Module 2

2.3 Continuous Distillation

2.3.1 Design
2.3.2 Operation
2.3.3 Ethanol Profiles
2.3.4 Congener Behaviour
2.3.5 Pre-distillation Influences on Quality

DIPLOMA IN DISTILLING

Contents

Abstract

In this Unit of the Diploma in Distilling, 2.3 Continuous Distillation, we will examine the concerns specific to continuous and column distillation. Batch and pot distillation is covered in section 2.2 Batch Distillation.

We will start by discussing still design and operation (2.3.1 & 2.3.2) before examining ethanol profiles and congener behavior (2.3.3 & 2.3.4).

Learning Outcomes

On completion of this section you should be able to:

1. *Understand the design elements of continuous stills.*
2. *Describe the operation of a continuous still.*
3. *Explain congener profile in rectification columns and the removal of feints.*

Prerequisite Understanding

Basic scientific knowledge and terminology, Unit 2.1 Distillation.

2.3.1 Continuous Distillation Design

Introduction

The separation of components of a liquid by differences in volatility (Unit 2.1 & 2.2) can be performed even more efficiently by continuous distillation

A basic design of continuous still for a 2-component mixture, e.g. ethanol and water, is shown in Figure 1.

The heated mixture (at bubble point temperature, is fed into the column of perforated plates at about half of its height. As the liquid falls down through the plates the more volatile component (ethanol) is preferentially stripped out in the lower half of the column by the rising hot vapour, and the less volatile water continues down. At the bottom, some water is boiled back to steam in the reboiler (heat exchanger) to provide a continuous supply of hot vapour, the remainder is discharged to drain. Meanwhile, vapour rising through the upper half of the column becomes increasingly rich in the more volatile ethanol, ultimately to the azeotropic concentration. Part of the product of the top condenser must be returned to the column as reflux, to provide a layer of liquid on each plate, the remainder is drawn off as alcohol.

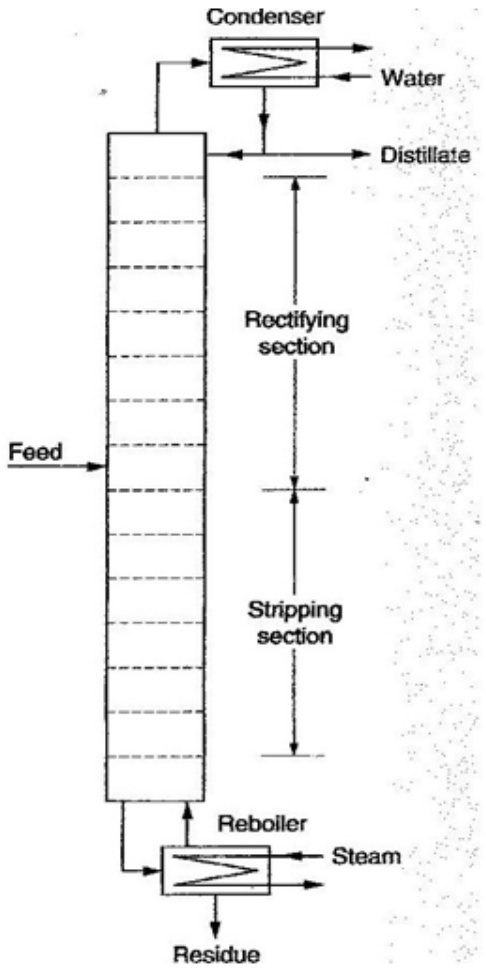

Figure 1 Simple distillation column.

However, that simple still design is unsuitable for distillation of potable spirits for the following reasons.

- The still is required to separate not only ethanol and water but several hundred flavour congeners from raw materials and fermentation. Therefore the number of plates required is so large that a single column would be too tall. So it is normal practice to operate with two linked columns side-by side.
- The top condenser can not be a total condenser (i.e. all vapour is condensed) as described above, since a proportion of the most volatile congeners must be continuously vented off to prevent unacceptable accumulation in the still.
- However, there is still an unacceptable concentration of these congeners at the highest plates so the best spirit is drawn

off lower down the still, not from the top condensate.

- Although a reboiler would be a more economical use of steam and boiler feed water, and would reduce the volume of water to be removed in stillage (spent wash), normally it cannot be used, since flavours produced by re-heating the spent beer/wash would be unacceptable. Also, yeast and other solids (e.g. cereal particles, in the case of whisky) could bake on to the steam coil, creating heat transfer problems as well as off-flavours. However, a reboiler is suitable with wine for brandy production, and perhaps with a clear beer for white rum.

Therefore a more practicable design is an analyser (stripper) column, representing the lower half of the still in Figure 1, and normally heated by direct steam injection, with heated beer/wash/wine feed on to the top plate. The hot spirit vapour from the top of the column passes to the bottom of the rectifier, equivalent to the upper half of Figure 1, and the individual components of the beer/wash/wine are separated over the height of that column, with the spirit being drawn off near the top. Three variations on that arrangement are described below.

Continuous still design for potable spirit production

The still system is two columns, each containing a stack of 30 – 50 perforated plates, usually about 0.4 m apart, with the feed into the top of the analyser/stripper column A of Figure 2. To maintain steady conditions the feed of beer/wash must be heated almost to the boiling point for its alcohol concentration (bubble point temperature. This is achieved in the still of Figure 2 by passing the feed through a heat exchanger, the top condenser of column B. Condensate is returned to the column as reflux, and the heated beer/wash is fed into the top of column A. Each perforated plate (sieve plate) carries about 5 cm depth of beer, wash or wine, supported by the upward flow of vapour through the holes, and maintained at 5 cm depth by over- flow weirs created by the "downcomer" pipes to the next lower level (Figure 3) projecting by that amount above each plate. The downcomers reach almost to the lower plate and are protected by circular 5 cm high weirs (seal pots), maintaining that depth of liquid to prevent escape of steam or vapour up the pipes. The downcomer pipes are alternately at opposite ends of rectangular plates, or diametrically opposite for circular, to create a flow across the plate, and so on down the column. Each individual plate is fitted with safety valves to protect against the effects of blockage. In addition, pressure and vacuum relief valves are fitted to the tops of the two still columns.

Column A is the analyser or stripper column, where all volatile material is evaporated by the upward flow of steam and hot vapour to pass to the base of column B, and non-volatile residue leaves the bottom of column A as stillage (spent wash). In theory, column B should be fitted on top of A (Figure 1) , so the pipe carrying hot spirit vapour from the top of A to the bottom of B is the equivelant of the flow from one sieve plate to the next one above.

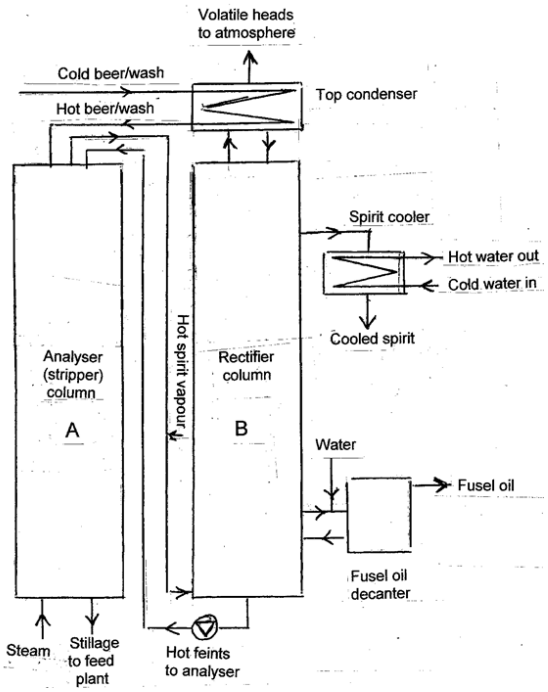

Figure 2 Continuous distillation of white rum or grain whisky.

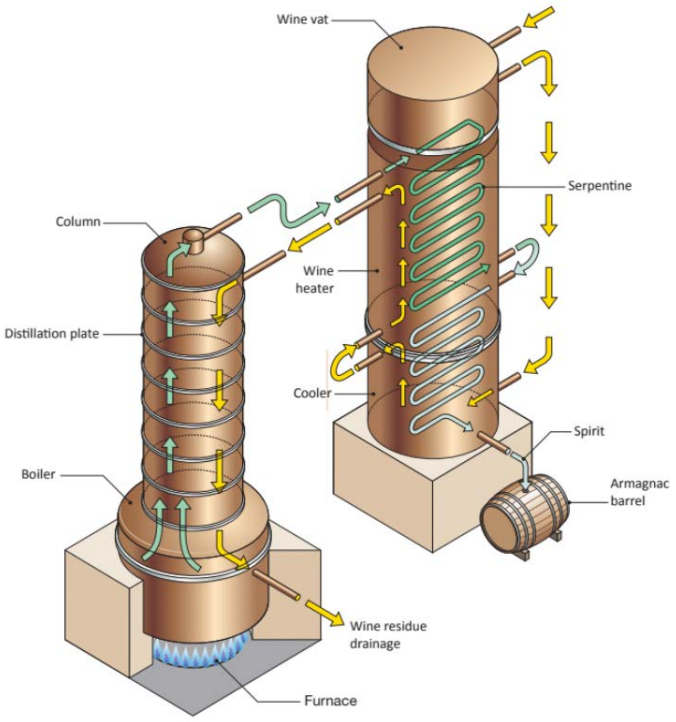

Figure 4 Column still for Armagnac production.

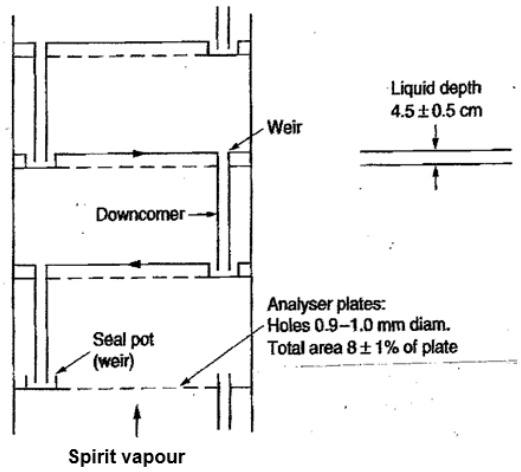

Figure 3 Flow across sieve plates between downcomer pipes.

Column B (Figure 2) is the rectifier section, with the upward flow of hot spirit vapour bubbling through, and partly condensing in, the 5 cm depth of liquid on each sieve plate. There is a gradual cooling as the vapour rises, so the rectification process is the separation of the least volatile, highest boiling-point congeners at hotter lower levels, and congeners of increasing volatility higher up the column. Spirit is collected as the liquid flooding the "spirit plate" near the top of the rectifier. Since it is already liquid, there is no need for a condenser, but a cooler is required to reduce the temperature of the spirit from about 79°C to 20°C. There is a significant ethanol concentration in the liquid cascading to the base of the rectifier, which is pumped to the top of column B. This flow is equivalent to the liquid in a single tall column falling to the next plate below.

Most of the vapour above the top sieve plate is condensed in the heat exchanger which heats the incoming beer/wash, but a proportion of the most volatile congeners must be allowed to escape to prevent their accumulation in the still system. Condensed ethanol and congeners of similar volatility are returned to the top plate as reflux, to begin the cascade of liquid phase back down the rectifier. This is the only true equivalent of the reflux in a pot still, since the still columns are enclosed in insulating jackets. However, the condensation and evaporation at each level, and the upward and downward flows of vapour and liquid respectively from each plate, create the same effect.

Since there is a significant content of higher alcohols in the wash, these compounds must also be continuously removed from the system to maintain steady-state conditions. The zone of highest concentration is in the lower section of the rectifier, from where they are drawn off as "fusel oil".

Traditionally the sieve plates are made of copper. The frame may also be copper but is more likely to be cast iron or stainless steel. However, many modern stills are now built entirely of stainless steel to reduce corrosion, but sacrificial copper, usually as copper turnings, must be included to remove sulphur compounds at sensitive points. Copper is especially important at the spirit plate of the rectifier. Also, copper mesh is fitted to the vapour pipes at the top of the rectifier, to remove entrained droplets and volatile sulphur compounds from the spirit vapour, as well as acting as a flame arrester.

Two other designs of still are sufficiently

common to justify explanation here.

Barbet Stills

Used in many white rum and wine/molasses neutral spirit distilleries, Barbet Stills are intended to avoid as much as possible the recycling of process streams between columns, which, it was believed, increased the accumulation of unwanted congeners.

The difference from the standard stripper column A of Figure 2 is that the beer/wine feed is about 5 plates down from the top of the column, and the hot spirit vapour for the rectifier leaves from about 5 plates below that. So the higher levels of the stripper column act as a rectifier, separating the most volatile congeners (whch are vented to atmosphere) from ethanol, which passes back down the column to leave either as liquid spirit or hot spirit vapour. These streams are fed into the rectifier about 20% up its height, not at the base. So the whole column must have 20% more plates than the rectifier of Figure 2. The bottom 20% of column B strips ethanol from the liquid descending from the lowest plate of the true rectifying section, which would otherwise have been recycled to A as hot feints. This needs a steam supply, generated by re-boiling the descending liquid, so the rectifying section proper receives hot vapour both from its own reboiler and from column A. The hot water with a small amount of low- volatile congeners which leaves the reboiler at the bottom of column B is passed through a heat exchanger (not shown in Figure 3) to drain. As in the standard still system, fusel oil is removed continuously from the zone of highest concentration in the lower part of the rectifying section and the ethanol/water phase is returned to the column.

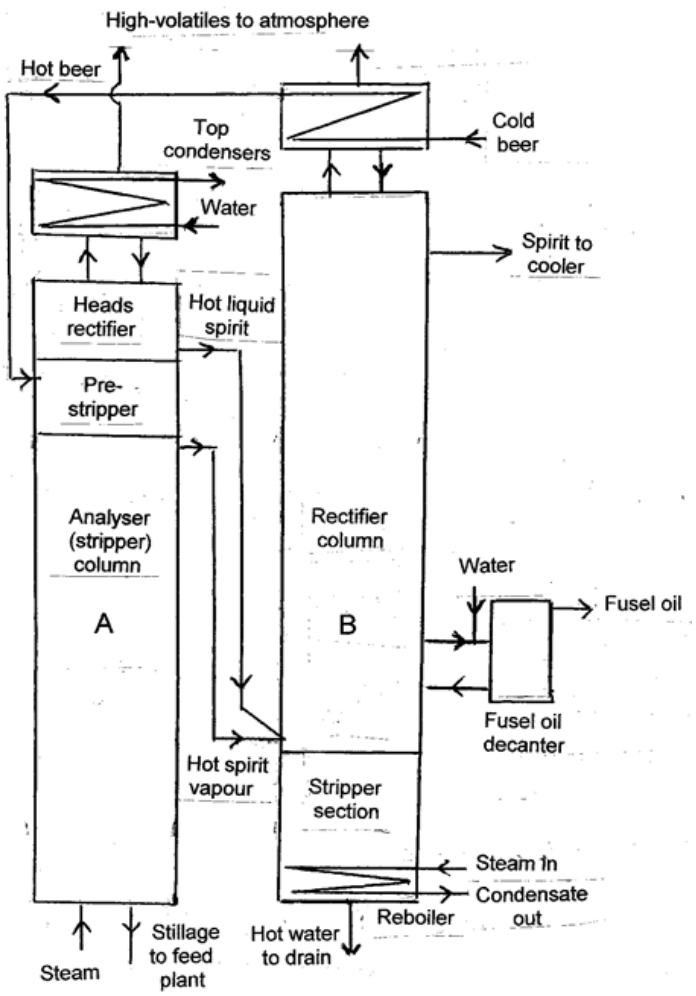

Figure 5 Barbet still.

Coffey Stills

Coffey stills (Figure 6) are mainly associated with whiskies of various nationalities, but are also used in some rum distilleries. The beer/wash is pre-heated in a copper coil throughout the height of the rectifier column B, so the temperature is close to bubble point as it is discharged into the analyser A. Condensation on the coil surface provides true reflux throughout the Coffey still rectifier, giving better rectification, and the additional surface area of copper increases reaction with sulphur compounds, but the disadvantage is more complicated construction and cleaning. Note that the top condenser of the rectifier is not involved in heating the wash. Its main purpose is to provide reflux to the column but it also provides hot water for the distillery. Figure 6 is shown with a fusel oil still rather than the

simple decanter system of Figures 2, but a fusel oil still could be equally well be fitted to the standard still of Figure 2.

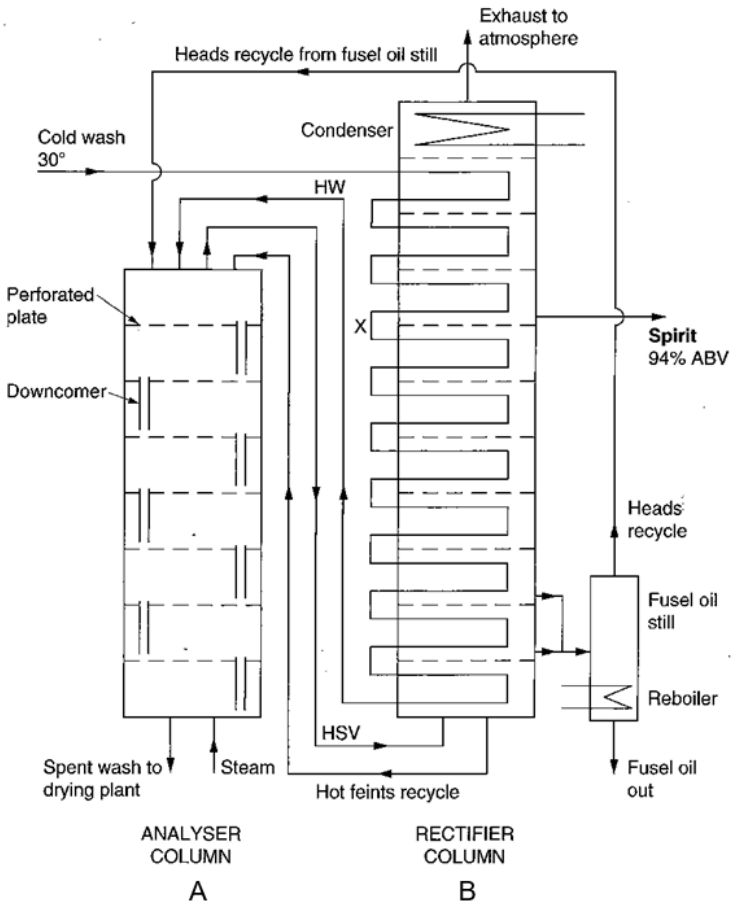

Figure 6 Coffey Still.

HSV = hot spirit vapour; HW = hot wash. X is the wash coil bend at the spirit plate where temperature is monitored to control wash feed rate. Within the rectifier the wash coils are on a horizontal plane. Only a few sieve plates are shown, but in reality each column has at least 30 plates.

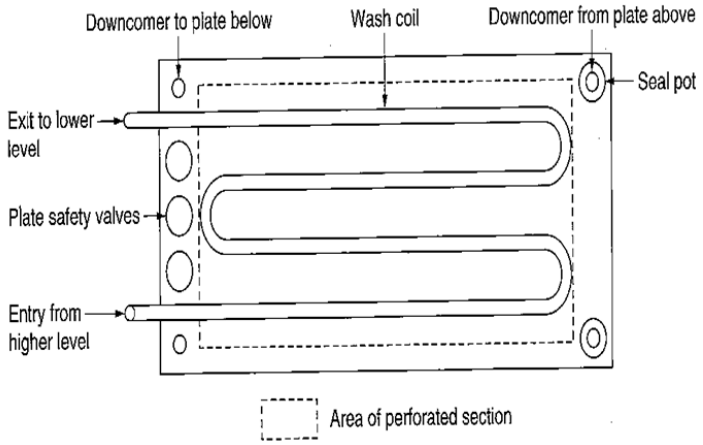

Figure 7 Coffey Still sieve plate.

The above rectangular sieve plate of a Coffey still showing the shape of the coil, which is supported above the 5-cm depth of liquid. On this particular plate, liquid flow is right to left. Except for their shape, and of course absence of a wash coil, circular plates of other types of still are of similar construction.

2.3.2 Continuous Still Operation

Distillation

The following description is based on the standard still system of Figure 2 but is also applicable in most respects to the operation of Barbet and Coffey stills.

Since the temperature rises to 30 - 34oC during fermentation, as an energy-saving measure the beer/wash should be distilled before any significant cooling occurs. It is common practice to discharge the beer/wash into a charger vessel of at least twice the capacity of the fermentation vessels. A mixer is required for three reasons::to ensure homogeneous alcohol content, to keep yeast and other solids in suspension, and to drive off as much as possible of the CO2 to reduce frothing in the still.

Constant alcoholic strength is required for stable conditions in the still. Although weaker beer/wash may be preferable for more stable

operation and optimum separation of flavour congeners, the potential energy savings from distillation of the strongest possible feed are now more important. However, stable operation becomes increasingly difficult above about 9% abv. The still can adjust itself to a change in the alcohol content during the run to a limited extent. Weaker beer/wash increases reflux, because of the increased amount of the less volatile compounds, particularly water. Stronger feed generates more ethanol, a decrease in reflux.and less fractionation of the various flavour congeners. In theory, increased alcohol content could be controlled by reducing the steam supply (as in pot stills, slower distillation means more reflux), but in practice that would probably not be possible, since the liquid would tend to drain through the holes in the plates. The still is designed for a specific upward flow rate of steam and spirit vapour, within narrow limits. Slightly larger holes are required in the analyser plates to prevent blockage if yeast and and other solids are intended to be present. With the constant flow rate of steam for the designed operating conditions, the rate of upward flow of vapour is sufficient to prevent all but a slight leak of liquid through the holes. In fact some “weeping” of liquid through the holes is said to be useful, to wash away any accumulation of solids and prevent blockage of the holes to allow a longer run. However, the still must eventually be stopped for cleaning, and to allow oxidative reactivation of copper surfaces.

Heated beer/wash is usually discharged into a tray or trough which overflows to flood the top plate of the analyser. For the necessary steady-state conditions it is more important to maintain a constant feed temperature than constant flow rate, but even so, substantial variation flow rate must be avoided. So the flow rate through the heat exchanger is adjusted as required to maintain the temperature of feed to the top of the analyzer at, say, 90°C. In the Coffey still, that feed temperature is controlled by maintaining constant temperature in the coil at the level of the spirit plate. The rate of the feed pump can be controlled automatically by a sensor at this point (X in Figure 7).

The hot vapour rising to the top of the analyser, containing all of the volatile components stripped from the beer/wash, flows through the vapour pipes to the base of the rectifier That spirit vapour, initially at the same temperature as the top plate of the analyser, is cooled as it rises up the rectifier, condensing and re-evaporating at each level. .Rising up the rectifier, the different components separate according to their volatility and boiling points. With the Coffey design of still, the temperature gradient is also partly due to the wash in the coil entering the system at 30 - 33° at the top and increasing to about 90° as the wash pipe leaves the bottom. But in general, evaporation of congeners is according to boiling point and relative volatility to the concentration of ethanol at each level, and the result is the least volatile components condense in the lower section; but highly volatile compounds remain in the vapour phase throughout the height of the rectifier.

Because of the 2-column design of the still, when the descending liquid phase of the rectifier column reaches the bottom plate, at about 15% abv, it must be pumped up to the feed plate of the analyser. Usually this is from a buffer tank (hot feints tank; see the final section of this unit) since the flow rate must be co-ordinated with the flow of fresh beer/ wash to maintain a constant alcohol concentration at the feed plate. And so a steady-state continuous process is maintained of this combined charge to the analyser cascading downwards, volatile components evaporating in the rising steam and vapour to pass to the rectifier as hot spirit vapour, and water and non-volatile liquid and solid components of the wash being removed at the base of the analyser as stillage/spent wash.

2.2.3 Ethanol Profiles & 2.2.4 Congener Distribution

Congener Distribution

Figure 8 is a modified version of a figure from the Batch Distillation unit, showing the relative volatility of type A and B congeners. In a continuous process, type C compounds, which are consistently less volatile than ethanol, either do not reach the rectifier column, or if they do, are returned to the analyser from the lowest rectifier plates in the hot feints. A similar effect occurs in the analyser, where the low-volatile type C congeners cascade down the column to leave eventually in the spent wash. Type A congeners, being more volatile at all concentrations of ethanol, migrate to the top of the column. According to the operating conditions of the top condenser, individual components of that mixture will either be vented to atmosphere or recycled in the reflux to the top plate of the rectifier. The situation with type B congeners is more complicated since their volatility is related to ethanol concentration, which varies over the height of the column. So type B compounds will accumulate at the level in the column where their volatility and that of ethanol are equal, and that is different for each compound.

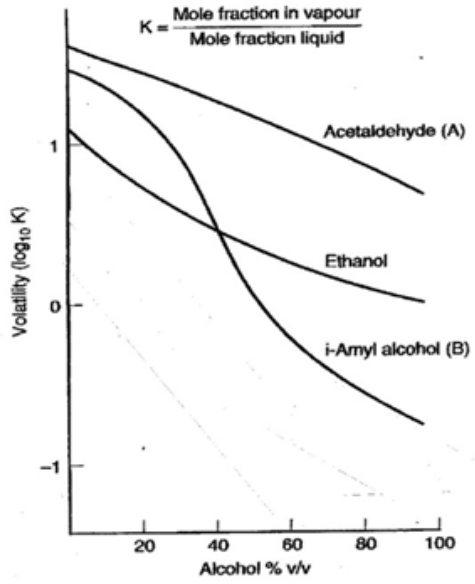

Figure 8 Relative volatility of flavour congeners.

Figure 9 shows the concentration of the principal alcohols, ethanol, n-propanol, butanols and amyl alcohols in the liquid on the individual plates of the rectifier. Each higher alcohol has a slightly different ethanol concentration where their volatilities are equal, i.e. a different level of the column. Of the butanols and amyl alcohols, only n-butanol and iso-amyl alcohol are shown, since they are present in greater amounts than their isomers, which have similar volatility.

The spirit plate is not necessarily the level with highest concentration of ethanol, since that may have an unacceptable level of type A congeners, e.g. acetaldehyde, methanol and any sulphur compounds which have escaped reaction with the copper in the system. The flavour of the spirit is more important than its strength, and ethanol rising to higher levels will find its way back to the spirit plate take-off point.

Note that concentrations of congeners are shown as percentages by volume in Figure 9, not the parts per million that would be present in the final spirit. They are present in considerable amount at their own level in the column, and congeners are still present at ppm levels and have a detectable effect on aroma for several plates above and below their disappearance from the graph.

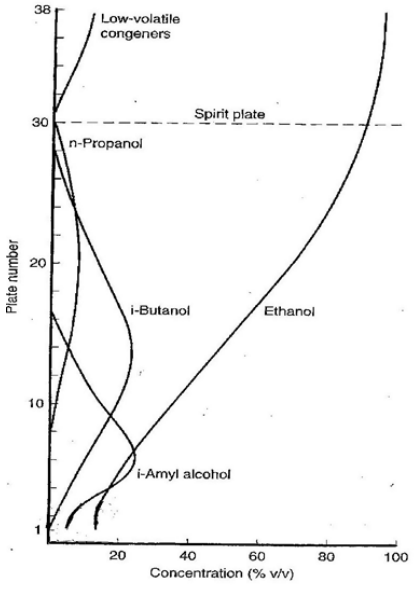

Figure 9 Congener profile in the rectifier column.

Although iso-amyl alcohol is the higher alcohol reaching the highest concentration in the column, all propanol, butanols and amyl alcohols must all be continuously drawn off at constant rate, otherwise they would gradually accumulate in the column and affect spirit quality over the duration of the run. Inspection of Figure 9 suggests that about plate 15, where all three overlap, they could be drawn off together. However, in practice it has been found preferable to operate several take-off points as feed for a fusel oil still. For steady-state conditions, these congeners must be drawn off at the same rate as their introduction in the wash, and this is most easily achieved as separate side streams. They can be adjusted independently, but cautiously, to control the congener content of the spirit if necessary. For example, to increase the butanol content, obviously less butanol should be drawn off as side stream, but in practice, the situation is more complex. If the previous rate removal of propanol is maintained, as the amount of butanols increases the propanol "bulge" in Figure 9 will be displaced upwards, increasing the amount of propanol at the level of the spirit plate. Therefore the rate of removal of propanol to the fusel oil still should be increased to maintain the original propanol content in the spirit. This effect will be enhanced if amyl alcohols are also removed more slowly.from lower levels, and so will further encourage the movement of butanols and propanol up the column. However, there are practical limits to this type of manipulation. Variation in alcohol content of the feed to the analyser also affects flavour, since the changes in alcohol strength over the height of the rectifier affect the levels at which the individual congeners of type B volatility accumulate.

In Japan, North America and European Union countries, whisk(e)y must be distilled at less than 94.8% abv (= 190° US proof), although Kentucky Bourbon whiskey has a much lower maximum, 80% abv (160° US proof), to produce stronger congener flavour. In Scotland, the spirit plate at which the spirit is drawn off is usually chosen to give an alcohol concentration at some precise point in the range 92 – 94%. Even that 2% difference has a detectable effect on flavour congeners. Spirit for gin and vodka production is collected from a higher plate, for least 96% abv.

Removal of Feints

In general, n-propanol, butanols and amyl alcohols are withdrawn continuously from the region of the maximum concentration to maintain constant conditions in the still. Ethanol is separated from the higher alcohols (fusel oil) either by a decanter or by distillation.

Fusel oil is a valuable co-product. UK Revenue & Customs permits up to 8.6% ethanol in fusel oil for sale, but that amount represents a wasteful loss from the system and itis usually possible to reduce the ethanol content to less than 1%.

A fusel oil still (Figure 10) is a continuous still in its own right and must operate under stable conditions, i.e. constant flow rate, temperature and composition of feed. Since it is only a small still, and of narrow diameter, its plates alone have insufficient area and reflux capability for efficient separation, which is achieved by packing the still with stainless steel or ceramic rings. Flavour is irrelevant here, so there is no need for copper and the still is built of stainless steel. The fusel oil still has an entirely liquid feed, so can be heated by a reboiler. As with any continuous still, part of the condensed top product must be recycled as reflux, but the remainder is returned to the main stills to recover the ethanol. For simplicity, the drawing of a Coffey still with associated fusel oil still in Figure 6 shows a direct return flow, but via the cold feints tank (see below) is more usual. The bottom product, mainly fusel oil and water, proceeds to the cooling and separation stages. Fusel oil, iso-amyl alcohol in particular, is only poorly soluble in the water phase and can be drawn off as the surface layer in the separator (decanter) tank. The lower water layer is of sufficiently low organic content (only traces of higher alcohols) to be

discharged to drain.

Although it is not as efficient as a fusel oil still for recovering ethanol, it is possible to operate with the decanter alone. A proportion of the liquid on the plate of maximum amyl alcohol concentration is drawn off and cooled by addition of water. The water and fusel oil phases separate; the fusel oil layer is decanted off and the alcoholic water phase is returned to a lower level of the rectifier column for recovery of its alcohol content.

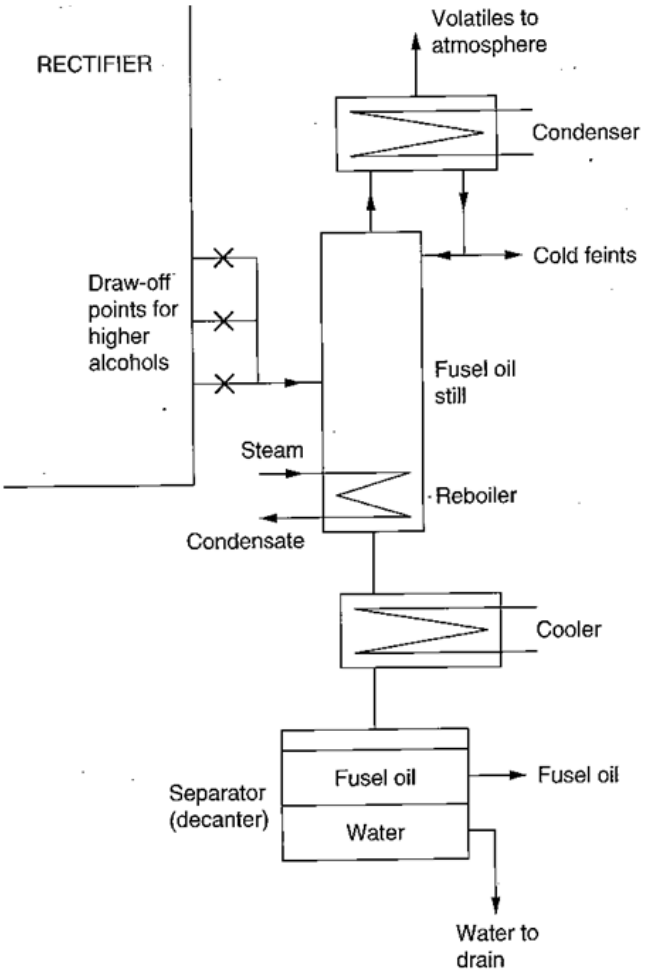

Figure 10 Fusel oil still.

Fusel oil contains up to 95% v/v amyl alcohols and small amounts of ethanol, propanol, butanols and various other metabolic products of fermentation. Although its removal obviously has a direct influence on flavour, there is the indirect benefit that high levels of iso-amyl alcohol inhibit the solution of copper, and therefore restrict the Cu-catalysed breakdown of ethyl carbamate precursors and other unwanted compounds. Also, high levels of iso-amyl alcohol in the lower section of the still inhibit the rectification of ethanol. Figure 10 shows the effect of accumulation of iso-amyl alcohol in the base of the still: the extent of condensation and re-evaporation of ethanol on the lowest plates is substantially reduced (the effect of relative volatility of amyl alcohols and ethanol as in Figure 8 again).

Figure 11 is copied from the lower section of Figure 9; but the different profile of part 12 shows that an increase in the concentration of amyl alcohol over the lower plates of the rectifier reduces the ethanol content. Although both ethanol and iso-amyl alcohol concentrations are normal at plate 12 and above, the over- all operation of the still is less efficient, since plates 1 – 11 have not been involved to the usual extent in rectification of the spirit. Therefore in several ways, removal of amyl alcohol contributes to a better quality of spirit.

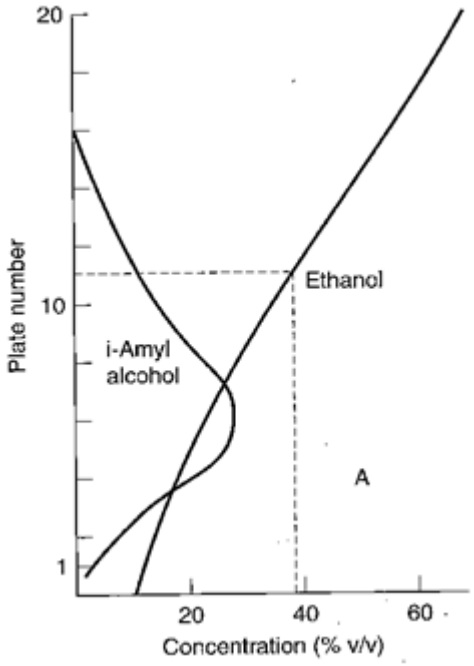

Figure 11 Effect of amyl alcohol concentration: normal operation of the lower section of the rectifier

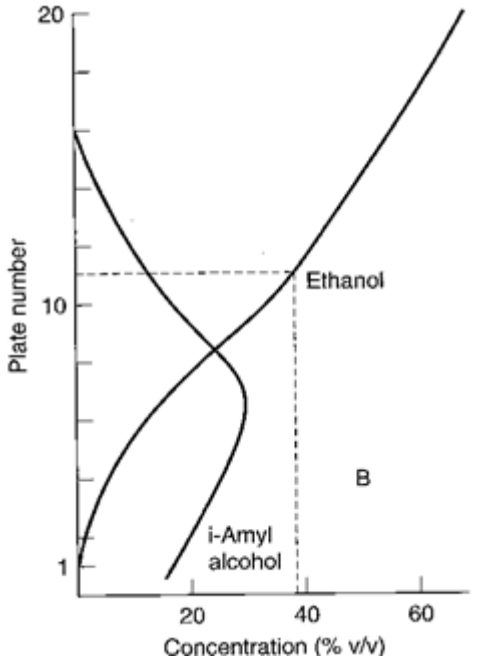

Figure 12 Effect of amyl alcohol concentration: restricted fractionation by high amyl alcohol concentration.

Aeration

With the current concern about levels of ethyl carbamate in distilled spirits it is now necessary to aerate the beer/wash to eliminate the cyanide precursors of ethyl carbamate. Also, aerated feed is more reactive with the copper surfaces of the stills, thus reducing the amount of sulphury off-flavour. Air is usually added at the pump from the charger vessel to the still. Throughout the still system, the dissolved oxygen encourages reactions between beer/wash and the copper of the plates (and coil, in a Coffey still), producing copper compounds or complexes which react with and remove sulphur congeners. The solution of Cu salts is particularly important at the bottom three plates of the rectifier, where the hot, aerobic (by air carried in the hot spirit vapour from the top of the analyser), acid (volatile fatty acids) conditions and relatively low concentrations of ethanol and fusel alcohols encourage removal of cyanides associated with ethyl carbamate production. Therefore unwanted congeners such as S compounds and cyanides are removed from the base of the column with the hot feints.

Unfortunately, aeration of the wash increases the rate of corrosion of the copper, and increases the copper content of animal feed co-products. But that is a price worth paying for good-quality spirit.

It is possible to pump the hot feints from the base of the rectifier directly to the top of the analyser column, but it may be preferable to aerate first, as shown in Figure 13, to evaporate the more volatile Cu complexes and break down the precursors of ethyl carbamate. Hot feints are only about 15% abv, but being at least 90°C, a valuable. amount of ethanol is stripped out by the air flow. That is condensed, cooled and recycled to the analyser as cold feints. However, during aeration at 90o there is no possibility of further solution of oxygen.

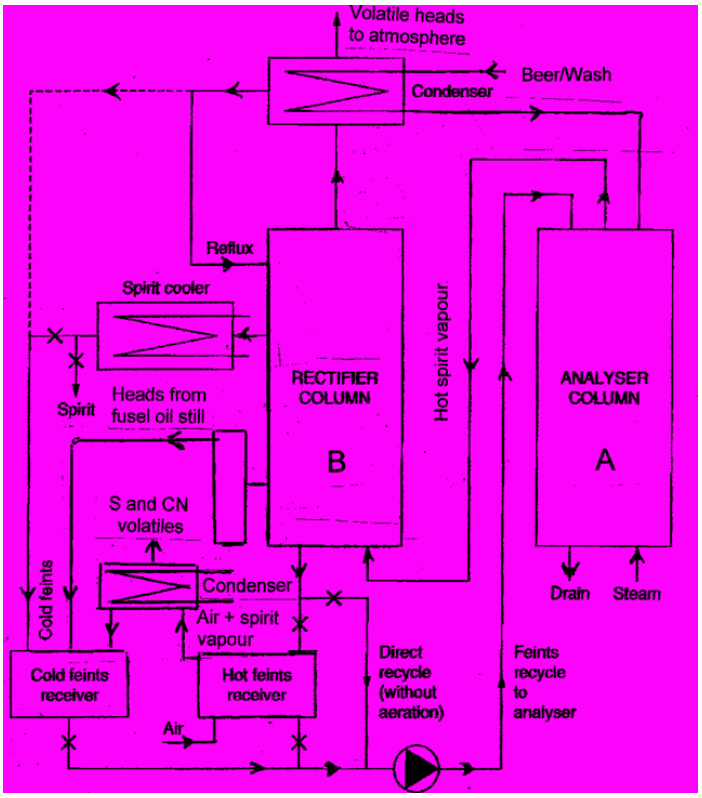

Figure 13 Hot feints and cold feints streams from continuous distillation.

Since cold feints were mentioned in the previous paragraph, it may be useful to summarise their sources at this point. Cold feints are about 20°C and of high alcohol content, about the same strength as spirit. Only small amounts are produced during

normal operation of the still, mainly the heads removed from the fusel oil still and the condensate from the condenser/cooler associated with aeration of hot feints. Normally all of the reflux from the top condenser is returned to the top plate of the rectifier, but f it is necessary to adjust the amount of reflux descending the rectifier column, the dotted line in Figure 13 represents the route for the excess which is diverted to the cold feints tank. The link shown between spirit collection and the cold feints system relates to the start-up and close-down procedures.

Summary of Continuous Still Operation

The relative volumes and typical alcohol concentrations throughout the process are summarised below.

Balance of continuous distillation from 7.5% abv.beer/wash (approximate values, since there is some variation between individual stills).

Analyser column

	UNITS IN			UNITS OUT	
	Volume	Ethanol		Volume	Ethanol
Beer/wash	100	7.5	Spent wash	103	0
Steam	12	0	Hot spirit vapour	20	9.4*
Cold feints recycle	1	0.9*	TOTAL	123	9.4
Hot feints recycle	10	1.0*			
TOTAL	123	9.4			

Rectifier column

	UNITS IN			UNITS OUT	
	Volume	Ethanol		Volume	Ethanol
Hot spirit vapour	20	9.4	Spirit	8	7.5
			Feints	2	0.9
			Condenser vent	< 0.1	< 0.1
			Hot feints recycle	10	1.0
			TOTAL	20	9.4

Fusel oil still

	UNITS IN			UNITS OUT	
	Volume	Ethanol		Volume	Ethanol
From rectifier	2	0.9	Cold feints	1	0.9
			Fusel oil product	1	0

*Cold feints: 1 unit of 0.9 = 90% abv. *Hot feints:10 units of 1.0 = 10% abv, Hot spirit vapour, 20 units of 9.4 = 5 x 9.4 = 47%.

The above table 1 is not strictly a mass balance since the quantities are expressed in volume rather than mass, but all units of ethanol are in the correct proportion to allow an estimate of the alcohol concentrations at different points of the system.

Stop and Start Procedures

Continuous stills should run for as long as possible, but usually after a few weeks' operation it is necessary to stop for one or more of the following reasons:

- The accumulation of solids on the analyser plates requires cleaning.
- Congeners may have accumulated to unacceptable levels.
- The internal copper surfaces of the still have become exhausted. Copper surfaces are oxidized and reactivated while the still is open to the air for cleaning.

Since a substantial amount of alcohol is in circulation in the system it is important to recover as much as possible during the close-down procedure:

- The feed is changed from beer/wash to water at the same temperature.
- From then on, the quality of spirit must be checked at frequent intervals. It will be acceptable quality for some time and then begin to deteriorate. At that stage, the flow is diverted to the cold feints tank, but at the same time the composition of the bottom product of the analyser must be checked as the amount of beer/wash material decreases. When sufficiently free of solids, it is run to drain rather than to the co- products plant.
- When no more cold feints can be collected, the steam supply is turned off.
- Then the contents of the rectifier column are drained into the hot feints tank, and stored to start the next run.
- Finally, the water supply to the condensers and coolers is turned off.

Similarly, the start-up routine must avoid any loss of the previously recovered alcohol. Although there will be some variation between distilleries, the procedure is basically one of two possible methods, starting with either hot feints or water. Hot feints, stored from the previous distillation and probably now cooled, is pumped at the normal flow rate and alcohol concentration of beer/wash, i.e. abv adjusted with water or cold feints as necessary, to the top of the analyser. From

there it provides feed for the analyser column and, evaporated by the steam supply, provides hot spirit vapour for the rectifier and its top condenser. Although the cold feints tank will also be fairly full at this stage, because of its high strength only the normal small proportion can be bled into the hot feints to supplement its alcohol content. When the temperature of the feints feed is steady at working temperature, it is replaced by beer/wash, and the hot feints feed is adjusted to its normal rate, which should then be kept constant during the run. When the first stillage appears at the bottom drain it is directed to the co-products plant. The distillate at the spirit plate is collected as cold feints until it meets quality specifications of spirit, or for a standard time that is known from previous experience to ensure collection of the desired quality of spirit.

In an alternative start-up procedure the still is heated to its working temperature with a water flow to the feed plate of the analyzer against the upward flow of steam, and the normal feed of beer/wash, hot feints and cold feints is started when the two columns and the top condenser reach working temperature. As before, alcohol is collected as cold feints until of spirit quality.

Because of its high alcohol content, any variation in addition of cold feints to the still creates unstable conditions. Only a relatively small quantity of cold feints is collected during normal operation. Most, or in some distilleries all, of the condensate from the top condenser is recycled as reflux to the rectifier plates, but the top product of the fusel oil still, with its high ethanol content, must be recycled as cold feints. Stored cold feints from the stop and start procedures is run on to the analyser column at a constant rate calculated to empty the cold feints tank over the planned duration of the run. However, if an excess accumulates it can be re-distilled in the same way as unacceptable spirit (see below).

Coffey stills

To start Coffey stills, water is pumped through the coil during the heating procedure at the normal feed rate for wash. Then the equivalent of the feints or water start-up procedures are either the hot water is drained from the bottom of the coil and hot feints are added to the top of the analyser or the hot water continues to the analyser and the still columns are heated up against the down-flow of that water. When normal working temperature is reached, the wash flow is turned on to replace water in the coil and the feed of supplementary hot and cold feints to the analyser is started.
When spent wash rather than water appears at the base of the analyser the flow is diverted to the processing plant. Again, spirit is collected as cold feints until of acceptable quality for maturation.

Spirit which fails to the meet chemical or nosing tests for quality must be re-distilled. Although an extremely rare event, when it happens the quantity may be too large for blending with normal feed as cold feints. In that case the still is run only on the failed spirit, diluted with warm water to the temperature and alcoholic strength of the normal beer/wash feed. Since the bottom product of the analyser is now water with trace amounts of congeners, it is run to drain after heat recovery, but otherwise the still operates normally until all of the faulty spirit has been re-distilled.

FURTHER READING

I.Campbell's chapter Grain Whisky Distillation in Whisky, Technology, Production and Marketing, ed. I. Russell (Academic Press, 2003) is mainly concerned with grain whisky distillation but most of the information is equally applicable to continuous distillation in general.

The equivalent chapter by R. J. Panek and A. R. Boucher in The Science and Technology of Whiskies, ed. J. R. Piggott et al. (Longman, 1989) may be more relevant to products other than Scotch grain whisky.