

**Sub Name: FOOD BEVERAGE TECHNOLOGY**

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## **DISTILLED SPIRIT**

Distilled spirit, also called distilled liquor, alcoholic beverage (such as brandy, whiskey, rum, or arrack) that is obtained by distillation from wine or other fermented fruit or plant juice or from a starchy material (such as various grains) that has first been brewed. The alcoholic content of distilled liquor is higher than that of beer or wine.

The fermentation and distillation process for producing whiskey. The production of whiskey begins with grinding grain into a meal, which is cooked. Malt is introduced to the meal, which results in mash that is cooled and pumped into a fermenter, where yeast is added. The fermented mixture is heated in a still, where the heat vaporizes the alcohol. The alcohol vapours are caught, cooled, condensed, and drawn off as clean, new whiskey. This liquid is stored in a cistern room, and water is added to lower the proof (absolute alcohol content) before the whiskey is placed in new charred oak barrels for aging and later bottling.

The production of distilled spirits is based upon fermentation, the natural process of decomposition of organic materials containing carbohydrates. It occurs in nature whenever the two necessary ingredients, carbohydrate and yeast, are available. Yeast is a vegetative microorganism that lives and multiplies in media containing carbohydrates—particularly simple sugars. It has been found throughout the world, including frozen areas and deserts.

Distilled spirits are all alcoholic beverages in which the concentration of ethyl alcohol has been increased above that of the original fermented mixture by a method called distillation. The principle of alcoholic distillation is based upon the different boiling points of alcohol (78.5 °C, or 173.3 °F) and water (100 °C, or 212 °F). If a liquid containing ethyl alcohol is heated to a temperature above 78.5 °C but below 100 °C and the vapour coming off the liquid is condensed, the condensate will have a higher alcohol concentration, or strength.

### **HISTORY OF DISTILLING**

Because the two ingredients necessary to alcoholic fermentation are widely spread and always appear together, civilizations in almost every part of the world developed some form of alcoholic beverage very early in their history. The Chinese were distilling a beverage from rice beer by 800 BCE, and arrack was distilled in the East Indies from sugarcane and rice. The Arabs developed a distillation method that was used to produce a distilled beverage from wine. Greek philosophers reported a crude distillation method. The Romans apparently produced distilled beverages, although no references concerning them are found in writings before 100 CE. Production of distilled spirits was reported in Britain before the Roman conquest. Spain, France, and the rest of western Europe probably produced distilled spirits at an earlier date, but production was apparently limited until the 8th century, after contact with the Arabs.

The first distilled spirits were made from sugar-based materials, primarily grapes and honey to make grape brandy and distilled mead, respectively. The earliest use of starchy grains to produce distilled spirits is not known, but their use certainly dates from the Middle Ages. Some government control dates from the 17th century. As production methods improved and volume increased, the distilled spirits industry became an important source of revenue. Rigid controls were often imposed on both production and sale of the liquor.

The earliest stills were composed simply of a heated closed container, a condenser, and a receptacle to receive the condensate. These evolved into the pot still, which is still in use, particularly for making malt whiskeys and some gins. The next refinement was heating the alcohol-containing liquid in a column made up of a series of vaporization chambers stacked on top of one another. By the early 19th century large-scale continuous stills, very similar to those used in the industry today, were operating in France and England. In 1831 the Irishman Aeneas Coffey designed such a still, which consisted of two columns in series.

Since distillation requires that the liquid portion of a fermentation mixture be vaporized, considerable heat must be applied to the process. The fuel used in distilling spirits has always been that which has been most readily available at the particular time and place. Peat, coal, and wood were the fuels used historically, while the fuels of choice today are coal, natural gas, and oil. The high steam requirement for continuous-still operation inhibited the development of rectifying columns for production of spirits until after the Industrial Revolution.

Many of the minor components of distilled spirits, which are present only in parts per million, are detectable by the senses of taste and smell, but efforts to identify and quantify these compounds chemically have often been hampered by the lower limits of detection by analytical methods. Classes of compounds such as aldehydes, organic acids, esters, and alcohols were easily identified by conventional methods, but many of them could not be determined until after the development of chromatography. The Russian botanist Mikhail Tsvet was an early pioneer of this measurement technique, reporting his first work in 1903. Refinements in both technique and equipment, made during the first half of the 20th century, allowed numerous flavour components in distilled spirits to be identified by gas chromatography.

## **PRODUCING THE MASH**

### **Raw materials**

The raw materials used for making a distilled spirit are of two basic types: (1) those containing a high concentration of natural sugars or (2) those containing other carbohydrates that can easily be converted to sugars by enzymes. Enzymes are proteins that act as catalysts to promote chemical reactions. Very small amounts of an enzyme can cause a fundamental change in a large amount of material. Most enzymes are specific in their action, so that a system of several enzymes is necessary, for example, to convert starch into sugar and ultimately into ethyl alcohol. The amylases are enzymes that convert starches into sugars; sprouting grains—especially barley—are natural sources of these enzymes. Yeast has a complex enzyme system that converts sugar into carbon dioxide and a multiplicity of other products, including ethyl alcohol.

Reduced activity of any enzyme in the system distorts the results, often forming unwanted products. Enzymes are easily poisoned by certain compounds; they are also sensitive to temperature variations and to the degree of acidity of the medium.

- ***Sugary materials***

Grapes, cultivated in most of the subtropic and warm temperate zones of the world, are the major fruit employed as the raw material of distilled spirits, and the final product of their fermentation is brandy. Other natural fruits, such as apples and peaches, are used to a lesser extent, and many fruits are limited to local importance.

Sugary vegetables include sugarcane, sugar beets, and Agave tequilana (a type of cactus). Sugarcane and its products, including cane juices, molasses, and sugar, are the most important of the vegetable group. Grown throughout the tropics and semitropics, sugarcane is used in making rum and an alcohol derived from rum. Sugarcane juice can be pressed from the cane for use as the base raw material for fermentation, or the juice may be concentrated for sugar production, with the molasses residue from the sugar crystallization used as a base for fermentation. This process is also applied to sugar beets.

- ***Starchy materials***

For many centuries, it was only feasible to employ local grain crops for liquor production, and, in this way, the basic characteristics of the local distilled beverage were established. Improved transportation removed this restriction, and today economic considerations frequently determine grain selection, with the principal grain used being the one available at the lowest price per unit of fermentable materials.

Corn (maize) is the most important cereal grain employed; it is produced worldwide. Rye grain, though less efficient in fermentation than corn, is used extensively in whiskey production, primarily for the flavour characteristics it imparts to the final product. It is particularly employed in Canada and the United States. Rice, a widely grown cereal, has limited use in distilled spirits production outside of Asia from India to Japan. Barley grain, probably the first cereal employed for distillation in large quantities, was formerly a major crop throughout Ireland and western Europe. Wheat, because of its high cost, is used only where corn is in short supply and is then

limited to production of grain alcohol for blending or in production of liqueurs. Potatoes have been used in distilled spirits production primarily in central Europe; in the tropics, other starchy roots are employed.

## **PREPARING THE MASH**

### ***Milling and pressing***

The purpose of milling and pressing is to make the starch or sugar more available for enzyme action. Crushing and pressing (grapes and other fruits), milling (cereal grains), or a combination of milling and pressing (sugarcane) are used. In milling, grains are reduced to a meal to allow wetting of their starch cells. Various types of mills are used. Roller mills, where the grain passes through a series of corrugated rollers, was long the most common type. The grinding action of the rollers is mainly a shearing action. More efficient and economical impact-type mills (such as hammer mills) are now gaining in importance.

After the Industrial Revolution, steam replaced water as the power source for milling. Since the mid-20th century, electricity has been almost the exclusive power source in milling.

### **MASHING.**

The purpose of the mashing operation is to (1) mix the proper proportions of grains, (2) increase the availability of the starch for enzyme action, and (3) convert the starches into fermentable sugars.

Mashing is done in a vessel called a mash tun, which is equipped with a means of agitation for mixing and is either jacketed or contains coils for heating and cooling. In mashing, the starch cells of the grain, enclosed in their own protective coatings, are broken to allow wetting and liquefaction of the entire starch mass. The process usually begins with the grain most difficult to treat. When corn is used, the ground meal is wetted at a temperature of approximately 66 °C (150 °F), and the temperature is then raised to boiling or sometimes higher while under pressure. The temperature is reduced when the starch cells are broken. The grain ranking second in cell resistance (usually rye) is added next. Other starchy substances, such as potatoes, are usually crushed and heated, exploding the starch cells. The temperature of the mash is reduced before ground malt meal, either in dry form or as a water slurry (insoluble mixture), is added. The amylase enzymes in the malt then produce a mixture in which the starches have been converted to fermentable sugars, suitable for utilization by the yeast. The sugars, principally dextrose and maltose, vary in concentration among producers but, generally, are sufficiently concentrated to make a final product ranging from 7 to 9 percent alcohol.

Any germinating cereal grain can be used for malt. In rare cases, rye malt is used in making rye whiskey, but, because the enzyme activity of malted barley is the highest, barley is used almost exclusively in the distilling industry. Barley malt contains sufficient enzymes to convert approximately 10 times its weight in other unmalted grains. Of the two enzymes— $\alpha$ -amylase and  $\beta$ -amylase—the former is the more important for conversion of other grains. In addition to converting starches from other carbohydrates to sugars, barley malt contains soluble proteins (amino acids), contributing flavour to the distillate secured from fermentation and distillation of grain-malt mixtures.

## **FERMENTING AND DISTILLING**

### **Fermentation**

#### **Yeast and yeast culture**

As mentioned above, yeasts are found throughout the world; more than 8,000 strains of this vegetative microorganism have been classified. Approximately nine or 10 pure strains, with their subclassifications, are used for fermentation of grain mashes; these all belong to the type *Saccharomyces cerevisiae*. Each strain has its own characteristics, imparting its special properties to the distillate derived from its fermentation. A limited number of yeasts are used in the fermentation of wines, from which brandy is distilled. Strains used in the fermentation of grain mashes are also used in fermentation for rum, tequila, and beer production.

In grain-based products, yeast cells are grown in grain mixtures. The preparation of a cooked mash of rye and barley malt is most common. The mash is sterilized, then inoculated with lactic-acid bacteria to increase acidity. (Yeast is more tolerant of higher acidity than many commonly occurring bacteria.) When the desired acidity is reached, the mixture is again sterilized and a pure yeast culture is added. The yeast is grown under controlled conditions until it reaches the optimum point for mixing with the grain mash. In liquid fermentation, as from fruits and sugarcane, the yeast is generally grown in a mixture similar to the one it will be used to ferment; for example, a yeast culture to be used for molasses fermentation is usually grown in molasses.

### ***Fermenting methods***

In the fermentation process, simple sugars, including dextrose and maltose, are converted to ethyl alcohol by the action of yeast enzymes. Several intermediate compounds are formed during this complex chemical process before the final ethyl alcohol is obtained.

Yeast functions best in a slightly acid medium, and the prepared grain mash, fruit juice, molasses, or other mixture must be checked for adequate acidity (pH value). If acidity is insufficient, acid or acid-bearing material is added to achieve the necessary adjustment. The previously prepared yeast is then added, and final dilution of the mixture is made. The final concentration of sugars is adjusted so that the yeast fermentation will produce a finished fermented mixture containing between 7 and 9 percent alcohol.

Commercial fermentation is carried on in large vats. In the past these were open and made of wood, usually cypress. Most plants now use closed stainless steel vats for easier cleaning, and many are equipped with jackets or cooling coils for better temperature control. The time required for completion of fermentation is mainly dependent upon the temperature of the fermenting mash. Normal yeast is most effective in breaking down all of the fermentable sugars at temperatures ranging from 24 to 29 °C (75 to 85 °F), and, in this range, completion of fermentation requires from 48 to 96 hours. Fermentation at lower temperatures requires longer periods. The mash is ready for distillation upon completion of fermentation. If fermentation is allowed to continue past this period, it will be adversely affected by bacterial action. The ethyl alcohol content will be reduced, and the flavour and aroma of the finished product will be tainted.

## **DISTILLATION**

As mentioned above, the difference in the boiling points of alcohol and water is utilized in distillation to separate these liquids from each other. Basic distillation apparatus consists of three parts: the still or retort, for heating the liquid; the condenser, for cooling the vapours; and the receiver, for collecting the distillate.

### ***The pot still***

The simple pot still is a large enclosed vessel, heated either by direct firing on the bottom or by steam coils within the vessel, with a cylindrical bulb at its top leading to a partially cooled vapour line. The bulb and vapour line separate entrained liquid particles from the vapour on its way to the final condenser. The usual pot-still operation involves a series of two or three pot stills. Any vapour falling below a predetermined alcoholic content is fed into a second still, and condensed vapour from the second still falling below the required alcoholic content is fed to the third. The condensed vapours of the desired alcoholic content from all three stills are then commingled in a single receiving container.

The pot still, used primarily in Scotland and Ireland for whiskey production and in France for brandies, has had only brief use in distilled spirits production elsewhere and is gradually becoming obsolete. Even in countries in which the pot still has long been used, it has been replaced by continuous distillation for the major portion of alcoholic-liquor production, and its current use is limited to production of flavouring whiskeys and other flavouring ingredients.

The flavour profile of a pot-still product is more complex than that of a continuous-still product of the same alcohol content. This is a result of the different distillation methods. At a given temperature and pressure, vapours over a boiling mixture have a composition that is a function of the vapour pressures of the components of the mixture. In a pot still, the temperature of the fermentation mixture rises as the lower-boiling-temperature alcohol vaporizes. Meanwhile, the alcohol content of the distillate drops as the rising temperature vaporizes more water along with the alcohol. Distillation is allowed to continue until the alcohol content of the distillate falls to a predetermined level. Because of the rising temperature encountered in distilling a single batch, the composition of

the first part of the condensate to leave the pot is different from that of the last part. The composition of the final product is the average of the composition of the vapours condensed during the entire run. By contrast, the temperature of the continuous still is held approximately constant throughout the run. This results in a flavour profile that is more uniform.

### ***The continuous still***

The continuous still, which came into use in the early 19th century, consists of a tall cylindrical column filled with perforated plates onto which water-rich vapours condense while alcohol-enriched vapours pass through. These plates thus serve as a series of small pot stills, one on top of the other. Live steam, used as the heat source, is fed into the bottom of the still, and the liquid to be distilled is fed near the top. Steam pressure holds the liquid on the plates, and, with any overflow caught by the plate below, the liquid level on each plate is maintained. Use of a sufficient number of plates assures that the concentration of alcohol in the vapour leaving the top of the still will be appropriate for the desired product and that the liquid leaving the bottom has been stripped of any alcohol.

Many distillation operations combine column and pot stills. The condensed distillate from the column still is fed to the doubler, a type of pot still heated by closed steam coils, and redistilled.

### ***The rectification still***

Rectification is the process of purifying alcohol by repeatedly or fractionally distilling it to remove water and undesirable compounds. As mentioned above, a fermentation mixture primarily contains water and ethyl alcohol and distillation involves increasing the percentage of ethyl alcohol in the mixture. Water vaporizes very easily, however, and, unless care is taken, the distillate of a fermentation mixture will contain unacceptably large quantities of water. The fermentation mixture furthermore contains small quantities of complex constituents that can contribute to the flavour of the product even if they are present only in parts per million. It is important to retain those components that make a positive contribution to the product and to remove those that are unwanted, primarily some organic aldehydes, acids, esters, and higher alcohols. The ones that remain in the product are called congeners, and the congener level is controlled by the particular rectification system and by the system's method of operation.

The multicolumn rectifying system usually consists of three to five columns. The first column is always a preliminary separation column called the beer still, or analyzer. It usually consists of a series of metal plates with holes punched in them and baffles to control the liquid levels on the plates. The product coming from this column is between 55 and 80 percent ethyl alcohol. A 95 percent product can be produced on a two-column system consisting of a beer column and a rectifying column. The bulk of congener removal is accomplished in the rectifier—esters and higher alcohols, for example, being drawn off as side streams. However, a multicolumn system of several specialized rectifiers allows better control of the finished product. An aldehyde column, or purifier, is frequently used to separate these highly volatile low-boiling components, and sometimes ethyl alcohol is recovered in an extractive column and returned to the rectifier.

Three characteristics determine the elimination or retention of flavouring compounds: (1) their boiling points, (2) their solubilities in ethyl alcohol and water, and (3) their specific gravities. Some higher alcohols, for example, are removed on the basis of their solubility and specific gravity. These higher alcohols have limited solubility in water, and their specific gravities are less than that of water. Also, their boiling points are higher than that of ethyl alcohol and lower than that of water. Since they tend to accumulate in the rectifying column at the region where their boiling points cause them to condense, they can be drawn off as a liquid side stream. This side stream also contains a considerable amount of water. The limited solubility in water, plus the lower specific gravities, cause the higher alcohols to float to the top of the alcohol–water mixture, from which they can be removed.

## **MATURATION, BLENDING, AND PACKAGING**

One method of classifying distilled liquors is as aged or unaged. Vodka, neutral spirits for use in a variety of products, most gins, and some rums and brandies are unaged. Aged products are predominantly whiskeys and most rums and brandies.

The term age refers to the actual duration of storage, while maturity expresses the degree to which chemical changes occur during storage. The maturation of whiskeys falls into two categories, according to whether storage is in new or reused cooperage. New charred, white-oak containers are required by law in the United States for the maturation of products to be called straight bourbon or rye whiskey. These containers, each containing 50 to 55 gallons, are stored in warehouses sometimes having controlled temperature and humidity. Older warehouses are called rick houses because the barrels are stored on stationary frames called ricks. In many newer houses, barrels are stacked on pallets.

White oak is one of the few woods that can hold liquids while allowing the process of breathing through the pores of the wood. The pore size of the wood is such that small molecules such as water move through the wood more easily than larger molecules such as alcohol. This breathing process is caused by temperature and humidity differences between the liquid in the barrel and the air in the warehouse. Charring the wood makes some of the wood compounds more soluble. As the liquid in the container moves back and forth through the wood, ingredients are extracted and carried back into the container's contents. Maturation also results from the contact of oxygen from the outside air with ingredients in the alcohol mixture. Therefore, maturation during aging consists of the interaction of the original compounds of the distillate, of oxidation reactions, and of the extraction of flavouring compounds from the wood. These factors must be well balanced in the properly matured product. The lower the level of the original congeners, the less wood extract required to achieve a good balance.

Outside the United States, reused cooperage is common. Since used containers have already yielded their initial oak extracts, the resulting product is low in extracted flavouring ingredients, which is desirable in some beverages. This maturation method, typified by Scotch and Irish whiskeys, can be carried on in casks holding up to 132 gallons. These casks have usually had previous use for storage or maturation of other whiskeys or wines and may be reused for many maturation cycles. Maturation in dry warehousing increases the alcoholic content of the liquid in the container, but the more common practice for Scotch and Irish whiskeys of maturation in high humidity warehouses reduces the alcoholic concentration.

The maturation procedure for brandies is similar to that of some whiskeys, but the brandies are usually matured in fairly large casks or oak containers. Most brandies are matured for three to five years, but some remain for as long as 20 to 40 years or even longer.

Rum is usually matured in reused oak containers; high concentrations of oak extracts are not considered desirable. Normal maturation time is two to three years, but rum, generally a blended product, may contain a percentage of older rums.

Most governments specify storage time for various products. The United States requires a two-year storage period for most whiskeys but has no requirement for any pure alcohol or neutral spirits (close to 100 percent alcohol) added to such whiskeys in the production of blended whiskey. Canada requires storage of two years for all distilled spirits. Scotland and England require a three-year storage and Ireland, five years for all products classified as whiskey; there are no requirements for vodka and gin.

### ***Blending***

Blending is another method of obtaining a balanced product with precise flavour characteristics. Blended products are composed of one or more highly flavoured components, a high-proof component with a low congener content, a colour adjustment ingredient, and perhaps an additional flavouring material. An example is a blended whiskey, which may contain several whiskeys, a grain spirit distilled at 90 to 95 percent alcohol, caramel colouring, and perhaps a small amount of a flavouring blender (part of which may be sherry or port wine). A blended Scotch consists of several highly flavoured malt whiskeys produced in pot stills and a base whiskey produced from grain in a continuous distillation system.

### ***Packaging***

#### ***Bottling***

Distilled spirits react upon exposure to many substances, extracting materials from the container that tend to destroy the liquor aroma and flavour. For this reason, glass, being nonreactive, has been the universal container for packaging alcoholic liquors. (A few products are now packaged in plastic bottles, but these are primarily 50-

millilitre miniatures, the light weight of which is particularly suited for use by airlines.) Packaging economics require containers that are standardized in size and shape and that lend themselves to automatic processes.

Early hand methods of filling, labeling, corking, and other operations have been replaced by highly mechanized bottling lines, with bottles cleaned, filled, capped, sealed, labeled, and placed in a shipping container at a rate as high as 400 bottles per minute. This progress became possible with the development of high-strength glass, plastic closures with inert liners, and high-speed machines. Even specialized packaging, long a hand operation, has been replaced by standardization of containers, allowing production on automatic lines.