

Distilled Spirits (Whisky, Rum, Gin, Vodka and Brandy)

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1. Introduction

The distilled alcoholic or spirit beverages are prepared by the fermentation of sugars derived directly from plant extracts and fruit juices, or indirectly from hydrolysed grain and root starch. These are potable products whose alcohol contents are increased by distillation. In the process of distillation volatile materials emerging directly from the fermented substrate or after microbial (especially yeast) metabolism introduce materials which have a great influence on the nature of beverage. The character of the beverage is also influenced by such post-distillation processes as ageing, blending, etc.

In English-speaking countries, such as the United States, Canada, the United Kingdom, and Australia, the alcoholic content of spirit beverages is given by the term 'proof'.

Percentage by weight is used in Germany for the ethanol content of a beverage or other liquid. Percentage by volume is used in many countries especially in Europe use the percentage by volume system.

In general the following steps are involved in the preparation of the distilled spirits. The details may differ slightly according to beverage.

(i) Preparation of the medium: In the grain beverages (whisky, vodka, gin) the grain starch is hydrolyzed to sugars with microbial enzymes or with the enzymes of barley malt. In others no hydrolysis is necessary as sugars are present in the fermenting substrate as in brandy (grape sugar) and rum (cane sugar).

(ii) Propagation of yeast inoculum: Large distilleries produce hundreds of liters of spirits daily for which fermentation broths many more times in volume are required. These broths are inoculated with up to 5% (v/v) of thick yeast broth. Although yeast is re-used there is still a need for regular inocula. In general the inocula are made of selected alcohol-tolerant yeast strains usually *S. cerevisiae* grown aerobically with agitation and in a molasses base. Progressively larger volumes of culture may be developed before the desired volume is attained.

(iii) Fermentation: When the nitrogen content of the medium is insufficient, nitrogen is added usually in the form of an ammonium salt. As in all alcohol fermentations the heat released must be reduced by cooling and temperatures are generally not permitted to exceed 35-37°C. The pH is usually in the range 4.5-4.7, when the buffering capacity of the medium is high. Higher pH values tend to lead to higher glycerol formation. When the buffering capacity is lower, the initial pH is 5.5 but this usually falls to about 3.5. During the fermentation contaminations can have serious effects on the process: sugars are used up leading to reduced yields; metabolic products from the contaminants may not only alter the flavor of the finished product, but metabolites such as acids affect the function of the yeast. The most important contaminants in distilling industries are lactic acid which affects the flavor of the product.

(iv) Distillation: Distillation is the separation of more volatile materials from less volatile ones by a process of vaporization and condensation. Three systems used in spirit distillation will be discussed.

(a) Rectifying Stills: If the condensate is repeatedly distilled, the successive distillates will contain components which are more and more volatile. The process of repeated distillation is known as *rectification*.

(b) Pot Still: These are traditional stills, usually made of copper. They are spherical at the lower-portion which is connected to a cooling coil. They are operated batchwise. Malt whisky, rum, and brandy are made in the pot still.

(c) Coffey (patent) Still: The Coffey still was patented in 1830 and the various modifications since then have not added much to the original design of the still. Its main feature is that it has a rectifying column besides the wash column in which the beer is first distilled.

(v) Maturation: Some of the distilled alcoholic beverages are aged for some years, often prescribed by legislation.

(vi) Blending: Before packaging, samples of various batches of different types of a given beverage are blended together to develop a particular aroma.

2. Whisky

Whisky is the alcoholic beverage derived from the distillation of fermented cereal. Various types of whiskies are produced; they differ principally in the cereal used. Although many countries including Japan and Australia now produce whisky for export, the countries best associated with whisky are first and foremost, Scotland followed by Ireland, USA and Canada.

The major cereals used for the manufacture of whisky are barley, wheat, rye and corn (maize). Malted barley is employed as a source of flavour and enzymes, which are not only responsible for converting the barley starch but also that of adjuncts to fermentable sugars. The main analytical criteria for whisky malts are their diastatic power, α -amylase and extract, especially when they are being used alongside adjunct. The malts may be 'peated', that is, flavoured with the smoke from peat burnt on the kiln. Such malts are classified on their content of phenols.

Rye (*Secale montanum*) is quite widely used in Eastern Europe and former USSR (Union of Soviet socialist Republics), and is sometimes malted. Wheat (*Triticum vulgare*) has largely replaced corn in Scotch grain whiskies as the cost of importing grain from the United States became prohibitive and it is also used in some American whiskies. However, in the United States, corn (*Zea mays*) is especially widely used.

For malt whisky, mashes of water : grist ratio of 4 : 1 will be mixed in at 64.5°C, the malt having been broken in a roller mill. Although modern malt distilleries are changing over to the use of lauter tun technology, traditional distillery mash tuns feature rotating paddles to mix the mash and these will be employed for approximately 20 min before allowing the mash to stand for an hour.

Fermentation of whisky was formerly performed widely with the surplus yeast generated in brewery fermentations. However, specific strains particularly suited to whisky production have been developed and these are supplied by yeast manufacturers in bulk for commercial

use. Hybrids emerged not only from the ale strain *Saccharomyces cerevisiae* but also from the 'wild yeast' *Saccharomyces diastaticus*, which produces a spectrum of enzymes fully capable of hydrolysing starch to fermentable sugar. Thus, the distilling strains enable high alcohol yield. The strains may also be selected on the basis of their ability to produce esters.

Fermentation on a small scale may be in closed wooden barrels, but on a larger scale, it will be in stainless steel vessels known as washbacks. Unlike in breweries, there is little temperature control during fermentation, other than to target the initial temperature, which may typically be in the range 19–22°C. The temperature may go as high as 34°C during fermentation, hence the need for ale-based strains rather than lager-based ones. Typically the fermentation is complete within 40–48 h. Some advocate holding a few hours prior to distillation in order to ensure that the endogenous lactic acid bacteria have an opportunity to enhance flavour.

Whiskey variants

Bourbon (United States) is made principally from corn (maize) plus added rye and barley and is aged in charred barrels. A close relative is *Tennessee whiskey* (United States), which is produced using a sour mash process. *Canadian whisky* (Canada) is a light product from rye and malted rye, with some corn and malted barley. *Corn whiskey* (United States) is from maize and is aged in barrels that have not been charred. *Rye whiskey* (United States) is from rye mixed with corn and barley and is aged in newly charred oak barrels.

3. Rum

Rum primarily originated in the Caribbean, although the first references to liqueurs obtained from sugar cane are from India. Sugar cane was introduced to the Caribbean by Christopher Columbus in 1493. The chief producing countries are Barbados and Santo Domingo. Nowadays the coastal planes of Guyana (Demerara) are rich in estates producing sugar cane (*Saccharum officinarum*).

At harvest time the fields of sugar cane are set alight in order to sanitise the soil, the stems are scorched in this process and the canes subsequently wither and are harvested by machete, a strategy thought to yield a superior product when compared with rum made from cane harvested by machine. The canes are topped to remove the leafy parts and the cane then ferried to mills. There is considerable contamination with *Leuconostoc mesenteroides*, which produces a gum that causes problems during extraction. It is important to avoid delays between cutting and milling, and the maximum time elapse should be less than 24 h. During processing, the canes are cut and crushed and the juice limed, clarified and evaporated. Various fractions are generated, but the key product for rum is molasses. Four to five tons of molasses are typically obtained per 100 tons cane.

The nature of the molasses depends on cane variety, soil type, climate, cultivation and harvesting conditions. They are delivered hot to the distillery either by pipe or by tanker and are stored at 45°C. The molasses are pumped at 85–88°Brix and are mixed with water in line. Lighter flavour rums may incorporate cane juice (12–16% w/v sucrose).

Formerly adventitious yeasts were used to effect fermentation, but nowadays pure cultures of *S. cerevisiae*, *S. bayanus* and *Schizosaccharomyces pombe* are used. They are propagated from slopes by successively scaled up incubations using sucrose as the carbon source.

Prior to fermentation, the molasses are diluted to 45°Brix and their temperature elevated to 70°C in order to destroy contaminating organisms. The pH is lowered by the addition of sulphuric acid and the whole clarified by putting into a conical-bottomed settling tank, from which the sludge can be decanted from the cone. Ammonium sulphate is added as a source of nitrogen. Fermentation is conducted at 30–33°C in cylindroconical vessels that may be closed or open. The final sugar content will be 16–20°Brix (Brix (°Bx) is defined as the percentage of sugar by weight in a solution) and this is reached in 24 h with an alcohol yield of 5–7% Alcohol By Volume (ABV). Some high-gravity fermentations nowadays furnish 10–13% ABV.

Distillation is conducted in pot stills that were traditionally of copper or wood but now more likely to be fabricated from stainless steel. As for whisky, there are also column stills of stainless steel or copper (Coffey stills). Pot stills afford heavier rums that need prolonged maturation, whereas the column stills are employed for lighter rums, or to generate the neutral spirits that can be used for the production of gin and vodka. Distillates are collected at 80–94% ABV for rums and >96% for neutral spirits.

Pot distillation of rum is exactly analogous to the techniques used in the production of whisky. The pot is charged with wash at approximately 5.5% ABV and the retort charged with low wines at 51–52% ABV from the previous distillation. The fractions obtained are heads, spirits, and feints. The heads are rich in esters and are collected for the initial 5 min in the low wines receiver. The ensuing spirits are collected for 1.5–2 h at 85% ABV. When the emerging strength drops to 43% ABV, the flow is again diverted to the low wines receiver in order to collect the feints. Distillation is completed when the distillate approaches some 1% ABV. Column distillation allows ten times more output than does pot distillation and is performed exactly analogously to the whisky process.

Rum is aged in Bourbon oak casks. It is racked at 83–85% ABV. As the main production locale is tropical, ageing is quite rapid and may be complete within 6 months. There may first have been a blending of light rums produced in column stills with heavier rums out of pots. Furthermore, there may be transfers between casks for successive maturation periods. Finally rum is chilled to –10°C and filtered to remove fatty acid esters prior to dilution to final strength and packaging.

4. Gin and Vodka

In *gin* production, the grain-spirits (ie., without the congeners) are distilled over juniper berries (*Juniperus communis*), dried angelica roots (*Angelica officinalis*) and others including citrus peels, cinnamon, nutmeg, etc. Russian *vodka* is produced from rye spirit, which is passed over specially activated wood charcoal. In other countries it is sometimes produced from potatoes or molasses. *Schnapps* are gin flavored with herbs.

Let us discuss gin and vodka in brief in this subunit.

Gin

The word gin is a corruption of *genievre*, the French word for juniper. Distilled gin is produced by distilling neutral alcohol and water in the presence of botanicals, of which

juniper, coriander and angelica are key ingredients. The product is diluted further with alcohol and finally brought to its final strength with water.

In the European Union, a drink can be called gin if it is produced by addition to ethanol (of agricultural origin) natural (or nature-identical) flavourants such that the taste is predominantly one of juniper. 'Compounded gin' is made by adding essences to ethanol and this can not be called gin. The alcohol for gin may come from grain-, molasses-, potato-, grape- or whey-based fermentations.

The prime traditional flavourants are the juniper berry (*Juniperus communis*), coriander seed (*Coriandrum sativum*) and Angelica (*Archangelicum officinalis*), together with the peel of orange and lemon.

Other materials may also be used in the formulation of gins and these include cassia bark, cubeb beris, liquorice, orris, almonds and grains of paradise. Water quality is critical for the production of gin and, as for beer, this explains the traditional locales where the drink was first made and became popular.

These days, as for beer, water purification and salt adjustment protocols mean that the production region is of no significance. Gin is produced in copper pot stills similar to those used in the production of whisky. Nowadays they tend to be steam-heated rather than direct fired. The still is charged with water prior to adding alcohol to the desired concentration which is typically 60% ABV. The botanics are added either loose or suspended in a bag. The still is closed and heated.

The 'heads' emerge first, followed by the main fraction, of some 80% ABV, which is collected as gin. The 'tails' comprise the later fractions in which alcohol concentration is falling. They are collected with maximum heating and are combined with the heads as 'feints' to be purified in a separate distillation or alternatively sent to the alcohol supplier.

Sloe gin is produced by steeping berries of the sloe (*Prunus spinosa*) in gin. The mix is sweetened with sugar, filtered and bottled. Nowadays flavourants may be employed in place of the berries.

Vodka

Vodka comprises pure unaged spirit distilled from alcoholic matrices of various origins and usually filtered through charcoal. It is defined in the EU as a: spirit drink produced by either rectifying ethyl alcohol of agricultural origin or filtering it through activated charcoal. Materials added in the production of vodka include sugar at up to 2 g/L and citric acid at up to 150 mg/L. Some vodkas have glycerol or propylene glycol added to enhance the mouth feel. Amongst the flavoured vodkas are ones infused with pepper, a Polish product in which buffalo grass is steeped in the spirit and a Russian variant in which the vodka is treated with apple and pear tree leaves, brandy and port.

The neutral alcohol base is frequently produced quite separately from the vodka, perhaps by a different company. It is chiefly produced from cereals (e.g. corn, wheat) but other sources of fermentable carbohydrate include beet and molasses in Western countries, cane sugar in South America and Africa, and potatoes in Poland and Russia.

The fermentation is, of course, effected by *Saccharomyces cerevisiae*, notably distillers' strains. The alcohol is purified and concentrated by continuous stills with 2–5 columns. The first of these is a 'wash column' that separates alcohol from the wash. The second major column is the 'rectifier' in which alcohol is concentrated. There may be a 'purifier' between the wash column and the final rectifier.

The wash column distillate is introduced halfway up the extractive distillation column and water (approximately 20 times more than wash) is fed in at the top. This procedure impacts the volatilisation of components of the wash and encourages the removal of volatiles. Ethanol mostly leaves with water at the base of the column, prior to concentration in the final rectification column. Treatment with activated carbon is either by using a dispersion of purified charcoal in a tank prior to its removal by filtration or by passing the spirit through columns that contain charcoal in granular form.

These gin and vodka beverages differ from whisky, rum and brandy in the following ways:

- a) Brandy, rum and whisky are pale-yellow colored straw to deep brown by extractives from wooden casks in which they are aged and which have sometimes been used to store molasses or sherry. To obtain consistent color caramel is sometimes added. Gin, and vodka are water clear.
- b) The flavor of brandy and whisky is due to congeners present in the fermented mash or must. For Gin, and vodka the congeners derived from fermentation are removed and flavoring is provided (except in vodka) with plant parts.
- c) The raw materials for their production is usually a cereal but potatoes or molasses may be used. For gin, maize is used, while for vodka rye is used. The cereals are gelatinized by cooking and mashed with malted barley. In recent times amylases produced by fungi or bacilli have been used since the flavor of malt is not necessary in the beverage. Congeners are removed by continuous distillation in multicolumn stills.

5. Brandy

Brandy is a distillate of fermented fruit juice. Thus, brandy can be produced from any fruit—strawberries, paw-paw, or cashew. However, when it is unqualified, the word brandy refers to the distillate from fermented grape juice. It is subject to a distillation limitation of 170° proof (85%). The fermented liquor is double distilled, without previous storage, in pot stills. A minimum of two years maturation in oak casks is required for maturation.

Some of the best brandies come from the cognac region of France. Brandies produced in other parts of France are merely *eau de vie* (water of life) and are not called brandy. Certain parts of Europe (e.g., Spain, where brandy is distilled from Jerez sherry) and South America as well as the USA produce special brandies.

Conclusion: Distilled spirits are alcoholic products that result from the distillation of yeast fermentations of grain, grain products, molasses, or fruit or fruit products. Whiskeys, gin, vodka, rum, cordials, and liqueurs are examples of distilled spirits. Although the process for producing most products of these types is quite similar to that for beers, the content of alcohol in the final products is considerably higher than for beers.