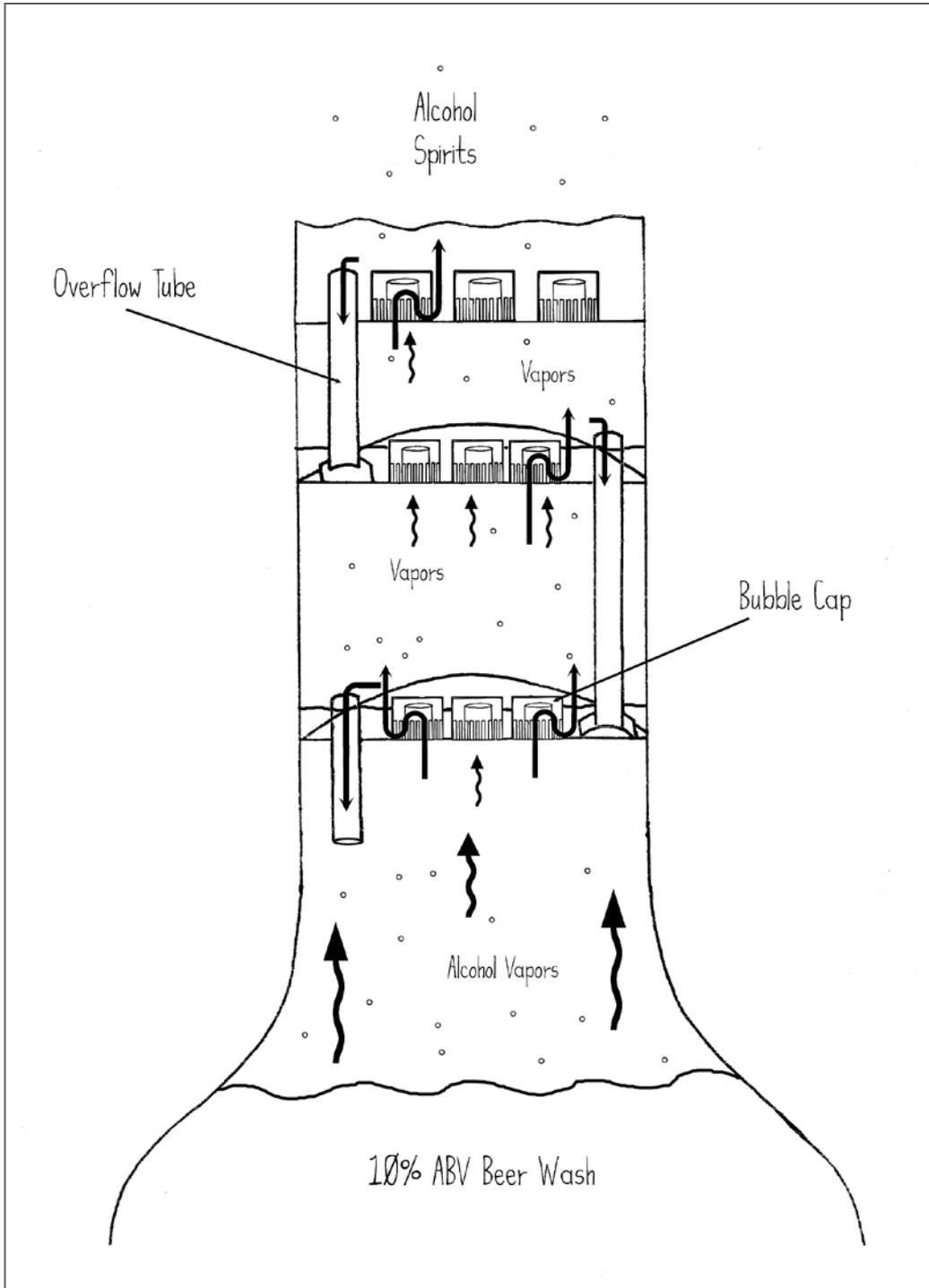


1

Distillation Principles



Most artisan stills are of the reflux or pot column design because of the inherent flexibility that they offer.

HOW DISTILLATION WORKS

Distillation is a physical process where compounds are separated by virtue of their different boiling points and vapor pressures.

The separation in distillation occurs when a mixture of compounds in the still is brought to boil. As a simplification, assume that the still contains only ethanol and water. Ignoring the azeotrope discussed below, for every mix ratio of ethanol to water there is one and only one new boiling point that lies between the boiling point of either. Conversely, for each boiling point, there is one and only one ratio of ethanol in the kettle, and an enriched ratio in the vapor and the distillate. If you know the temperature in the kettle, you can look up the exact ethanol ratio both in the kettle and in the vapor/distillate, in a simple table [figure 1/page 5].

Assume a mixture of 90% water and 10% ethanol (by volume) is to be separated by distillation. Water has a boiling point of 212°F and ethanol has a boiling point of 173°F, but this 10% ethanol mixture will boil at 197°F; it will not boil at 173°F. The vapor above the liquid will be 61% ethanol, as will the distillate. In a simple kettle, the ethanol percentage will drop during the boil because more ethanol than water is being removed, and neither are being replenished. This alone accounts for the increase in boiling point from start to finish—the ratio changes, so the BP changes.

Note that we started with 10% ethanol in the kettle, and now have a distillate at 61% ethanol, a 6-fold increase in strength. Referring to the table and graph below, if we now distill this condensate again, the new distillate will be 86%, and if we distill that, we will have a 91%, and again 92%, and again 93% and after six distillations, we may get 94%. As the concentration of the impurity (water) decreases, it becomes more difficult to remove. This

notion is very important for other products of fermentation in our wash. No matter what the concentration and boiling point of a given impurity, some of it will escape the kettle and find its way to the distillate throughout the distilling run. This means that head cuts can never be precise because these lighter impurities do not all vaporize before the hearts begin. Likewise, some tail impurities manage to vaporize well before they are expected. Compounds with boiling points between water and ethanol, such as diacetyl at 190°F may be impossible to remove by distillation. Therefore, distilling a bad wash never makes a good whiskey— and a good whiskey always starts with a good wash.

High-separation vodka stills employ a reflux column with many plates where vapors can condense, then like small kettles, re-vaporize the new enriched liquid, further enriching the vapor. Multiple cycles of condensation and re-boiling, one cycle per plate, occur in a single pass as vapors rise through the column before distillate is drawn from the still near the top. Even these stills can not enrich beyond 96.5% ABV because ethanol and water form an azeotrope where some mix ratios have a boiling point not between the boiling points of the constituents. This prevents complete distillation. Nonetheless, reflux columns attached to pot still can sharpen separation, making head and tail cuts easier, but most believe that this leads to a lesser whiskey because the cleaner separation strips away character. Artisan distillers want to preserve the character of their whiskey, so if a column is employed, the plates are opened to reduce reflux and more closely match the results from the neck and arm of a traditional whiskey still.

If your kettle temperature is 198.5°F then your kettle contains 9% ethanol and the vapor contains 59% ethanol. As the run goes on, ethanol is removed from the kettle, the kettle temperature rises toward 212°F and the vapor concentration decreases.

THE DIFFERENT TYPES OF STILLS

There are a number of different designs of stills. The most basic design is a “pot still,” with a pipe leading from the lid into a condenser coil. The condenser coil can either be long enough to air cool the vapors or it can be shorter and immersed in a water jacket. Such a still affords minimum separation since there is almost no separation of the vapors once they leave the boiler. Although this design of still is not suitable for producing beverage alcohol by modern standards, it will still concentrate an 8 or 10% abv wash to 60% in a fairly fast run.

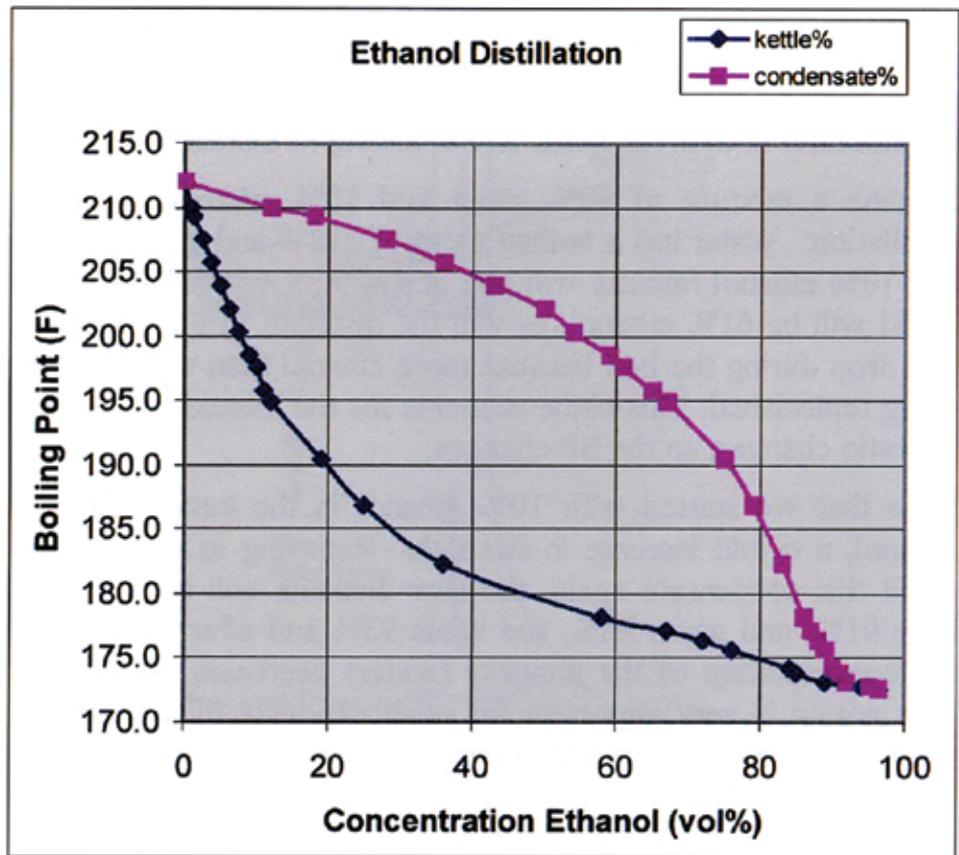
The next type of still is the “whiskey still,” sometimes called a “gooseneck still.” This design is technically a form of pot still and has been in use for centuries for commercial whiskey production, and is just as popular today in modern whiskey distilleries as it has ever been. A whiskey still has a large boiler with a long broad neck rising from it. The neck bends at the top and leads to a condenser coil immersed in water. This design is very similar to the crude pot still, except the tall broad neck affords enough separation to hold back most of the fusel alcohols from the distillate while retaining the desired flavors in the finished spirit. They are suited to the production of whiskey, brandy, rum, schnapps, and

other non-neutral spirits, for which they are very widely used commercially. However, the whiskey still is not suitable for the production of vodka, gin, or other spirits derived from neutral alcohol, which requires a high-separation still capable of producing pure ethanol.

This brings us to the high-separation still design called a “column still” or a “fractionating still.” A fractionating still is used to produce pure ethanol by fractional distillation for vodka and gin, or for pharmaceutical and laboratory use.

[Figure 1] This is a table and graph of boiling points for ethanol/water mixtures by volume. (Hint— copy this and paste it onto your still).

BP (F)	kettle%	cond%
212.0	0	0
210.0	0.87	12
209.3	1.25	18
207.5	2.5	28
205.7	3.75	36
203.9	5	43
202.1	6.25	50
200.3	7.5	54
198.5	9	59
197.6	10	61
195.8	11	65
194.9	12	67
190.4	19	75
186.8	25	79
182.3	36	83
178.2	58	86
177.1	67	87
176.4	72	88
175.6	76	89
174.2	84	90
173.8	85	91
173.1	89	92
172.8	94	95
172.6	96.5	96.5



In smaller fractionating stills the vapors emerging from the boiling mixture pass up a column packed with small pieces of glass, ceramic, stainless steel, copper or other material, inert to the process. This material is called the “column packing.” In larger fractionating stills, the columns have baffle plates with holes or bubble caps instead of packing material. Each piece of packing, or bubble cap, can hold a small amount of liquid, either internally (if they have internal crevices) or in the interstices between adjacent particles. At the top of the column, the emerging vapor is condensed into a liquid by means of a heat exchanger with cold water running through it. The condensed liquid runs back down the column until it reaches the boiler where it is reheated, converted into vapor once more, and once again moves up the column.

At equilibrium, which takes about an hour to achieve in the case of pure-ethanol production, the system consists of vapor rising up the column meeting a flow of liquid running down the column from the heat exchanger. At each vapor-liquid interface on the packing material within the column, a partial separation occurs wherein the more volatile components of the mixture go into the vapor phase and rise while the less volatile components go into the liquid phase and are carried down toward the boiler. At equilibrium, the various components in the mixture become stacked up in the column in the order of their boiling points, the most volatile at the top and the least volatile at the bottom.

There’s a variation of fractionating still called the “continuous-run still.” With the continuous-run design of fractionating still, the fermented wash is fed into a small boiling chamber from a reservoir and is vaporized immediately upon entry to the chamber. The different components of the mixture are drawn off at various heights along the column, and the spent residue is drained off at the bottom. This process can continue indefinitely as long as fermented wash is fed into the boiling chamber. Acetone, for example, would be continuously drawn off from the top of the column while ethanol would be continuously drawn off from a point a little further down.

The last still design to be discussed in this text is the “reflux still.” A reflux still is very similar in design to the fractionating still except it doesn’t have a heat exchanger at the top of the column forcing a complete condensation of all the vapor that reaches the top. It has a packed column like a fractionating still, but the vapor that reaches the top exits to the condenser and is received as output. While a reflux still benefits by the purifying process of the redistilling at the packing surfaces like a fractionating still does, without the forced reflux of a top heat exchanger it doesn’t produce as pure a neutral ethanol as a fractionating still. However, reflux stills are very commonly used in the artisan distillation of whiskey, and other non-neutral spirits, and it’s this type of still that will be discussed in the rest of this text.

Most artisan stills are of the reflux or pot column design because of the inherent flexibility that they offer. The best known manufacturers of pot and reflux stills are: Vendome, Holstein, Christian Carl, and Forsyth. The Index contains a complete list of manufactur-



ers. These brands of still come with multiple bubble-cap trays, and each tray can be bypassed by operating a lever. These stills can also be purchased with a dephlegmator, which is a chilling apparatus at the top of the column comprised of a bank of tubes with cold water running through them to increase the reflux, and therefore the purity of the distillate. The still can be operated with the dephlegmator disabled, or with cold water running through it at whatever rate the operator chooses. Between the variability of the dephlegmator and the ability to bypass, or not, the multiple bubble-cap trays, just about any level of separation can be achieved with these artisan stills. That's why they make such excellent whiskey stills.

THE FLAVOR OF SHAPE

The whiskey still has four parts: pot, swan neck, lyne arm, and condenser. The shape of each section affects rectification and the taste of the spirits. The pot is heated (to 172°). It can be heated by direct fire, steam, gas, or wood. All systems have advantages and disadvantages. There is no right way to heat wash. Most manufacturers, however, prefer a double-jacketed steam-water system that provides a gentle heat to the wash. Mainly, you don't want to burn the wash. Most pots have a sight glass so the distiller can check for foaming during the distillation process.

- The swan neck sits on top of the pot. It can be tall, short, straight, or tapered. Often the swan neck is connected to the pot via an ogee, a bubble-shaped chamber. The ogee allows the distillate to expand, condense, and fall back into the pot during distillation. Most pot stills have a tapered swan neck, allowing for better separation and better enriching of the spirits during distilling.
- The lyne arm sits on top of the swan neck. It can be tilted up or down, and it can be tapered or straight. Most arms are tapered down. Often pot stills are fitted with a dephlegmator, or what the Scots call a purifier. The dephlegmator is fitted with baffles that use water plates or tubes to cool the distillate, sending 90 percent of it back to the pot. Its main purpose is the enrichment of the spirits before they're sent on to the condenser.
- The condenser, or worm is used for cooling the spirits and providing a small stream to a collection tank or pail.

DISTILLATION PROCESS

In distilling parlance, the compounds in the wash that are not ethanol or water are called congeners. Some congeners such as acetaldehyde, methanol, and certain esters and aldehydes, have boiling points lower than ethanol, while certain other esters, the higher alcohols (fusel alcohols), and water, have higher boiling points than ethanol. This means the lower-boiling-point congeners come out in high concentration at the beginning of the distillation run, and the higher-boiling-point ones come out in high concentration towards the end of the run, leaving the ethanol as the most abundant compound during the middle of the run.



So, when distillation takes place in an artisan still, such as the reflux stills discussed above, the distillate that comes out is divided into three phases called: heads, hearts and, tails. The heads contain the unwanted lower-boiling-point congeners that come out at the beginning of the run, and the tails contain the unwanted higher-boiling-point congeners that come out at the end of the run. And, the hearts are the desired spirit.

Since whiskey is not distilled at a high-separation level, it means that each phase bleeds into the adjacent phase. That is to say, there's a considerable amount of ethanol in the heads phase, and there are late heads congeners at the beginning of the hearts phase. Similarly, there 's a significant amount of early tails congeners at the end of the hearts, and there 's a considerable amount of ethanol in the tails phase. The hearts are the whiskey, and while they are comprised mostly of ethanol and water, they have a delicate balance of late-heads and early-tails congeners that make up the flavor profile of the whiskey.

Since both the heads and the tails contain a lot of ethanol and residual desirable flavor, they are mixed together and saved for future recovery. The heads and tails when mixed together are called feints. They can be distilled separately to produce another whiskey run, or they can be mixed in with a future spirit run, where their ethanol and flavors are recovered as a part of that run. However, each subsequent distillation produces its own set of heads, hearts, and tails, and the feints from those runs are also saved for future recovery.

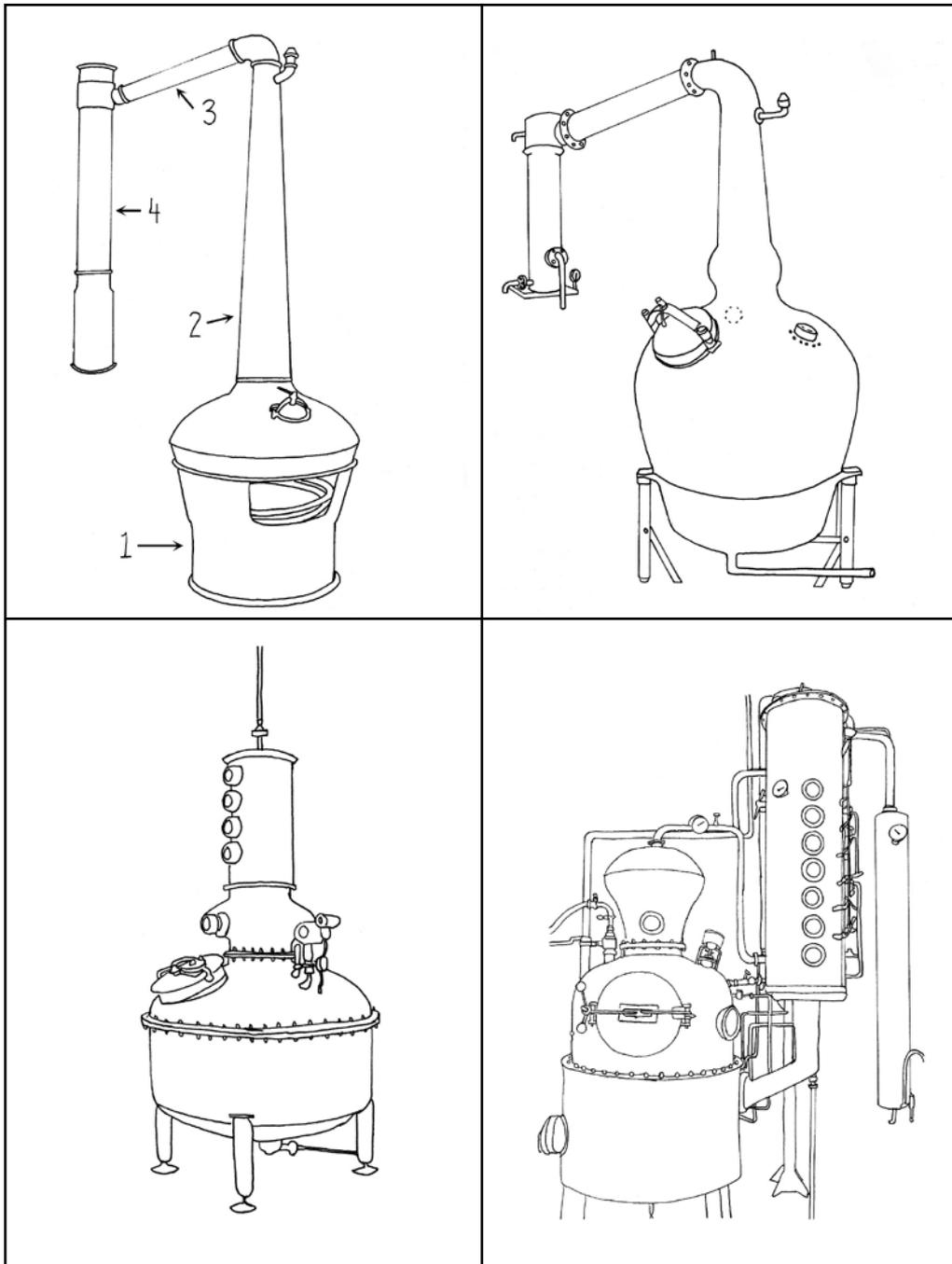
When whiskey is made, it's usually done in two distillation runs: a beer-stripping run; and, a spirit run. The beer-stripping run is generally done in a larger, high-volume pot still called a beer stripper. The beer stripper is used to distill the fermented mash and concentrate the ethanol and all the impurities into a distillate of about 35% ethanol, called low-wine. The spirit-run is done in a smaller whiskey still, either a gooseneck or an artisan reflux still, called a "spirit still." The spirit still is used to distill the low-wine and refine them into the finished spirit. There are the two outputs retained from the spirit-run: the finished spirit; and, the feints

For a beer-stripping run, the fermented mash, which is typically about 8% abv, is loaded into the beer-stripper, and the contents are brought to boil. Since this run is just a primary distillation, the heads, hearts, and tails are not separated out. The entire output from this run is collected in a single lot, and the run is continued until the aggregate percent alcohol is down to 35% abv. This distillate is the low-wine, which is the input to the spirit run.

To produce the finished whiskey, the spirit still is filled with the low-wine from the beer-stripping run, and often a measure of feints from previous spirit-runs. The spirit still is then brought to boil. It is with the spirit run that the distiller adjusts the boil-up rate to achieve a gentle, slow flow of distillate and carefully separates out the heads, hearts, and tails.

Some whiskey distilleries produce their whiskey in one single distillation. In effect, they do a spirit run directly from the wash. The artisan reflux stills discussed above are excellently suited to doing this type of whiskey distillation, but operationally it's very labor in-

POT STILL CONSTRUCTION



tensive and a lot of attention must be paid by the distiller to numerous smaller runs rather than one larger run.

Some people find the whiskey from a single distillation run to be richer and to have a more natural flavor, while others find it to be harsh and unrefined. In this text, the more-common double-distillation method is used.

MAKING THE CUTS

Probably the most elusive part of the distilling process for making whiskey, is making the cuts from heads to hearts and then to tails. Making a cut from one phase to the next is the point where the distiller switches the output so that it is collected in a different receiver than the previous phase. At the end of the spirit run, the heads will be in one container, the hearts in another, and the tails in a third one. The question is, when to switch from one phase to the next?

Experienced distillers do this by taste. Even though there are measurable parameters like still-head temperature and percent alcohol of the evolving spirit that can be used to judge when to make the cuts, taste and smell still remain the most reliable method of determining them.

The empirical parameters for judging the cuts are: the percent alcohol of the spirit that's flowing out of the still (i.e. the evolving spirit); and, the still-head temperature. However, these vary from one still to the next, and vary based on the properties of the low-wine (e.g., percent alcohol, and quantity). It is possible to develop a consistent process using the same still and the same quantity and a formulation of low-wine, such that the parameters remain the same for each run. For example, if a spirit run is being done in an artisan reflux still with low-wine that is 35% abv, the begin-cut (i.e. the cut from heads to hearts) is usually done when the evolving distillate is at about 80% and when the still-head temperature is about 180 degrees. And, the end-cut (i.e., the cut from hearts to tails) is often done at about 65% and when the still-head temperature is about 200 degrees. However, a spirit distilled from a straight malt wash, can often be end-cut as low as 60%. It's because of these nuances that smell and taste become the only truly reliable indicators of when to make the cuts.

When making the begin-cut, the taste characteristics that the distiller is looking for are as follows. When a spirit run comes to boil and the first distillate starts flowing from the still, this is the beginning of the heads phase. The distiller can collect a small sample of the distillate on a spoon or in a wine glass and smell it. At this stage, the distillate will have the sickening smell of solvents like nail polish remover or paintbrush cleaner. However, before long this solvent smell will diminish, and even when a sample is tasted these compounds will be very faint. As the solvent character disappears completely, the distillate will start to take on a hint of whiskey flavor. This flavor will increase until it becomes very pronounced and highly concentrated. It's when this flavor is clearly evident (i.e., more than just a hint) but is still increasing in intensity that the distiller cuts to the hearts phase.

To make the end-cut the distiller needs to monitor the flavor of the hearts through the following changes in taste. At the beginning of the hearts phase, the intensity of the whiskey flavor will still be increasing, and will continue to do so until it becomes very strong. However, as the hearts continue, the intense whiskey flavor will fade into a smooth, sweet, pleasant flavor that will persist for most of the hearts. The flavor will change slightly as the hearts progress but it will remain sweet and pleasant. Towards the end of the hearts, the flavor will start losing its sweetness and a trace of harsh bitterness will begin to appear in the flavour. This harsh, bitter flavor is the onset of the tails. While a small amount of this bitterness is considered to contribute to the “bite” character of the whiskey, the distiller should cut to the tails receiver before much of it is allowed to enter the hearts.

The tails can be collected until the evolving distillate is down to about 10% and the still-head temperature is about 210 degrees. The reason for doing this is to render all the residual alcohol that’s left in the still at the end of the hearts phase. This alcohol can then be recovered in a future spirit run.

The tails phase starts out bitter, and the bitterness becomes more intense as the tails continue, but as the tails progress, the bitterness subsides and gives way to a sweet-tasting water. This sweet water is called “backins.”