma of sulphur compounds; this will be discussed further later.

SPIRIT SAFE

The spirit safe, which is under lock and key to prevent illicit sampling (first introduced circa 1823 at the Port Ellen distillery on Islay), is used to monitor the cut point, strength, and temperature of the outflowing distillates prior to delivery to the relevant receivers. Traditionally, by means of spirit hydrometers, the strength of low wines from a wash still or foreshots, middle cuts, and feints from a spirit still can be ascertained. The wash still distillation is monitored using a hydrometer, calibrated at 20 °C in the full range of 0 to 75% alcohol by volume (abv), or with a narrow-range hydrometer reading 0 to 10% abv, to determine the completion of wash distillation.

The spirit still is controlled by two hydrometers, the tailpipe outflow being directed to one or the other of two collecting bowls, one for foreshots and feints and the other for the potable spirit. The bowl receiving the spirit may on occasions have a muslin cloth placed across it to act as a filter for verdigris particles emanating from the condenser. Such a piece of muslin is referred to historically as a *Hippocratum*. 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 to distinguish between foreshots and true potable spirit. By mixing foreshots with water to a strength of 46% abv, a time is reached when the normally milky/turbid mixture becomes clear at this strength and pure spirit is flowing. It is this test that determines the primary cut point, while the second cut point is chosen according to the desired bouquet and strength of the final collected spirit, usually 68 to 70% abv. The demisting test hydrometer jar is equipped with a three-way sampling valve. Distillate overflows from the hydrometer jar, draining via a small bore pipe to the low wines and feints receiver. In some instances, the demisting test has been abandoned, and collection of new-make spirit commences immediately when the distillate flow enters the safe. This is driven by the desire to capture all of the flavour-enhancing congeners.

CONSTRUCTION OF STILLS AND ANCILLARY EQUIPMENT

Any reputable coppersmith or distillery engineer should be capable of manufacturing the necessary equipment, from stills to receivers. As previously mentioned, stills are totally constructed from copper, including the condensers and worms. The outer shell of a shell-and-tube condenser can be fashioned from stainless steel and the tubes from copper. Man-doors and valves can be made from brass or stainless steel. The pipework, 50 to 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 and capped with mushroom domes, and the vents can be extended to the external atmosphere through flame arresters.

Vessels for collecting charges—wash charger, low wines, foreshots and feints receivers, and intermediate spirit receivers—are now usually made of stainless steel. Coopered oak wood, epoxy-lined COR-TEN® 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, the valves must be lockable. Vessels containing spirit are fitted with agitators or rousing devices (compressed air or mechanical screw agitation) to achieve good mixing prior to taking account. Venting is also necessary in spirit, receiving, and charging vessels, with the vents leading to the external atmosphere via flame arresters.

All electrical equipment must conform to current flameproofing practice under Health and Safety Regulations. Pipe runs should be constructed to avoid dead legs and should be angled slightly toward the receiving vessels to provide for complete drainage.

WASH STILL OPERATION

A number of texts provide details regarding wash and spirit still distillation (see, for example, Lyons, 2003; Lyons and Rose, 1977; Nicol, 1989, 1997; Piggott and Connor, 1995; Whitby, 1992). Fermented wash, with an original gravity (OG) of 1050 to 1060° (12.5 to 15° Plato), is pumped or gravity fed for wash preheating to the wash charger, preheating being one of the energy-saving techniques. The charge volume (two-thirds of the wash still capacity, usually lipping the base of the man-door) is heated by either direct or indirect firing. Preheated wash, heat-exchanged with hot discharging pot ale within a few degrees of boiling, is gently brought in. Although the initial heat can be applied vigorously as soon as the flap on the spout in the safe indicates the arrival of expanding air and the condensed distillate will not be long in arriving, the heat should be reduced to prevent the still contents from boiling over, resulting in a foul distillation. As previously mentioned, this should be avoided at all costs.

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

The sight glass on the side of the still indicates the degree of frothing that 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 can be controlled to prevent a foul distillation. When the frothing subsides, the heat can be increased to allow a steady and 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% abv, when the distillation can be deemed complete. The end point of distillation at 1% abv ensures that time and fuel are not wasted in recovering a small amount of very weak spirit. A distillation cycle can last from five to eight hours, in parallel with the mashing and fermentation cycle.

When the distillation is complete and the low wines have been collected, the air valve is opened to equilibrate the internal still pressure with the external 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 open.

A record is kept of the original dip in the low wines receiver and of the final dip after distillation. The temperature and low wines strength, corrected to 20 °C in the hydrometer jar, are recorded every 15 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 minimised by ensuring that excessive heat is not applied to the cold wash, thus maintaining a minimal temperature differential.

Wash preheating reduces this charring effect, which is equivalent to a cold protein break. Deteriorating heat transfer indicates the need for caustic cleaning. Caustic soda (1 to 2% w/v) is boiled up within the still to strip deposits from the heating surfaces. Should the charring be intractable and fail to respond to the caustic solution, it may be necessary to manually scrub the heating surfaces 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, with the resultant solution boiling violently. Under prevailing health and safety regulations, work permits should be issued for both caustic cleaning and entry into a confined space. Adequate protective clothing, including chemical-resistant gauntlets and goggles must be provided, and the alkaline washing residues must be neutralised with acidic effluent prior to downstream processing.

Cleaning-in-place (CIP) systems are now available and are preferred to manual cleaning, as they greatly reduce the risk factors involved in handling aggressive and dangerous chemicals; another benefit is the ability to recycle the cleaning agent. Rinsing out with water is necessary to prevent the wash from being contaminated with cleaning agent residues.

The wash still is emptied via the discharge valve, and the discharging pot ale is heat-exchanged with the incoming wash. Hot pot ale or hot preheated wash can be held prior to charging the empty still.

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, with the flash steam produced being used to drive the still. Such techniques require the use of a spirit subcooler to ensure that the low wines are collected at a temperature no higher than 20 °C.

Wash that is not fully fermented is at risk of causing foul distillations. This exacerbates any potential ethyl carbamate problem amongst other flavour problems; hence, distillation of short fermentations should be avoided. The volume of low wines collected is approximately one-third of the original wash charge volume.

SPIRIT STILL OPERATION

Due to the increased risk of alcohol loss, low wines and feints are not normally preheated, although the discharge of spent lees may be treated similarly to that of pot ale as a source for preheating. As in a wash still, the spirit is charged to a level not exceeding two-thirds of the working capacity of the spirit still. 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 excisable value and therefore require assiduous handling. Any loss of the charge 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 spirit will flow is also dipped, both at commencement of the new spirit run and at its completion, when as feints the flow is directed toward the low wines and feints receiver. A spirit distillation is divided into three fractions:

- Foreshots
- Middle cut
- Feints.

The foreshots are the first runnings of the spirit distillation. In most cases, they are not deemed worthy of collection as potable spirit, as they contain highly volatile and aromatic compounds such as ethyl acetate. The time on foreshots is usually about 15 to 30 minutes, when the incoming strength of the distillate (~85% abv) drops to 75% abv.

Normally, a demisting test is carried out that involves mixing foreshots with water in a hydrometer jar in the safe and reducing the strength of the foreshots to 45.7% abv (old 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 displacement of the water-insoluble, long-chain fatty acids and esters (C14 and above) that have remained attached as a film to the inner surfaces of the still and in the residual subpool at the bottom of the spirit still condenser from the previous distillation. Being soluble in the high-strength incoming foreshots, they are flushed 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 low wines and feints receiver to the spirit receiver by means of the swivelling spout and is collected as new spirit.

Some blenders and distillers have abandoned the time-honoured demisting test, preferring to collect the foreshots as new spirit after a timed run, with no resort to the demisting test, regardless of the potability of the spirit. Such final distillates are high in fatty acid ester concentration, making future chill proofing of mature whisky more difficult. Regardless of the way in which the spirit is deemed potable, collection of new spirit lasts for about 2½ to 3 hours, during which time the strength drops from 72 to 60% abv, depending on the chosen final cut point.

The amount of heat applied to the still (as foreshots distil) and during the spirit distillation affects 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 action 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 endpoint of 1% abv, and the resulting residue (spent lees) can be discharged while observing the safety procedures adopted for discharging the wash still. Chemical cleaning of the heating surfaces of a spirit still is rarely necessary to avoid disrupting the internal patina, the disruption of which is implicated in flavour reactions in the still.

Sulphur compounds present in the distillate vapour (as with wash stills) are highly volatile and these odorous substances 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. As mentioned earlier, these solids are the origin of the use of the muslin gauze filter, or Hippocratum, placed over the collecting bowl in the safe. Attack by carbon dioxide, sulphur, and solids (in wash still) also thins the copper, so eventually areas subject to this attack (above the boiling line, the shoulder, the swan neck, the lyne arm, condenser tubes, and start of worm) erode, requiring patching or replacement. A still affected by erosion emulates the breathing of a dog, with the shoulders rising and falling in a rhythmic pattern called *panting*. Such a condition renders the still more vulnerable to collapse, and the offending pot should be replaced.

Like a wash distillation, a spirit distillation should last from five to eight hours, paralleling the wash 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% 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 spirit 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 time basis.

Low wines and feints receivers and chargers act as separating vessels. The last runnings of a spirit distillation contain heavy oils and 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% abv, these compounds undergo a phase separation, such that the esters float on top of the aqueous layer while a small concentration is dissolved in the low-alcohol aqueous layer. If the concentration of the lower alcoholic strength aqueous layer is allowed to exceed 30% abv, these floating surface oils will migrate into this layer, completely dissolving. This effect eventually impacts not only the demisting test but also the entire spirit distillation—potable spirit cannot be collected, as the low wines and feints charge 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% abv, it is still possible to suffer from distillation problems. Presentation of the floating surface layer of heavy oils or higher fatty acid esters as a charge to the still (by 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 entire 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% ABV and thus stimulating hydroseparation. The surface phase in the low wines and feints charger must not be allowed to enter the still on charging. Adherence to these principles will ensure a consistent product, with regard to both nose and analysis. The low wines and feints components will reach a steady concentration state and maintain equilibrium during subsequent distillations.

PRODUCT QUALITY

A competent distiller ensures that the distillery staff is fully aware of the parameters that must be controlled to provide 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 that tradition.

First, the wash (whether traditionally derived from a wort's recipe producing an OG of 1050° or 1060° using high-gravity brewing techniques) 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, exceeding 48 hours in duration, undergo malolactic fermentation, the products of which, when distilled, produce a superior, more mellow spirit. Even at 48 hours, such a secondary fermentation is unlikely to have occurred, as it relies on the autolysis of yeast cells with the spilling of the cell contents to provide nutrients for the lactic acid bacteria.

Fermentations should not be less than two days long to avoid an excessively gassy wash. A gassy lively wash is difficult to distil and has much frothing, leading to the risk of foul distillation and producing a final spirit with an unacceptable ethyl carbamate concentration. The wash (still) charge should ideally not exceed two-thirds 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 gently to minimise 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 heating elements, coils, pans, or kettles). With wash preheating, the temperature difference is much reduced and charring is less prevalent.

An adequate supply of cooling water must be provided to prevent hot, uncondensed vapours from reaching the spirit safe, causing irreparable damage to safe instruments, jars, bowls, and the final distillate quality. Spirit-filled thermometers have now replaced all mercury-filled thermometers for obvious health and safety reasons.

The bouquet of the final spirit is influenced initially by the raw materials—particularly the variety of barley malted and the pitching yeast. Water also influences spirit character. Process parameters, including mashing temperatures, washback setting temperatures, length of fermentation, and variable new spirit cut points will impact the flavour characteristics of the spirit. Such production variations can be ameliorated by collecting several days' distillations in one large vat. Variability in individual production quality will then be eliminated by averaging congener concentrations.

With peated malts, the spirit produced reflects a marked concentration of steam volatile phenols, whose arrival in the final spirit tends to concentrate toward the end of the middle cut collection as the alcohol-to-water ratio changes with decreasing spirit strength, favouring phenol entrainment. To enhance the phenol concentration in the new spirit, the strength of the second cut point in the middle cut can be reduced, but not at the expense of producing a feinty spirit. A cut point of not less than 60% abv would be acceptable.

The rate of distillation is critical. Too rapid a distillation will result in an unpalatable spirit, fiery in aroma and taste and lacking a refined congener balance. Foreshots and the middle cut should be carefully and gently distilled to ensure adequate reflux, with foreshots completely purging the oily residues of the previous distillation. As mentioned earlier, it is these residues that demand the demisting test. Slow spirit distillation ensures the production of a clean balanced spirit devoid of aroma and flavour blemishes.

It must be emphasised that an adequate supply of cold water to condensers or worm tubs should be maintained. Inadequate cooling (>20 °C) will lead to spirit endowed with an aroma reflecting higher concentrations of compounds associated with the feints. This is also true of forced or too rapid distillation. Warm weather, with resultant warmer cooling condenser water, demands that the distillation rate be reduced to allow the spirit to be collected at the desired temperature (20 °C). Prolonged distillation times will have an adverse effect on production schedules (e.g., mashing, fermentation). Distilling spirit, during times of high ambient temperatures, will increase losses due to evaporation. Thus, the practice of malt distilling requires low ambient air and water temperatures, as experienced during late autumn, winter, and early spring. Distillation in past 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 high summer temperatures. Summer was the "silent season", when distillery staff were engaged in the maintenance of plant and buildings, harvesting barley, and bringing the peats home.

Chemical contaminants that can bedevil the malt distiller are nitrosamines, ethyl carbamate, methanol, pesticide residues, haloforms, polycyclic aromatic hydrocarbons, and pesticide and herbicide residues, all of which are monitored in compliance with government regulations and procedures. Some of these contaminants are derived from the raw materials and others from processing malt or during distillation. Genetically modified cereals and yeast come under scrutiny, as the definition of Scotch whisky demands the use of only pure water, yeast, and cereals derived from natural sources.

Copper has already been mentioned as a silent contributor to spirit quality as it removes highly volatile sulphur compounds. It is also 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 distillation equipment to avoid compromising quality, but it can be used for ancillary pipework and vessels. The original gravity (OG) of the wash impacts on spirit quality, and it has been determined that OG values in the range of

1045 to 1050° (11.3 to 12.5° Plato) encourage the formation of esters, thus imparting a fruity, sweet aroma to the finished product. The role and impact of copper on the quality of potable spirit are discussed further in Chapter 11.

EFFICIENCY AND PRODUCTION YIELD

Following the design and construction of a well-balanced distillery, where milling, mashing, fermentation, and distillation are in harmony, it is fairly easy to establish in-tandem programmes of mashing. This is achieved by ensuring that the time cycles for mashing, fermentation, and distillation are in step, with the week being divided into set periods reflecting the mashing cycle. Consequently, if mashing takes six hours to complete (with washbacks individually filled within this time period), the maximum distillation time from charging to discharging should not exceed six hours. This enables four mashes per day to be performed. The distillery may be fully automated, removing the human element and its uncertainty.

One tonne of malted barley, fully modified and efficiently mashed, should ensure complete extraction of available fermentable sugars, resulting in an overall distillery yield approaching 425 litres of pure 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.

In the distillery, it is essential that the integrity of the pipework, vessels, and stills is maintained without leaks. Spirit can be lost through insidious, invisible vapour leaks that are not easily detected. The worm or tube bundle in a shell-and-tube condenser is under constant attack by sulphur compounds and carbonic acid emanating from the vapour phase, which eventually corrodes the copper. Condenser or worm leaks are noticeable by the entry of cooling water into the product side, reducing the strength of the distillate (as detected by the safe hydrometers) and most definitely producing a water flow into the safe when the still is off. Such a scenario requires that the distillation be stopped, with the offending tubes spiled or worms patched under strict safety conditions, before continuing further distillation. In the case of shell-and-tube condensers, several tubes may be affected, and the condenser should be pressure tested for further possible tube weaknesses. In the event of multiple tube failures, the condenser will require retubing.

Thinning copper on the shoulder of the pot, due to erosion at the boiling surface, swan neck, or lyne arm, can lead to pinhole leaks as the copper becomes spongy. Such leaks are rectified by soldering or the temporary use of molecular metal. Soldering requires the use of a blow torch, and flammable vapours must be purged from the system by blanking off receivers to prevent explosion and fire. Such repairs require the complete cessation of distilling operations. Leaks are not acceptable and must be dealt with as soon as is practically possible.

Other losses occur via the pot ale or spent wash and spent lees, when the distillation endpoint is not accurately observed. Again, these endpoints would indicate that energy is being wasted if the distillation is continued beyond 1% abv, as indicated by the safe hydrometers. Premature ceasing of distillation will result in significant detectable ethanol being present in the pot ale or spent lees. Permissible spirit losses are as follows:

- Pot ale <0.03% abv
- Spent lees <0.03% abv
- Condensate <0.0001% abv
- Condenser water <0.0001% abv

Where pot ale is evaporated for syrup, residual ethanol interferes with the evaporation efficiency.

The distillery yield is calculated from the weekly production figures. It takes account of the weight of malted barley used and the amount of spirit remaining in the low wines and feints receiver, the intermediate spirit receiver (ISR), and the final spirit receiver/warehouse (W/H) vat. Depending feints, carried forward from the previous week, are deducted from the total sum of spirit produced, expressed in litres of absolute alcohol (LAA).

CALCULATION OF DISTILLERY YIELD

(a) Depending feints	= 15,500 LAA (carried forward from previous week)
(b) Spirit produced in ISR	= 7200 LAA
(c) Spirit receiver W/H vat	= 30,300 LAA
(d) Feints remaining	= 13,500 LAA
(e) Spirit produced	= (b) + (c) + (d) – (a)
	= 7200 + 30,300 + 13,500 – 15,500
	= 35,500 LAA
(f) Tonnes of malt mashed	= 85.54 t
(g) Distillery yield	= Spirit produced (LAA) ÷ malt used (t)
	= 35,500 LAA ÷ 85.54 t
	= 415 LAA per tonne

Her Majesty's Revenue and Customs (HMRC) 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

As an example, the attenuation across several fermentations comprising a week's production is 57° gravity from an OG of 1055° (13.75° Plato) and a final gravity (FG) of 998° or –02 (02 under). For ten fermentations with a total volume of 488,500 litres of wash and an average attenuation of 57°, the attenuation charge is calculated as follows:

$$\begin{aligned}\text{Attentuation charge} &= \frac{\text{Litres of wash} \times \text{Average attenuation}}{8 \times 100} \\ &= \frac{485{,}000 \times 57}{800} \\ &= 34{,}556\ \text{LAA}\end{aligned}$$

HMRC calculates the percentage over-attenuation from the following formula:

$$\left(\frac{\text{Spirit produced (LAA)} \times 100}{\text{Attenuation charge}} - 100\right) \times 100$$

$$= \left(\frac{35{,}500\ \text{LAA} \times 100}{34{,}556\ \text{LAA}} - 100\right) \times 100 = 2.73\%$$

Hence, an acceptable percentage over-attenuation has been achieved at 2.73%. A value of 3.0% is deemed acceptable. Any departure from this figure by more than 1 to 2% either way demands an investigation.

Under-declaring the OG of worts collected will inflate the figure, while over-declaring will reduce the figure. Recourse to laboratory analysis for OG determinations is necessary. The law demands that at least six declared washbacks per week should be analysed to find the true declarations, with the distiller adjusting the saccharometer readings to allow for the amount of work conducted relating to the temperature and gravity of the wash 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 final spirit at higher natural strength than double-distilled whiskies and is primarily carried out in lowland distilleries. It is similar 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, and 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 and spirit stills impacts on the final bouquet and strength of the new spirit. This is collected at a strength in excess of that of normal double-distilled products, which are usually in the region of 68 to 72% abv. The triple-distilled product can approach a strength of 90% ABV. The Irish distillers boast very large pot stills in comparison to the double-distilling techniques of their Scottish counterparts.

DEALING WITH DISTILLATION PROBLEMS

As with the all manufacturing processes, problems can occur that impact the quality of the finished product if not rapidly addressed. Such problems can occur during the mashing, fermentation, and distillation stages. Declaration problems related to over- or under-attenuation attract the unwelcome attention of Revenue and this is best avoided!

Atypical over-attenuation percentages can be attributed to false declarations when determining the OGs in a set washback. As previously discussed, this can be overcome by recourse to laboratory determinations of OG in at least six declared washbacks, calculating the allowance for loss in gravity due to fermentation and to temperature. The average calculated OG and the difference between what was observed at the time of declaration and the true OG from the laboratory checks are the figures 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 wort and temperature is then not suspect guesswork but a true reflection.

All worts and final wash should be attemperated to 20 °C prior to taking readings. Product losses, with low percentages of over-attenuation accompanied by low yields when compared with the potential spirit yields obtained by analysis, are invariably caused by one or a combination of the following problems:

- Poor mash tun extraction
- Infection (Geddes, 1985)
- High wash fermentation temperatures
- Physical losses (worts, wash, low wines, feints, spirit)
- Human error

To satisfy Revenue, any loss should be accountable to avoid the possibility of excise penalties, as long as the loss is accidental.

To improve mash tun extraction efficiencies, it is necessary to examine sparge-to-grist ratios as well as mashing temperatures, especially the first water. Overloading a mash tun with goods is definitely counterproductive, exacerbating fermentable sugar losses in the draff.

Bacterial infection, which competes with yeast for fermentable sugars, is alleviated by paying close attention to cleaning regimes, concentrating on mashing, fermentation equipment, and pipe work; any dead legs should be eliminated (see Chapter 17).

Attention should be paid to setting proper fermentation temperatures depending on prevailing atmospheric conditions. Fermentations that exceed 33 °C contribute to evaporation losses and may also contribute to the growth of strains of *Lactobacillus*, which will be responsible for off-notes and the production of acrolein during distillation. Physical losses of process materials can be accidental due to plant or human failure, demanding investigation to satisfy Revenue.

The nature of the wash, with dissolved carbon dioxide, can lead to foul distillations when the still boils over into the safe, damaging the safe instrumentation and glasswork. Low wines and feints, thus contaminated with wash, can be a source of increased ethyl carbamate formation. Overzealous application of heat, an overloaded wash still, young lively wash, or blocked condenser tubes can also lead to this problem. Sensory or organoleptic problems, presenting themselves as feinty spirit, are caused by the overrun of the middle cut. Safe spirit hydrometers and high condensate temperatures should be suspected and the necessary checks made. It is possible to be plagued with blank spirit runs when no potable spirit is collected, producing adverse effects on fuel usage. This is caused by weak low wines and feints charges or by an increasing charge strength (>30% abv), when higher concentrations of fusel oils dissolve in the alcoholic aqueous layer. The problem can be overcome by reducing the strength of the charge (<30% abv) by adding water.

Discharging the total content of the low wines and feints charger into a spirit still is a recipe for blank runs, as the surface layer of fusel oils contaminates the still, making it impossible to have a successful demisting test when applied to the foreshots. Increasing time lengths to clear foreshots can indicate an increasing concentration of fusel oils in the foreshots. A balanced distillation regime can help to alleviate these problems.

The collection of weak spirit, resulting in a congener imbalance, can be attributable to poor reflux, poor cooling, or a leaking condenser. To overcome this effect, the distillation rate can be lowered to improve reflux and cooling. Leaking condenser tubes manifest themselves by presenting a water flow to the safe when the distillation is complete. The condenser tubes should be checked by pressure testing with water. Any offending tubes should be temporarily blanked off by spiling, and a constant check should be maintained for future potential tube failure. One leaking tube indicates that neighbouring tubes should be treated with suspicion.

Wash stills are prone to fouling of direct or indirectly fired heating surfaces due to the nature of the wash with its solids and unfermentable sugars. Fouling impedes heat transfer, resulting in increasing distillation times and energy wastage. A 1 to 2% caustic soda solution treatment of the still or heating elements should be sufficient to solve this problem.

High ambient water and air temperatures, especially in summer, combine to elevate the temperature of distillates entering the safe. This demands that the distillation should be slowed so that the low wines, feints, and spirit are collected at a slower rate and at temperatures as close to 20 °C as possible. Thus, evaporation losses and detrimental organoleptic effects are reduced but unfortunately at a slower production rate. However, some distillation losses are difficult to detect. An almost inaudible high-pitched hissing noise emanating from a steam coil indicates a steam leak, which will not only dilute the charge but also result in the charge entering the condensate system, with resultant losses and condensate contamination. Most indirect heating material is constructed from stainless steel and less so from copper. Nevertheless, regardless of the material of construction, the integrity of all flanges should be examined, and, in the case of copper, the existence of any cracks or pinholes should be determined.

Pinhole vapour leaks occurring above the charge line in the still can be readily detected when external verdigris blue–green staining occurs on any of the surfaces of the still components, from neck to head and lyne arm. If not dealt with quickly, these insidious and unsightly leaks, especially if occurring at inaccessible places, can rapidly grow. Passing valves, air and anti-collapse valves, and flanges on pipe runs can all contribute to product loss. Management must therefore maintain a high level of inspection for physical losses due to an ageing plant, especially copper, fundamental to the quality of the final product. When the body of a copper still begins to thin above the charge line, like an old dog it begins to pant, visibly heaving up and down. Such a still is reaching the end of its useful days and will require either patching or, in extreme cases, replacement.

THE ROLE OF COPPER IN THE QUALITY OF NEW WHISKY

Copper has been mined as an ore since the Bronze Age (~3500 BC). As a metal it has many uses relating to its properties. It is soft and easy to work in its annealed state. Its hardness and tensile strength can be doubled by working it cold through hammering, beating, rolling, or drawing. For distillery purposes, its properties related to its malleability, thermal conductivity, and resistance to corrosion make it an ideal metal for the manufacture of a distillation apparatus.

It was not until the introduction of stainless steels that a more subtle property of copper manifested itself. It was known that a copper distillation apparatus had to be replaced periodically, as it was subject to wear. Certain distillers believed that to prolong the life of the distillation equipment stainless steel would make a suitable substitute. Plans were made to replace the copper condenser tubes and even the stills with longer lasting stainless steel, as American distillers had done in their distilleries.

It was not until the new spirit took on a sulphury odour that the impact of stainless steel on the bouquet of new spirit was realised. New spirit derived from worm tubs reflected a similar note but of less intensity. It was therefore back to the drawing board! To focus on the area most likely to have an impact on this aroma, a pair of Quickfit glass stills was assembled under laboratory conditions. In the first glass still, copper turnings were placed immediately above the condenser, in the splash head, and also in the connection between the glass still and the condenser. The other glass still was assembled without copper (unpublished results, 1968). Low wines were obtained from a distillery that boasted stainless steel condensers in the wash stills. Using the glass laboratory apparatus, the low wines were distilled through both small stills, one with and the other without copper turnings.

The products from each still were examined organoleptically. The low wines distilled over the copper produced a noticeably clean spirit compared with the still without copper. Further trials were carried out to find out which part of the apparatus containing copper had the most impact on the bouquet. The conclusion was that hot spirit vapours reacted readily with copper in the tube bundle at the top of the condenser and to a lesser extent on the copper surface in the lyne arm. Where the reaction was greatest at the top of the tube bundle, it was associated with the erosion of copper, requiring eventual retubing of the condenser with new copper. However, little had been known about the substances contained within the low wines vapour, except that such aromas were derived from sulphur compounds associated with foul smells. Stills with all-copper condensers, including the tube bundles, produced an aroma with a lesser contribution from sulphur, and stills with worm tubs possessed a hint of a sulphury aroma. It was not until recently that the impact of the offending sulphur compounds was identified through work carried out by a team at the Scotch Whisky Research Institute (Harrison et al., 2011)

The primary offending compound, dimethyltrisulphide (DMTS), was identified as having the most meaty and sulphury aroma. It was also concluded that sulphury or meaty aromas were established in the wash still condensation phase on the copper condenser and in the low wines or spirit still on the inner copper surface of the body of the still, reflecting the results derived using the Quickfit apparatus with and without copper turnings. This is a scientifically interesting area that demands further investigation with regard to the mechanisms involved.

One of the recently constructed distilleries has incorporated two condensers for each wash still, one of copper and the other of stainless steel. They were being shared by means of a bifurcate valve arrangement in the lyne arm, so that the still can produce either a sulphury or sulphur free spirit. The organoleptic contribution of highly volatile sulphur compounds to new spirit decreases upon maturation, unless it has been obtained from casks treated with burning sulphur sticks prior to purchase.

THE FUTURE

Environmental, energy, fiscal, and health and safety pressures continue to be experienced by distillers due to greater public awareness of diminishing wildlife species and oil and gas supplies, profligate governmental schemes and banking mismanagement, and ever-present health and safety issues that demand greater monetary contributions through taxation. With the introduction of computerised controls in almost every distillery department—from malt intake to milling, mashing, fermentation, distillation, and cask filling—the industry has reduced its production labour force as a cost-cutting exercise; one person can now operate a distillery, but Law requires that person to have a working companion. This trend towards automation is set to continue. Raw materials (barley, malt, yeast, and water) will continue to come under scrutiny during the search for ways to improve distillery yields so whisky, the elixir of life, will continue to dominate the spirits market. Other countries are attempting to reproduce the success of Scotch whisky by producing products similar to Scotch. By retaining age-old but modernised techniques and a standard recipe the continued success of whisky is assured.

REFERENCES

Energy Technology Support Unit, 1985. Whisky distilling. In: Drying, Evaporation, Distillation: The Potential for Improving Energy Efficiency in Twelve Industrial Sectors. Vector Publishing, Farnborough, U.K., pp. 34–41.

Geddes, P.J., 1985. Bacteriology in the whisky industry. Journal of the Institute of Brewing 91, 56–57.

Harrison, B., Fagnen, O., Jack, F., Brosnan, J., 2011. The impact of copper in different parts of malt whisky pot stills on new make spirit composition and aroma. Journal of the Institute of Brewing 117 (1), 106–112.

Lyons, T.P., 2003. The production of Scotch and Irish whiskies: their history and evolution. In: Jacques, K.A., Lyons, T.P., Kelsall, D.R. (Eds.), The Alcohol Textbook. 4th ed. Nottingham University Press, Nottingham, U.K., pp. 193–222.

Lyons, T.P., Rose, A.H., 1977. Whisky. In: Rose, A.H. (Ed.), Economic Microbiology, Vol. 1. Alcoholic Beverages Academic Press, New York, pp. 635–692.

Nicol, D.A., 1989. Batch distillation. In: Piggott, J.R., Sharp, R., Duncan, R.E.B. (Eds.), The Science and Technology of Whiskies. Longman Scientific & Technical, Harlow, U.K., pp. 118–147.

Nicol, D.A., 1997. Distilling, past, present and future. Ferment 10 (6), 382–391.

Piggott, J.R., Connor, J.M., 1995. Whiskies. In: Lea, A.G.H., Piggott, J.R. (Eds.), Fermented Beverage Production. Blackie Academic & Professional, London, pp. 247–274.

Riffkin, H.L., Wilson, R., Bringhurst, T.A., 1989. Ethyl carbamate formation in the production of pot still whisky. Journal of the Institute of Brewing 95, 115–119.

Smith, G.D., 1997. A–Z of Whisky. Neil Wilson Publishing, Glasgow.

Watson, J.G., 1989. Energy management. In: Piggott, J.R., Sharp, R., Duncan, R.E.B. (Eds.), The Science and Technology of Whiskies. Longman Scientific & Technical, Harlow, pp. 327–359.

Whitby, B.R., 1992. Traditional distillation in the whisky industry. Ferment 5 (4), 261–267.