AP42 Section: **9.12.3 Distilled Spirits**

Reference **4**

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BEVERAGE SPIRITS, DISTILLED

The word alcohol, like alchemy, has its origins in the Middle East. The Arabs are said to have made cosmetic paints by heating and vaporizing a mixture of compounds. The residue was used to paint eyelids and called "kohl." When they later heated wines, they gave the product the same name as the cosmetic "kohl" or "al kohl." The word whiskey is said to be derived from the Celtic "uisge baugh" or "water of life."

Egyptians purportedly practiced distillation around 1000–2000 BC by heating wine and making a product called arden spirits. China and India are also said to have carried out distillation in the pre-Christian era. The Chinese reportedly made a distilled beverage from rice beer around 800 BC. The Arabs learned about distillation from the Egyptians and developed an apparatus in the form of a closed heated container that was called an alembic.

In some of his work, Pliny the Elder (24–79 AD) wrote of the heating of wine with flames. In the tenth century, the Persian philosopher Avicenna (980–1037 AD) described a distillation still. Magister Salernus wrote about "aqua ardens" around 1150 AD. The German alchemist and philosopher, Albertus Magnus (1200–1280 AD), studied wine distillation, made improvements, and wrote a manuscript on the production of aqua ardens.

Study of the alembic continued throughout the sixteenth century. Hieronymus Brunswick wrote *Liber De Arte Distillanti* in 1500 and described various improvements in distillation. Ryff produced a book on advances in the alembic still in Germany in 1556. In the eighteenth century, many further advances were made in the alembic, particularly by the French who worked with fruit brandies as opposed to the thick, grain mashes used by the Germans and English. These improvements are seen in the pot stills of today, used in the production of malt whiskeys in Scotland and brandy in France (Fig. 1).

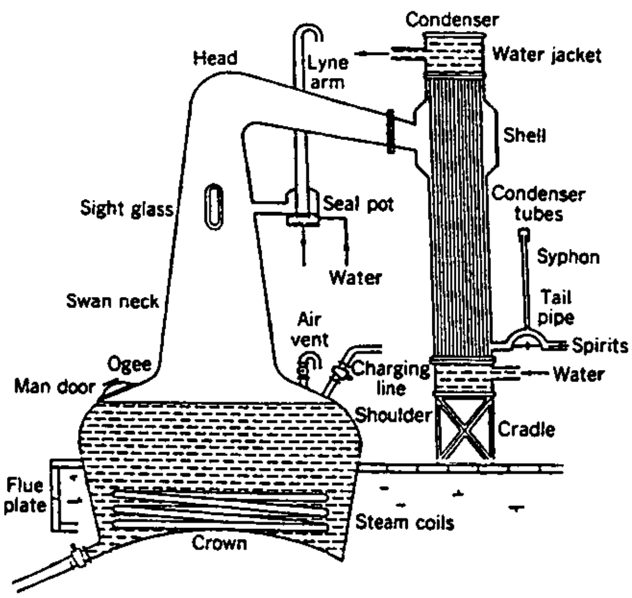

Fig. 1 Pot (batch) still.

In 1801, Edward Adam discovered redistillation or rectification. He designed an apparatus in which vapors pass from the kettle through an egg-shaped vessel into the condenser. Progress in distillation knowledge and equipment continued throughout the nineteenth century. In 1831 Aencas Coffey, in Dublin, Ireland, developed a continuous still that gave faster and lower cost distillations and yielded both higher proofs and a better quality product than the batch process pot still. The Coffey Still (Fig. 2) has two distillation columns: the beer still and the rectifier. The fermented mash (beer) is preheated in the rectifying column and then fed to the top of the beer still along with the feints, or spent portion of the mash. The alcohol is removed in the beer still and further refined by distillation in the rectifier.

In America, the Indians had fermented beverages made from maple syrup,

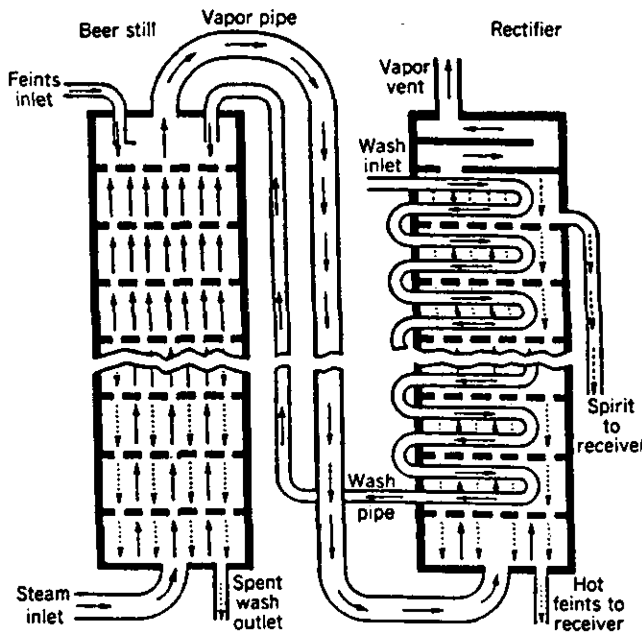

Fig. 2 Coffey continuous still.

corn, acorns, and other nuts. In the time of Columbus, they were drinking mezcal distilled from the fermented sap of maguey.

The colonists are said to have practiced distillation before 1650. The Virginia settlers made brandies and those in New England and the middle colonies distilled a variety of products including apple whiskey (apple jack), rum, and brandy. The first beverages made by the colonists from corn and rye were distilled on Staten Island, New York, in 1640 by William Kieft. Rum was produced in Barbados from molasses around 1650 and in colonial Massachusetts in 1657.

Distilling ingredients changed between 1790 and 1830. Rum was replaced by whiskey made from rye and corn, since molasses was becoming difficult to import. The Reverend Elijah Craig is credited with producing the first sour mash bourbon whiskey in 1789 in the area that is now Georgetown, Kentucky, located in Bourbon County and thus giving the product its name. Kentucky Bourbon was made in a manner similar to whiskey made in Scotland and Ireland except a different mash was used. It contained at least 51% corn to distinguish it from Pennsylvania rye whiskey. The mash was fermented from three to five days, distilled, and then redistilled. Even though it was known that whiskey stored in charred oak barrels becomes mellow and golden in color, most whiskey in those days was sold in its natural white state or artificially colored to resemble brandy.

In the 1880s and 1890s, whiskey production grew significantly. Excessive production and intense competition resulted in mergers such as the Whiskey Trust in Peoria and the Kentucky Distilleries and Warehouse Companies. They attempted to control production and raise prices but had little success in doing so.

Distillers distributed their whiskey in barrels. It was bottled or served in the taverns; this led to tampering and excessive dilution. In 1870 George Garvin

Brown started bottling and sealing whiskey to ensure its quality. The Bottled in Bond Act of 1897 encouraged putting whiskey in bottles. It required that whiskey be bottled in bond must be 100° proof, at least four years old, and produced in one season at one distillery. The bottle was sealed with a green stamp, indicating tax payment and compliance with federal law. In 1900 the Pure Food and Drug Act was passed which required a statement of the manufacturing process and ingredients on the label. In 1909, under President Taft, whiskey was finally defined as "any volatile liquor distilled from grain." Standards of identity were developed based on current manufacturing processes for the various whiskies including bourbon and rye.

Prohibition, which made the sale of spirits illegal from 1920 to 1933, actually resulted in increased consumption from 530 to 750 million liters annually. At the end of Prohibition, many mergers occurred and modern technology was introduced to the industry.

Government Regulations and Taxation

Distilled spirits and the industry have always been subject to heavy taxation. Not only has it been an excellent source of revenue for many governments throughout the world, but high taxes can also be rationalized as having an inhibitory effect on consumption.

In England, the Magna Carta provided a standard of measurement for the sale of ale and wine. In 1643, the English Parliament proposed the first tax on distilled spirits. In the American colonies, William Kieft, Director General of New Netherland, imposed the first liquor tax of two guilders on each half vat of beer in 1640. Alexander Hamilton initiated an excise tax on domestic spirits in 1791. The tax was resented and ultimately repealed in 1800 by Thomas Jefferson. Except during the War of 1812, domestic spirits remained untaxed until 1862. At that time, a tax of $0.02/L was implemented, which has been increased periodically. In January 1991, the Federal Excise Tax on distilled spirits was raised to $3.56 per liter or $13.50 per proof gallon. In addition, many states have substantially increased the state excise taxes on distilled spirits.

In the United States, the Alcohol Tax Unit came into being with the repeal of Prohibition in 1933, and it became the Alcohol and Tobacco Tax division of the Internal Revenue Service in 1952. The Bureau of Alcohol, Tobacco, and Firearms (ATF), established in 1972, and the Department of the Treasury closely regulate the manufacture of distilled spirits.

Production and Consumption Patterns

United States distilleries produced 32 million liters (8.5 million gallons) in 1810. Production and consumption grew with the population over the next 100 years. In anticipation of Prohibition, over 1.1 billion liters (300 million gallons) of distilled spirits were produced in 1917 including 225 million liters of whiskey. Although total consumption peaked in 1981 at 1.7 billion liters, individual adult consumption peaked in 1971 at 11.6 liters per adult (Fig. 3). The decline has continued

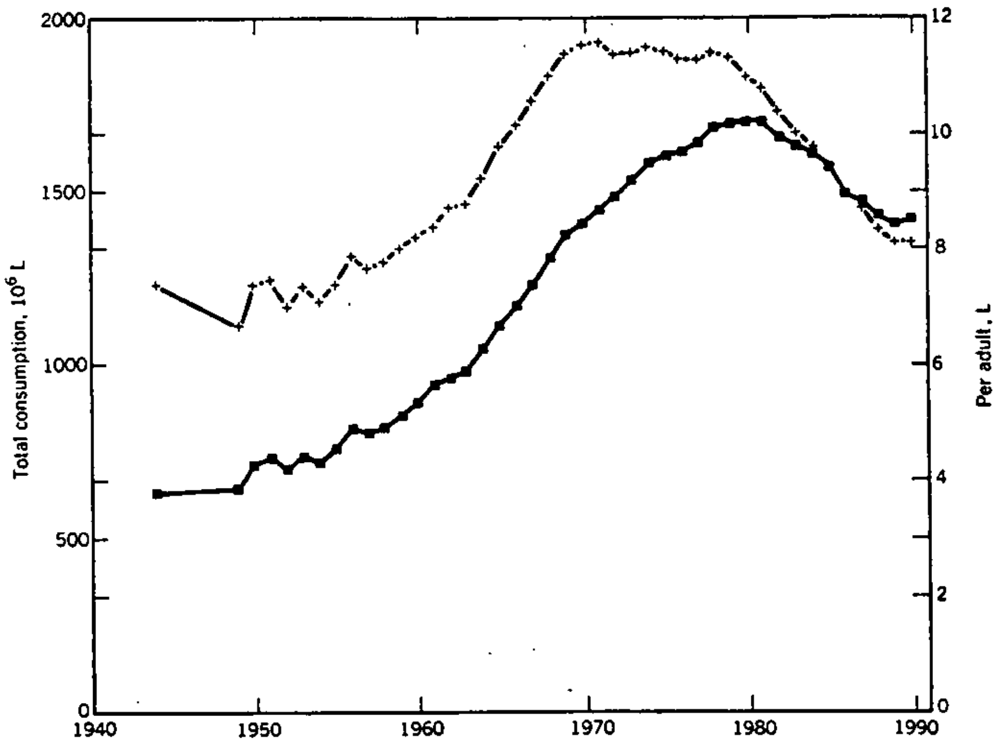

Fig. 3 Consumption of distilled spirits from 1940 to 1990, where ■, total; +, per adult (1).

through 1990 and is projected to continue because of increasing taxes and changing attitudes and lifestyles. The consumption trend is toward lower proof, lighter alcoholic beverages covering an ever widening spectrum of products including premixed cocktails, cordials, creams, light whiskeys, and wine coolers. Although overall spirit consumption is declining, the premium segment including whiskey, vodka, and flavored products has recently shown growth, indicating a more selective, upscale consumption pattern.

Distillers' Definitions

Alcohol Yield. With corn containing 60% starch, distillers traditionally obtain 19–19.7 L (5.0–5.2 proof gallons)/0.03 m^3 (bushel). Theoretical yields as liters of absolute alcohol/100 kg of starch are

stoichiometric 72.0 L (100%)
maximum level 68.4 L (95%)
industrial standard 62–65 L (86–90%)

For the maximum level, 5% or more of carbon substrate is consumed by yeast growth and by-product formation.

Backset. Backset is the screened aqueous by-product from distillation. It is recycled and added to the cooked grain mash prior to fermentation.

Balling. Balling is a measure of the sugar concentration in a grain mash, expressed in degrees. It approximates percent by weight of the sugar in solution.

Beer. Beer is the alcoholic product arising from the yeast fermentation of saccharified grain mash. It may or may not include stillage from a previous fermentation/distillation (see BEER).

Bonded Whiskey. Bonded whiskey is whiskey stored at least four years in wooden containers where the spirits have been in contact with the wood surface. It is unaltered from the original character by the addition or subtraction of any substance other than by filtration or chill proofing, is reduced in proof by the addition of water to 100° proof (50 vol %) and bottled at 100° proof, and is produced at the same distillery in the same season (January through June or July through December).

Congeners. Congeners are the flavor constituents in beverage spirits that are responsible for its flavor and aroma and that result from the fermentation, distillation, and maturation processes.

Conversion. Conversion describes the enzymatic starch hydrolysis processes, liquification, and saccharification.

Cooking. Cooking is the gelatinization by heat treatment and α-amylase liquification of raw material starch (qv).

Doubler. A doubler is a pot still used to redistill whiskey and low wines from a beer still. The low wines are fed into the doubler where they are redistilled by way of steam enclosed in a scroll at the bottom of the still. The bottoms, the organic components remaining at the bottom of the still, are returned to the beer still to extract the alcohol.

Feints. Feints are the third fraction of the distillation cycle derived from the distillation of low wines in a pot still. This scotch term is also used to describe the undesirable constituents of the wash that are removed during the distillation of grain whiskey in a continuous patent still (Coffey). These are mostly aldehydes and fusel oils.

Fermentable Sugars. Fermentable sugars like glucose [*50-99-7*], maltose [*69-79-4*], and maltotriose [*1109-28-0*], can be fermented by distiller's yeast.

Foreshots. Foreshots is the first fraction of the scotch distillation cycle derived from the distillation of low wines in a pot still.

Fusel Oil. Fusel oil is an inclusive term for heavier, pungent tasting alcohols produced during fermentation. Fusel oils are composed of a mixture of *n*-propyl, isobutyl, and isoamyl alcohols.

Grain Whiskey. Grain whiskey is an alcoholic distillate from a fermented wort derived from malted and unmalted barley and corn, in varying proportions, and distilled in a continuous patent still (Coffey).

Heads. Heads is distillate containing a high percentage of low boiling components such as aldehydes.

High Wines. High wines is an all-inclusive term for beverage spirit distillates that have undergone complete distillation.

Infusion Mashing. Infusion mashing is the process of simultaneously cooking and converting small grains (rye, barley, and wheat).

Limit Dextrins. Limit dextrins are oligosaccharides containing one or more 1,6-α-linkages (see CARBOHYDRATES).

Low Wines. Low wines is the term for the initial product obtained by separating (in a pot or Coffey still) the beverage spirits and congeners from the wash. Low wines are subjected to at least one more pot still distillation to attain a greater degree of refinement in the malt whiskey.

Malt Whiskey. Malt whiskey is an alcoholic distillate made from a fermented wort derived from malted barley only and distilled in pot stills. It is the second fraction (heart of the run) of the distillation process.

Proof. The alcoholic concentration of beverage spirits is expressed in terms of proof in Canada, the United Kingdom, and the United States. U.S. regulations define this standard as follows: proof spirit shall be held to be that alcoholic liquor which contains one-half its volume of alcohol of a specific gravity of 0.7939 at 15.6°C ie, the figure for proof is always twice the percent alcohol content by volume. For example, 100° proof means 50% alcohol by volume. In the United Kingdom as well as Canada, proof spirit is such that at 10.6°C alcohol weighs exactly twelve-thirteenths of the weight of an equal bulk of distilled water. A proof of 87.7° indicates an alcohol concentration of 50%. A conversion factor of 1.142 can be used to change British proof to U.S. proof.

Wash. Wash is the liquid obtained by fermenting wort with yeast. It contains the beverage spirits and congeners developed during fermentation.

Wine Gallon. Wine gallon is the measure of actual volume; a U.S. gallon (3.785 L) contains 3785 cm^3 (231.0 cubic in.); a British (Imperial) gallon contains 4546 cm^3 (277.4 cubic in.).

Wort. Wort is the liquid drained off the mash tun containing maltose, a grain sugar derived from the conversion of starch during the mashing process by the action of the organic enzyme, maltase, found in barley malt.

Proof Gallon. Proof gallon is a U.S. gallon of proof spirits or the alcoholic equivalent thereof, ie, a U.S. gallon 3785 cm^3 (231 cubic in.) containing 50% of ethyl alcohol by volume. Thus a gallon of liquor at 120° proof is 1.2 proof gallons; a gallon at 86° proof is 0.86 proof gallons. A British and Canadian proof gallon is an imperial gallon of 4546 cm^3 (277.4 cubic in.) at 100° proof (57.1% of ethyl alcohol by volume). An imperial gallon is equivalent to 1.2 U.S. gallons. To convert British proof gallons to U.S. proof gallons, multiply by 1.37. Since excise taxes are paid on the basis of proof gallons, this term is synonymous with tax gallons.

Single Whiskey. Single whiskey is the whiskey, either grain or malt, produced by one particular distillery. Blended Scotch whisky is not a single whiskey.

Sour Mash. Sour mash is made with a lactic culture and not less than 20% stillage added back to the fermentor and fermented for at least 72 h.

Spirits. Spirits are distilled spirits including all singular whiskeys, gin, brandy, rum, cordials, and others made by a distillation process for nonindustrial use.

Stillage. Stillage is dealcoholized fermented mash.

Tails. Tails is a residual alcoholic distillate.

Spirit Types

In spite of a decline over the past 50 years, whiskeys are still the most popular distilled alcoholic beverage group in the United States (Table 1) (1). However, vodka consumption has increased significantly to 22% of total distilled spirits in 1990.

Table 1. United States Liquor Consumption by Type, %

Type	1949	1960	1966	1978	1990
blends	66.2	31.3	24.3	10.4	5.9
straights	8.7	25.4	23.9	14.0	10.6
bonds	5.4	4.1	2.4	0.7	0.1
Scotch	4.4	8.1	10.4	12.0	8.5
Canadian	2.7	5.2	6.9	11.4	13.0
other	0.4	0.3	0.3	0.1	0.1
Total whiskey	*87.8*	*74.4*	*68.2*	*48.6*	*38.2*
gin	7.1	9.3	10.5	9.5	8.6
vodka	0.0	7.8	10.4	20.0	22.7
cordials	2.2	3.8	4.3	7.9	11.1
brandy	1.3	2.6	3.2	3.8	4.9
rum	1.3	1.6	2.2	5.8	8.3
tequila and other	0.3	0.5	1.2	4.4	6.2
Total nonwhiskey	*12.2*	*25.6*	*31.8*	*51.4*	*61.8*
Total consumption, million liters[a]	*641.5*	*888.3*	*1169.2*	*1786.1*	*1386.0*

[a]To convert liters to gallons, divide by 3.785.

Because of the economic interest in distilled spirits, each country has established standards for their various types of distilled beverages, and countries mutually respect each other's alcoholic beverage standards. U.S. Standards of Identity are given by the Bureau of Alcohol, Tobacco, and Firearms (ATF) (2).

Within each type of distilled spirits, wide variations of flavor can be achieved by the type and amount of starting grains or other fermentable materials, methods of preparation, types of yeasts, fermentation conditions, distillation process, maturation time and temperature, blending, and use of new technologies such as membrane separation.

The flavor and aroma of distilled spirits are derived primarily from minor constituents called congeners that are produced and augmented in the fermentation and maturation processes. The congener profiles for various distilled spirits are shown in Table 2.

Canadian. By government regulation, Canadian whiskeys contain no distilled spirits less than three years old. They are usually blended products and are often up to six years of age. Canadian whisky tends to be light bodied and delicate in flavor. The Canadian government sets no limitations as to mashing formulas, distilling proofs, or types of cooperage used in maturation.

As in the United States, Canadians use corn, rye, and barley malt. Their process is essentially the same as the one used by many distilleries in the United States. Since they have no limitations on distillation proofs, distillers operate their systems for optimum separation and congener concentration. In addition, they are permitted to add blenders or flavoring components up to 9.06% by volume in the final blending after the aging process.

Table 2. Congeneric Content of Various Distilled Alcoholic Beverages[a]

Component	Canadian	Scotch	Bourbon whiskey	Kentucky whiskey	Cognac brandy	Tequila
fusel oil	53.0	105.0	199.0	195.0	193.0	195.0
total acids[b]	20.0	15.0	69.0	63.0	36.0	
esters[c]	4.6	11.4	23.0	18.0	41.0	12.9
aldehydes[d]	2.0	4.1	3.9	3.2	7.6	5.3
furfural [*98-01-00*]	0.11	0.11	0.45	0.90	0.67	
total solids, g/100 mL	82.0	109.0	102.0	80.0	698.0	
tannins	18.0	8.0	52.0	48.0	25.0	21.0
color at 420 nm	5.4	5.6	9.5	8.0	11.0	

[a]Grams per 100 liters at 100° proof (50%). Determinations were made according to the official methods of analysis of the Association of Official Analytical Chemists, 15th ed., 1990.
[b]As acetic acid [*64-19-7*].
[c]As ethyl acetate [*141-78-6*].
[d]As acetaldehyde [*75-07-0*].

White oak barrels of 190 liters (50 U.S. gallons) that have been previously used for bourbon maturation are often used a second and third time to age Canadian whisky. This used cooperage along with the higher proof distillation gives Canadians their characteristic light flavor compared to the heavy flavor of most bourbons aged in new charred oak barrels.

Scotch. In 1988, the Scotch Association Council approved a new, tighter definition for Scotch whisky which is as follows: "Scotch whisky is a potable spirit—

a) which has been produced in Scotland:
   (i) from water and malted barley, with or without whole grains of other cereals, wholly processed at the distillery into a mash, converted to a fermentable substrate solely by the indigenous enzyme systems, fermented with the addition of yeast only;
   (ii) by distillation of the wash obtained there at an alcoholic strength by volume of less than 94.8% in such a way that the distillate has an aroma and flavor derived from the said materials and process; and
b) which has been matured in Scotland:
   (i) in oak cask of a capacity not exceeding 700 liters;
   (ii) for a period of not less than three years;
   (iii) in excise warehouse, which for the purpose of this definition has the meaning assigned to it by Section One of the Customs, Excise Management Act 1979; and
c) which retains the color, aroma, and taste resulting naturally from the above process; and
d) to which no substances may be added other than:
   (i) water, and
   (ii) spirit caramel and
e) whose alcoholic strength apart from any natural evaporation losses may be reduced only by the addition of water to a bottling strength of not less than 40% alcohol by volume."

Whole grains means grains of cereals from which no part has been intentionally removed. The unique taste characteristics and smokey flavor of Scotch is developed from peat used in the whisky production process. The character and amount of peat used in malting the barley have a critical affect on the flavor intensity of the final product. The aroma of the burning peat is absorbed by the barley malt and is carried through the distillation process.

The dried malted barley is ground and mashed in a tub, after which the liquid portion is drained off, cooled, and placed in the fermentor. After fermentation, a batch distillation system is usually used to separate the whisky from the fermented wort. The still consists of a copper kettle with a spiral tube or "worm" leading from the top. The dimensions and shape of the stills have a critical effect on the character of the whisky. The product taken off in the first part of the distillation is called foreshots (heads). The middle portion is the high wines and the last portion is the feints (tails). The middle portion is redistilled at the 140–160° proof (70–80%) range and matured in used oak cooperage.

The grain whiskeys used in Scotch blends are produced using corn, rye, and barley malt and are distilled using a continuous multicolumn still at 180–186° proof (90–93%). Grain whiskeys are aged in used oak barrels of 190 liter capacities. The used barrels are often purchased in the United States from bourbon distilleries.

The single malt Scotch or malt Scotch, which has recently become popular in the United States, is made from a mash of only malted barley. Single malts are usually darker with heavier flavor than blended Scotches because of increased aging and the absence of the lighter grain whisky.

Irish Whiskey. Irish whiskeys are blends of grain and malt spirits three or more years of age that are produced in either the Republic of Ireland or Northern Ireland and comply with the respective laws regulating their manufacture. Since no peat is used in the malting process, Irish whiskey lacks the smokey character of Scotch. In the manufacturing process, the malt is soaked in water and milled to produce the wort. The fermentation usually takes about 60 hours. The first distillation in a pot still yields a 22–23% alcohol product. A second pot still distillation produces a product that is 45–46% alcohol. This is followed by a third distillation in another pot still to yield the Irish whiskey of about 68–70% alcohol.

Irish whiskey is matured in used barrels at about 63% alcohol. It is usually considered more flavorful and heavier bodied than blended Scotch whiskeys.

United States Spirits. The manufacture of distilled spirits is tightly controlled within narrow limits that are specified in reference 2. ATF regulations require that a detailed statement of the production process be submitted for approval prior to placing any process in operation.

Distilled beverages are classified according to type, materials, composition, distillation and maturation proofs, types of barrels, and maturation time.

Whiskey. Whiskey refers to any alcoholic distillate made from a fermented grain mash at less than 190° proof (95%) in such a manner that it possesses the taste, aroma, and characteristics generally attributed to whiskey. It is matured in new or used charred oak barrels. Whiskey can be further delineated by the cereal grains used and the maturation time and blending, if any.

Neutral Spirits. Neutral spirits are produced from any fermentable material and are distilled at or above 190° proof and bottled at 80° proof or higher. The substrate must be specified unless it is grain.

Grain Spirits. Grain spirits are neutral spirits from grain that are matured in used oak barrels and bottled at 80° proof or higher. The period of aging in oak may be declared on the bottle.

Vodka. Vodka is a neutral spirit made from any fermentable material and distilled in such a manner that is without any distinctive character, taste, aroma, or color. Charcoal filtration is often used in processing vodka which is bottled at 80° proof or higher. In the United States, the substrate must be specified if it is not grain. Any flavoring, if added, must be stated. The product must be bottled at not less than 70° proof and called a flavored vodka.

Light Whiskey. Light whiskey is distilled at not less than 160° proof and not more than 190° proof. It is matured in used charred-oak barrels or new uncharred barrels. Blended light whiskey is light whiskey mixed with less than 20% straight whiskey on a proof gallon basis. Distillers enjoy some latitude in the production of light whiskey matured in used barrels. As long as it is 189° proof as received in the cistern room, the distiller may do some additional blending prior to entry into the barrel.

Bourbon. Bourbon, and also rye, wheat, malt, and rye malt whiskeys, are made from a fermented mash not less than 51% corn, rye, wheat, malt, or rye malt, respectively. They are distilled at not over 160° proof and matured at not more than 125° proof in new charred oak barrels and bottled at not less than 80° proof. If stored for less than four years, it must be declared on the label.

Corn Whiskey. Corn whiskey must be distilled from a fermentable mash that contains at least 80% corn and at not over 160° proof. It is usually matured in new uncharred oak barrels or used oak barrels and bottled at not more than 125° proof.

Straight Whiskey. Straight whiskey is distilled at not over 160° proof and barreled at not more than 125° proof. It must be matured for at least two years in new charred oak barrels and bottled at not less than 80° proof. This whiskey may be called "bottled in bond" if it has been distilled at one plant, matured for at least four years, and bottled at 100° proof by the same distiller.

Sour mash fermentations must have not less than 20% stillage added back (backset) to the mash and be fermented for not less than 72 hours. A lactic culture is used and is permitted to develop for a period of not less than six hours.

Blended Whiskey. Blended whiskey is made with at least 20% of 100° proof straight whiskey either separately or in combination with whiskey or grain neutral spirits. When a blended whiskey contains at least 51% (v/v) of straight whiskey (eg, bourbon), it may be labeled as blended bourbon or bourbon whiskey, a blend.

Tennessee Whiskey. Tennessee whiskey is a product made by Tennessee distillers and processed in a manner similar to bourbon. However, Tennessee whiskey is filtered through maple charcoal prior to maturing which gives it its distinctive flavor. Tennessee distillers make their own charcoal by slowly burning 1.8-m lengths of hard maple wood. During the burning process the wood is periodically wet down to cause it to char rather than disintegrate into ashes. The charcoal is pulverized and packed into tanks and the new whiskey is allowed to filter through the charcoal. This adds an extra smoothness character to the whiskey.

Gin. Gin is a botanical flavored spirit first produced in 1650 by Franciscus de La Boe, a professor of medicine at the University of Leyden, attempting to produce a palatable, therapeutic medicine. He distilled alcohol in the presence of juniper berries being aware that the Latin juniperus communis means youth giving.

Gins derive their character from the type of mash used to produce the grain neutral spirits and the quality of the juniper berries and other botanicals used in the redistillation process. A wide variety of botanicals is used in gin including angelical root, anise, carroway seeds, citrus peels, licorice, and other barks, herbs, and roots.

U.S. regulations define two types of gin; distilled gin and compounded gin. Distilled gin is produced from the original mash or the redistillation of neutral spirits with juniper berries and other botanicals. Distilled gin may retain this labeling as long as juniper berries are present during distillation and other aromatics used in the formula may be added as liquid concentrates purchased or produced by the distiller.

Compounded gin is produced by adding extracts of juniper berries and other botanicals to high proof neutral spirits. This gin is perceived to be a lower quality than distilled gin and not much is produced by this method.

Gin is usually distilled at 180–190° proof. In the second distillation, crushed juniper berries are placed on mesh trays or perforated racks called gin heads in the distillation column. The vapors then extract the aromatic flavoring oils and carry them over with the distillate.

London dry gin is produced from a mash containing more barley and less corn, which is said to give the product more smoothness. This gin is distilled at high proof often under reduced pressure at about 57°C to avoid thermal decomposition and enhance smoothness.

Other Types of Spirits. *Brandy.* Brandy is a distillate from fermented juice, mash, fruit wine, or fruit residues. It is distilled at less than 190° proof in such a manner as to produce the taste, aroma, and characteristics generally attributed to brandy. Fruit brandy is distilled solely from the fermented juice or mash of whole, ripe fruit or from standard grape, citrus, or other fruit wine. Brandy distilled exclusively from one variety of fruit must be so designated, except grape brandy which can be identified by the term brandy. Brandy must be matured a minimum of two years in oak barrels, otherwise it must be labeled immature.

Brandies are distilled using batch or continuous systems. Variations of the pot still are used in France. Elsewhere, both systems are used. The batch system yields a more flavorful product, whereas the continuous still yields a lighter flavor. The first distillate using a pot still is taken off at 60° proof. It is then redistilled to 148–160° proof. Brandy is matured in charred-oak barrels for two to eight years and bottled at 80° proof or higher.

The most famous brandy comes from the Cognac region of France. It is double distilled in traditional pot stills by small farmers and sold to the blenders for aging in limousin oak casks. French law requires that the stills be 3000 liters or less and that the distillation be completed by March 31.

Armagnac, another well-known French brandy produced since 1422, must originate from the Armagnac district of France to be so labeled. It is described as fuller, richer, and more mellow than Cognac. Armagnac is produced from wines using continuous copper stills and aged in 400-L oak casks with ridged staves to expose more of the surface area.

In the United States about 95% of the brandy comes from California. The first brandy was made in 1837 though it was not produced in quantity until 1867.

All California brandy must be made from grapes grown and distilled in the state and aged a minimum of two years in oak barrels.

Rum. Rum is a distillate from the fermented juice of sugar cane, sugar cane syrup, molasses, sugar beets, or other by-products distilled at less than 190° proof in such a manner that it possesses the taste, aroma, and characteristics generally attributed to rum. It is bottled at not less than 80° proof.

There are three types of rum: light or amber, full-bodied, and aromatic rums. Light rum is also called white rum and is usually colorless having a very light molasses flavor. It is distilled on a multicolumn continuous still at 160–180° proof and can be matured in either glass, stainless steel, or uncharred oak barrels. The age of the rum does not need to be declared on the labels. Light-bodied rums are distilled in Puerto Rico, the Virgin Islands, Cuba, the Dominican Republic, and Haiti.

Amber or gold rums can be matured in wood barrels three years, though the color in gold rums should not necessarily imply that it was derived from aging. Often the color is achieved by adding caramel color to the product. They are more flavorful than light rum.

Full-bodied rums are allowed to ferment from 12 to 20 days, often relying on natural or wild fermentation in which the mash is inoculated by the yeast present in the air and starting materials. These rums are twice-distilled in pot stills to 140–160° proof. Full-bodied rums are often aged from five to seven years in oak barrels. Caramel color can also be added to give them a darker color. They are produced in Jamaica, Barbados, Martinique, Trinidad, and Guyana.

Aromatic rum is produced on the Island of Java in Indonesia. It derives its unique aromatic character from the addition of dried red Javanese rice cakes to the fermenting mash. After maturating for three to four years, the rum is shipped to Holland for additional aging prior to blending and bottling.

Tequila. Tequila is an alcoholic distillate produced in Mexico from the fermented juice of the heads of the Agave Tequilana Weber (blue variety) cactus. It is cultivated and takes 8 to 12 years to mature. Only the heart or the head of the plant, which weighs 36–50 kg, is used. It is chopped and steamed in a masonry oven for 9 to 24 hours depending on the rate at which inulin is converted to fermentable sugars. After a 12-h cooling period, the cooked heads are shredded and rolled to separate the juice from the pulp. The pulp residue is washed with water to remove additional sugars. Both the juice and pulp washings are then pumped into 3800–7500-L fermentors. The juice and pulp wash can be supplemented with sugar cane syrup or brown sugar not to exceed 50% of the total fermentable sugar. Ammonium sulfate can be used as a nutrient for fermentation. Water is added to the fermentable mixture so that the sugar content is about 9% (w/v). The fermentation takes 38 to 42 hours at a temperature of about 36°C. The final alcohol concentration is about 4.5% (w/v).

The fermented mash is pumped into a 1100-L copper pot still. The primary distillate is collected at 28° proof. It is redistilled in a larger pot still at 110° proof. The residual distillate is combined with fermented mash to start a new cycle in the first distillation.

Tequila is usually bottled at 80–86° proof. It is sold unaged as white tequila or it can be matured in oak barrels. Aging gives Tequila a golden color and a pleasant mellowness without altering its basic taste.

Tequila can only be produced in an area of Mexico known as Tequila in the state of Jalisco, about 40 miles from Guadalajara. It is called mezcal or maguey when produced outside of Tequila.

Cordials and Liqueurs. Cordials and liqueurs are the same products, with the different names being the American and European designations, respectively. They are produced by blending or redistilling neutral spirits, brandy, or other distilled spirits, with fruits, flowers, plants, juices, or concentrates, and other natural flavoring materials or extracts derived from infusions, percolations, or macerations of such materials. Cordials must contain a minimum of 2.5% (w/w) of sugar or dextrose or a combination of both. If the added sugar and dextrose are less than 10% (w/w), the cordial may be designated as dry. Most cordials contain larger amounts of sugar and other sweeteners. U.S. cordials containing synthetic or artificial flavoring materials must be labeled and are considered a spirits specialty.

A tremendous variety of cordials are available in a wide spectrum of flavors from fruits, peels, leaves, roots, herbs, seeds, and barks. The proofs range from 25° to 100°.

Cordials were said to be produced in ancient Egypt and Athens. Commercial production started in the Middle Ages when alchemists, physicians, and monks were searching for the "elixir of life." Many well-known cordials were developed in this period, such as Benedictine and Chartreuse, both bearing the names of the monasteries where they were first developed.

Benedictine was made in 1510 by Dom Bernardo Vincelli at the abbey in Frecomp, Normandy. It is one of the few liqueurs that is aged for four years after blending. Benedictine and Brandy (B&B) was introduced in 1937 after the discovery that Americans were adding brandy to Benedictine. Chartreuse, first made in 1605, is formulated with over 130 herbs and spices macerated in brandy.

Manufacturing Process

Ethyl alcohol [*64-17-5*], C_2H_6O, is produced by the fermentation of materials containing sugar or substances convertible to sugar, such as starches and fruit processing residues. Cereal grains are usually used in the production of beverage distilled spirits. Beverage alcohol is always ethyl alcohol, CH_3CH_2OH. Higher alcohols may be present in distilled spirits and are referred to as fusel oils or by specific name.

Composition of grains varies considerably and depends on factors such as climate, soil, and hybrid variety. Another variable is the malt; it is generally germinated barley, though rye malt or wheat malt can be used. The malting process develops the active enzymes (amylases) in the grain that convert grain starch into dextrins and then to maltose, a fermentable sugar. The malt and malting technique also can affect the final flavor and aroma of the alcohol, as in the case of Scotch whisky.

Grain Handling and Milling. The beverage industry utilizes premium cereal grains with particular specifications, especially in regard to the elimination of grain with objectionable odors which may have developed during storage or drying at the elevators. Hybrid corn, usually of the yellow dent variety, along with rye, barley, and wheat (small grains), is used for beverage alcohol production. The corn usually contains 60% starch and 12–14% moisture. U.S. No. 2 grade or better corn is used in the distilling industry. The small grains are selected for the unique flavors they add to distillates.

Distilleries receive grain in either hopper railcars or trucks. It is usually transferred from the unloading pit by a pneumatic conveyor system or auger system. Even though the grain has been subjected to a cleaning process at the elevator, it is passed over receiving separators, a series of vibrating screens that sift out foreign materials. Air jets and dust collectors remove light materials and magnetic separators remove items containing iron.

Milling breaks the outer cellulose protective wall around the kernel and exposes the starch to the cooking and conversion processes. Distillers require an even grind with as small a particle size as can be physically handled by the facility.

Milling is usually accomplished by two methods. Hammer mills use a series of revolving hammers within a close-fitting casing, rotating at 1800–3600 rpm to shear the grain to a meal that is removed by suction through a screen with different meshes for various types of grain. Cage mills use a series of counter rotating bars at high speed to grind the grain by impact. The grind is, however, not as uniform as hammer mills and produces much more flour.

Mashing. The mashing process consists of cooking (gelatinization) the starch and converting (saccharification) it to grain sugar (maltose). Cooking can be carried out at or above atmospheric pressure in either a batch or continuous system. For whiskey production, batch cooking at atmospheric pressure is widely used, although some batch pressure cooking is practiced. For grain neutral spirits production, both batch and continuous systems are used under pressure. After cooling, conversion is accomplished in the cooking vessel by adding barley malt or enzymes from other sources to the cooked grain. Some distillers immediately pump the mash to a converter for the necessary holding time and thus make the cooking vessel available for the next cook. The converted mash is cooled and pumped to the fermentors.

Some bourbon distillers use gibberellin-treated malt along with glucoamylase to reduce by 50% the amount of malt in their grain bills and thereby lower production costs.

Distillers vary mashing procedures, but generally conform to basic principles, especially in the maintenance of sanitary conditions. The cooking and conversion equipment is provided with direct or indirect steam, propeller agitation, and cooling coils.

Rye. In the preparation of a bourbon mash, rye is not always subjected to the corn cooking process. However, rye undergoes liquefaction at a much lower temperature than corn. This avoids thermal decomposition of critical grain constituents adversely affecting the final flavor of the distillate. Rye is often mashed separately.

Corn. Although the starch in corn converts easily, higher cooking temperatures are needed to make the starch available. Usually malt is not added at the beginning; one-half percent premalt may be added before cooking, preferably at around 66°C, to reduce viscosity. Thin stillage (residual dealcoholized fermented mash from the distillation process) is added by some producers to adjust pH to

5.2–5.4. For cookers operating at atmospheric pressure, a mashing ratio of 95–115 L (25–30 gal) of slurry (grain, water, and stillage mixture) per 0.03 m^3 (1 bushel) and a holding time of 30 min at 100°C is preferable. The mash is cooled to 67°C and malt is added.

Conversion. Primary conversion refers to the saccharification taking place during conversion and is in the order of 75–85% of the available starches. The remainder of the conversion to fermentable sugar takes place during the fermentation process and is referred to as secondary conversion. For batch cooking under pressure, only 83–99 L (22–26 gal) of water/bushel are drawn, and the maximum temperature is 120–152°C. In continuous pressure cooking, water is drawn at a ratio of 30 L/m^3 (24 gal/bu) of meal and sufficient thin stillage is added to adjust the pH to 5.2–5.4. The mash is pumped through the continuous pressure cooker, where it is exposed to temperatures of 170–177°C for 2–6 minutes, and then into a flash chamber, where it is cooled immediately to the malting (conversion) temperature of 63°C. A malt slurry is continuously introduced and the mixture proceeds through the water cooling system to the fermentors.

Particle size and cooking condition for the grain slurry vary depending on the type of distilled spirit that is to be produced. In the case of corn grain fermentations, distillers use small size, high temperature, and low beer gallonage (higher starch concentrations) for neutral spirits production at 120–170°C and 76–91 L/0.03 m^3. Bourbon distillates call for low temperatures (100–150°C) and thinner mash of 95–115 L/0.03 m^3 (saccharified starch slurries) out of flavor considerations. (0.03 m^3 is approximately a bushel).

Saccharification enzymes shorten (liquefy) the cooked starch paste by randomly hydrolyzing α-1,4 linkages. α-Amylase [*9000-85-5*] enzymes are the principal catalysts of this activity. β-Amylase [*9000-91-3*] successively forms two unit maltose sugar from the starch chain pieces. α-1,6 Linkages within the starch molecules are cleaved by limit dextrineases from malt; glucoamylase of fungal origin can hydrolyze both α-1,4 and 1,6 linkages, splitting off one unit of glucose sugar. In all cases, the 1,6 breakdown proceeds slower than 1,4 breakdown.

The catalytic activities of enzymes are optimized within pH values of 4.8–5.2. Temperatures of 60–65°C are commonly employed to secure good conversion, prior to addition of yeast.

Fermentation. The saccharified grain mash is cooled to around 20°C prior to setting the fermentor and inoculation with yeast. It is general practice to dilute the hot grain mash to its final solids concentration by adding backset stillage or water. Stillage is screened dealcoholized fermented grain beer, taken from the bottom of the alcohol distillation beer still. The use of backset stillage offers the distiller water conservation, nutrient supplements, pH adjustment of the fermentation, and a medium that inhibits the formation of by-products, such as glycerol.

Selected yeast strains of *Saccharomyces cerevisiae* are used to inoculate the mash. Two to four percent (v/v) is a minimum for bourbon, which represents over four million cells per milliliter of mash. During fermentation the cells grow in number via budding and the final counts are increased a minimum of 100-fold.

Yeast (qv) metabolize maltose and glucose sugars via the Embden-Meyerhof pathway to pyruvate, and via acetaldehyde to ethanol. All distillers' yeast strains can be expected to produce 6% (v/v) ethanol from a mash containing 11% (w/v) starch. Ethanol concentration up to 18% can be tolerated by some yeasts. Secondary products (congeners) arise during fermentation and are retained in the distillation of whiskey. These include aldehydes, esters, and higher alcohols (fusel oils). Naturally occurring lactic acid bacteria may simultaneously ferment within the mash and contribute to the whiskey flavor profile.

A typical bourbon fermentation continues for 72 hours at a fermentation temperature within the 31–35°C range. Many fermentation vessels are equipped with agitation and/or cooling coils that facilitate temperature control. Significant increases in yeast numbers occur during the first 30 hours of fermentation. Over 75% of the carbohydrate is consumed and converted to ethanol. Within 48 hours, 95% or more of the ethanol production is complete.

The finished beer (final grain residue alcohol mixture) is ultimately agitated to resuspend its solids, and transferred to the beer well storage vessel for holding until it is pumped to the beer still. Distillers try to minimize aeration at this point to avoid formation of excessive aldehydes.

From the beer well, the residue–alcohol suspension passes through a preheater where it is warmed by heat transfer from the vapors leaving the still. The preheated beer is then ready for distillation. The condensate from the preheater is returned to the beer still.

German Mashing Process. In this process, less water is used during the cooking and conversion procedure. A mashing ratio of 84 L of water per 0.03 m^3 of grain mash is used. As the heat of fermentation builds up, cold water is added to maintain the desired fermentation temperature of 31–32°C. The water is added at intervals to the fermentor until the final beer gallonage is reached for distillation in the approximate range of 133 L per 0.03 m^3. The amount of added water will depend on the individual distiller and the initial set temperature of the fermentor.

This system has many advantages. It can produce distillates of different flavors. The process conserves energy in the cooking operation because there is less water to heat and less cooling required to lower the mash to conversion temperature. Water conservation means less pumping and lower sewer discharge volumes. The process yields increased capacity of the plant at the mashing stages as well as fewer capital requirements for heat exchangers.

The converted mash is pumped to a clean sterilized fermentor and the yeast inoculum is added. The set temperature range for whiskey fermentation of 72 hours is usually 17–21°C. At the beginning, the mash converted composition is approximately 80% sugars, mainly maltose and some (<1%) dextrose (primary conversion). The pH is adjusted to reduce initial bacterial growth. Grain neutral spirits are usually set at 27–29°C to expedite fermentation. Temperatures above 35°C inhibit yeast reproduction and promote rapid bacterial growth. Above 40°C actual yeast kill occurs.

After 30 hours, the maximum and critical fermentation is underway and the pH must remain above 4.0 for optimal fermentation. However, accompanying bacterial contamination from various sources such as yeast contamination, improper cleaning procedures, slow yeast growth, or excessive temperatures can result in a pH below 4.0. The remaining amylase enzymes, referred to as secondary conversion agents, are inactivated and can no longer convert the dextrins to maltose. Under these circumstances, the fermentor pH continues to drop because of acid production of the bacteria, and the pH can drop to as low as 3.0. The obvious result is a low ethanol yield and quality deterioration.

Distillation. Distillation separates and concentrates the alcoholic products of yeast fermentation from the fermented grain mash (3). In addition to the alcohol and the desirable congeners, the fermented mash contains solid grain particles, yeast cells, water-soluble proteins, mineral salts, lactic acid, fatty acids, and traces of glycerol and succinic acid. Although a great number of different distillation processes are available, the most common systems used in the United States include: the continuous whiskey separating column (beer still), with or without an auxiliary doubler unit for the production of straight whiskeys; the continuous multicolumn system used for the production of grain neutral spirits; and the batch rectifying column and kettle unit, used primarily in the production of grain neutral spirits that are subsequently stored in barrels for maturation purposes. In the batch and extractive distillation systems, the head and tail fractions are separated from the product resulting from the middle portion of the distillation cycle.

Absorptive distillation, involving the addition of water to the upper section of a column in the whiskey distillation system, is a method of controlling the level of heavier components in a product.

In the beverage distillation industry, stills and auxiliary piping are generally fabricated of copper, although stainless steel is also used. All piping that conveys finished products is tin lined copper, stainless steel, or glass.

Whiskey Distillation. The whiskey column consists of a cylindrical shell divided into three sections: stripping, entrainment removal, and rectifying. The stripping section contains from 14 to 21 perforated plates, spaced 56–61 cm apart. The sieve holes are usually 1–1.25 cm in diameter and take up about 7–10% of the plate area. The vapors from the bottom of the still pass through the perforations with a velocity of 6–12 m/s. The fermented mash is introduced at the top of the stripping section and then passes from plate to plate through descending pipes until it reaches the base where the residual mash is discharged.

Whiskey stills are usually fitted with entrainment removal sections that consist of a plate above the top stripping plate to remove particles trapped in the vapor.

The rectifying section contains three or four bubble cap (wine) plates in the top section of the still to produce distillates up to 160° proof. Whiskey stills are usually made of copper, especially in the rectifying section, which often yields a superior product. Additional copper surface in the upper section of the column may be provided by a demister, a flat disk of copper mesh. Stainless steel is also used in some stills.

Steam is introduced at the base of the whiskey column through a sparger. Where economy is an important factor, as in a fuel alcohol plant, a calandria is employed as the source of indirect heat. The diameter of the still, number of perforated and bubble cap plates, capacity of the doubler, and proof of distillation are the critical factors that largely determine the characteristics of a whiskey.

Bourbon Distillation. The basic distillation system for the production of bourbon and other straight whiskeys consists of a beer still and a beer heater, thumper, or doubler (Fig. 4). The whiskey still consists of between 14 and 21 stripping trays. The upper portion of the still is fitted with either a bubble cap section or a section packed with copper rings to enhance the removal of unwanted flavors and ethyl carbamate precursors. The reduction of carbamate precursors

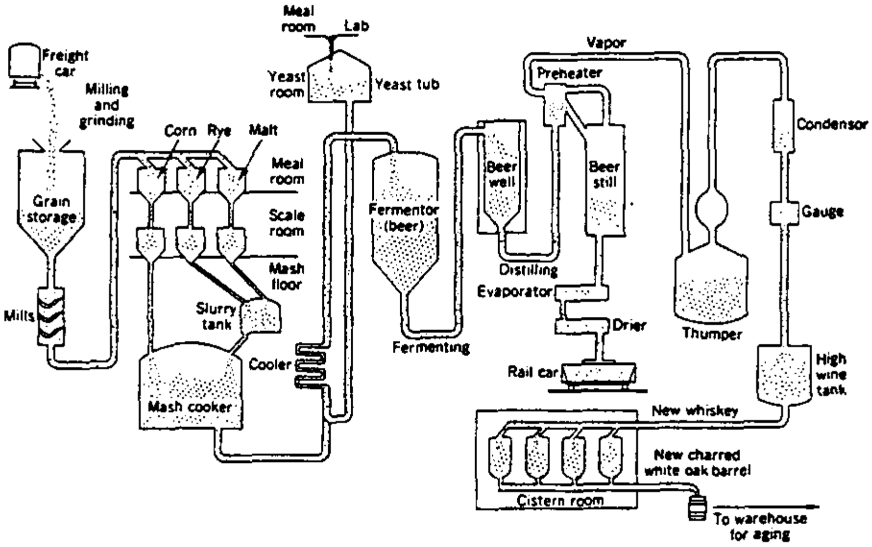

Fig. 4 Distilled beverage plant process flow sheet.

requires strict adherence to a cleaning protocol with a 5% caustic solution as often as twice a week.

Grain Neutral Spirits Distillation. The distillation system for the production of grain neutral spirits usually consists of a whiskey separating column, an aldehyde column (selective distillation column), a product concentrating column (alcohol or rectifying column) from which the product is drawn, and a fusel oil concentrating column. In addition, some distillers may include an aldehyde concentrating column (heads concentrating column), or a fusel oil stripping column.

A fermented mash (generally 90% corn and 1–5% barley malt) with an alcohol concentration of approximately 7% (v/v) is pumped into the whiskey column for stripping somewhere between the thirteenth and nineteenth perforated plate. The spent beer is discharged at the base and pumped to the feed recovery plant where it is separated into stillage (backset) and by-products. The overhead distillate, ranging in proof from 105° to 135°, is fed to the selective distillation column (aldehyde column), which has over 75 bubble cap plates. The main stream (10–20° proof) from the selective distillation column is pumped to the product concentrating column. A heads draw (aldehydes and esters) from the condenser is pumped to the heads (aldehyde) concentrating column, and a fusel oil and ester draw is pumped to the fusel oil concentrating column. The product is withdrawn from the product concentrating column.

By-Products. After the removal of alcohol, the fermentation residues are processed to produce distillers grains. These residues consist of proteins, fats, minerals, vitamins, and fiber that are concentrated threefold by removal of the starch. Distillers grains are usually divided into one of four groups including distillers dry grains (DDG), distillers dry solubles (DDS), distillers dry grains with solubles (DDG/S), and condensed distillers solubles (CDS).

Distillers grains are often classified as light or dark. Processing the whole stillage (5–8% solids) yields DDG/S (dark grains). The light fraction results from screening out the coarse material to yield thin stillage which is concentrated by centrifugation and/or evaporation to give the CDS. DDS are produced by spray or drum drying the CDS.

Typical composition for corn DDG/S is 26–28% protein, 14–20% fat, 11–16% fiber, and 6–9% moisture; for DDS it is 25–30% protein, 15–20% fat, 5–8% fiber, and 5–9% moisture.

The annual production of DDG is over one million tons. Most of it is used in animal feed. However, increasing quantities are being sold as food ingredients because of its excellent nutrient and fiber content.

Maturation

The oak barrels used for aging distilled spirits play a significant role in determining the final aroma and flavor of the beverage. Newly distilled whiskey is colorless, grainy, and harsh. The new whiskey undergoes many types of physical and chemical changes in the maturation process that smooth it out and give it character. These changes include extraction of the wood compounds, decomposition and diffusion of the wood macromolecules into the alcohol, reactions of the wood and distillate components with each other, and diffusion through the wood and evaporation of components.

Much work has been reported and summarized in the literature on the maturing of various whiskeys in charred or uncharred white-oak barrels (4–7). The early literature indicates that total acids, aldehydes, esters, solids, and color increased with aging time and that their concentrations were inversely proportional to proof. Thus aging at higher proofs (over 127°) yields less color and flavor. The maximum allowable entry proof for straight whiskeys was increased from 110° to 125° by the U.S. Treasury Department in 1962.

It has been shown that aromatic aldehydes including vanillin, syringaldehyde, coniferaldehyde, sinapaldehyde, and ethyl lignin come from charred wood, the length of maturing directly affects the amount of aldehydes formed, the lower proof spirits have more aldehydes than higher proof spirits do, and the used and new uncharred barrels produce about one-third of the aromatic aldehydes found in new charred barrels (Table 3) (8).

Table 3. Concentration of Aromatic Aldehydes in Bourbon as Influenced by Barrel Treatment, Age, and Proof

Barrel treatment	Age, yr	Proof[a]	Concentration, g/100 L at 100 proof[a]
reused	3.5	136.6	0.33
	6.0	137.0	0.27
new charred	4.0	107.6	0.88
	1.5	140.8	0.77
	6.0	136.7	0.29
reused, recharred	3.5	136.0	0.48

[a]100 proof = 50 vol % alcohol.

It has also been proposed that under the acidic conditions found in whiskeys, ethanol reacts with lignin (qv) to reduce an alcohol-soluble form of lignin (ethanol lignin). This can be converted into coniferyl alcohol, which can be oxidized to coniferaldehyde. The partial oxidation of ethanol lignin can produce sinapic and coniferyl alcohols that can be converted to syringaldehyde and vanillin, respectively (8).

Age versus congener development in bourbon stored for eight years has been studied (9). Results indicate a high correlation of congener formation with age (Table 4).

Table 4. Statistical Analysis of Age Versus Congener Formation in Bourbon

Property/Substance	Correlation coefficient[a]
total acids	0.699
esters	0.751
fusel oil	0.378
aldehydes	0.679
total color	0.686
organic-soluble color	0.672
solids	0.750
ethyl acetate	0.679
n-propyl alcohol	0.188
isobutyl alcohol	0.276
isoamyl alcohol	0.403
sodium	0.054
potassium	0.662
calcium	0.484
magnesium	0.102

[a]Correlation coefficients above 0.403 are significant.

It has been found that the cloudiness in whiskey could be caused by certain steroids extracted from the wood during aging. These include β-sitosterol-D-glucoside, β-sitosterol, stigmasterol, and campesterol (10). The monosaccharide composition of whiskey aged up to 12 years also has been followed. Arabinose, glucose, xylose, rhamnose, and galactose increased throughout the 12 years of aging while fructose increased linearly for six to seven years. This indicates that the sugars found in aged whiskey are derived from the free sugars in the wood and/or the hydrolysis of hemicellulose (qv).

A study of the effect on the congener levels of up to eight refills of a used whiskey barrel indicate that volatile acids, esters, colors, solids, and tannins showed the greatest percentage change between the first and second use of a new charred barrel (11).

The amounts of color, solids, fixed acids, and tannins found in whiskey aged for two years were found to be greater in recharred barrels than in new barrels reused once. Both these levels were less than the amounts found in new charred barrels, indicating that some congener precursors are depleted as the barrels are reused and/or recharred. The same study indicates that congeners increased with storage temperature. The principal congeners increased between 2% and 3% per degree centigrade over a range of 18–23°C.

The lignin and tannin contents of oak that had been in contact with both whiskey and air also has been studied (12). The lignin and tannin contents were 7 to 26 times lower at the whiskey surface versus the air surface. The aromatic aldehyde content was 8–15 times greater at the whiskey surface. New barrels contributed over two times more lignins and tannin to whiskey than the used barrels. Aldehydes were four times greater in whiskey aged in new barrels versus used barrels. When two 15-year-old whiskeys were analyzed, one characterized as light and the other heavy and flavorful (Table 5), the compounds derived from wood were significantly higher in the more flavorful whiskey, demonstrating the important contribution that wood makes to the flavor of whiskey.

Table 5. Concentration, mg/L, of Wood Components in Light and Heavy 15-Year-Old Whiskey

Property	Light	Heavy
tannins	192.0	540.0
lignin complex	515.0	1431.0
vanillin [*121-33-5*]	4.3	13.6
syringaldehyde [*134-96-3*]	8.7	29.9
coniferaldehyde [*458-36-6*]	0.8	5.7
sinapaldehyde [*4206-58-0*]	0.78	3.5
total aldehydes	14.58	52.7

Alcohol Reduction

Pervaporation. Vapor arbitrated pervaporation is used to remove ethanol from whiskey by selective passage of the alcohol through a membrane. Whiskey flows on one side of a membrane. A water-vapor stream flows on the other side and sweeps away the ethanol that permeates the membrane. Thus alcohol reduction and selective retention of flavor and aroma components can be achieved using membranes with a particular porosity. The ethanol can be recovered by condensing or scrubbing the vapor stream. Pervaporation systems operate at or slightly above atmospheric pressure (Fig. 5).

In this process the addition of water vapor to the sweep stream can be controlled so that the water activity of the gas phase equals that of the beverage. When this occurs, there is no transport of water across the membrane. The water content of both the beverage feed and the sweep stream is kept constant. These conditions must be maintained for optimum alcohol reduction. The pervaporation system controls the feed, membrane, airstream moisture level, and ethanol recovery functions. An operational system has been developed (13).

The feed system handles the storage, circulation, and temperature control of the whiskey. Since permeability increases with temperature, and considering the heat stability of whiskeys, it is desirable to operate the system above ambient temperatures. Operating at higher temperatures facilitates temperature control of the process, since heat losses can be compensated by the addition of heat.

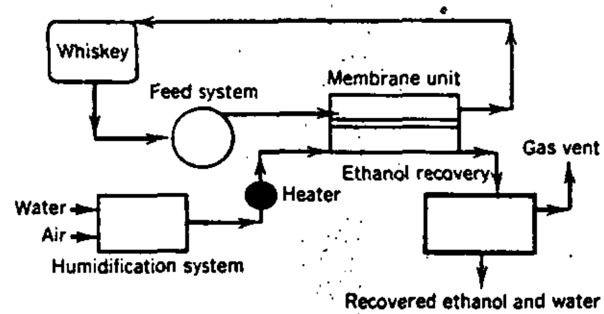

Fig. 5 Alcohol reduction pervaporation system.

The membrane system consists of multiple plate and frame stacks holding the thin-film composite membranes clamped together. The system capacity is increased by increasing the number of plates.

A humidification subsystem controls the temperature, flow rate, and relative humidity of the sweep stream. Air and water can be fed to a liquid-gas packed contactor to produce the desired moisture level in the vapor stream. The saturation temperature controls the water loading of the air which can be heated to give exactly the desired relative humidity.

Ethanol removed by the vapor stream can be recovered by condensation, vapor recompression, or scrubbing. In the first two methods, the concentration of the recovered ethanol depends on the relative humidity of the sweep stream and the ratio of sweep and permeation rates. In scrubbing, the rate of water delivery to the liquid-gas contactor affects the ethanol concentration in the recovered stream.

Pervaporation has been successfully used both to reduce the alcohol content and to concentrate the congeners in bourbon whiskey (Table 6). The resulting products had taste, aroma, and color characteristics similar to the original bourbon but at a substantially higher intensity, reflecting the effect of the alcohol reduction and congener concentration.

Table 6. Alcohol Reduction Using Pervaporation Process

	Example 1	Example 2
initial whiskey volume, mL	1160.0	1410.0
initial alcohol concentration, %	65.3	59.7
final whiskey volume, mL	670.0	940.0
final alcohol concentration, %	41.9	38.8
processing time, h	2.6	3.1
whiskey temperature, °C	30.0	30.0
whisky flow rate, mL/min	500.0	420.0
sweep air temperature, °C	30.0	30.0
sweep air flow rate, L/min	22.5	22.5
relative humidity of sweep airstream, %	77 ± 2	80 ± 2
ethanol flux, 10^{-3} mL/(cm^2·h)	92.0	77.0
water flux, 10^{-3} mL/(cm^2·h)	6.9	2.4
ethanol concentration in condensed permeate, %	80.0	79.9

Reverse Osmosis. A reverse osmosis (RO) process has been developed to remove alcohol from distilled spirits without affecting the sensory properties (14). It consists of passing barrel-strength whiskey through a permeable membrane at high pressure, causing the alcohol to permeate the membrane and concentrating the flavor components in the retentate.

The one-pass system consists of a feed tank, filter, pump, and membrane system (Fig. 6). The feed tank contains whiskey at approximately 100° proof. It is filtered through a cellulose filter and then pumped into the membrane system where the separation takes place. Dupont B-10 Aramid hollow fiber membranes are used in series or parallel and are able to withstand the high pressures, 689–1034 kPa (6.8–10 atm), necessary to achieve separation.

Operational temperatures of 4–27°C are maintained. In this process the flavor components are concentrated in the retentate. A reduced alcohol product is obtained by adding back water to give the desired flavor impact. Typical gas chromatographic results, comparing unprocessed 80° proof whiskey with reverse osmosis processed 54° proof whiskey and diluted 54° proof whiskey, indicate good congener retention in the alcohol-reduced (RO) processed whiskey (Table 7).

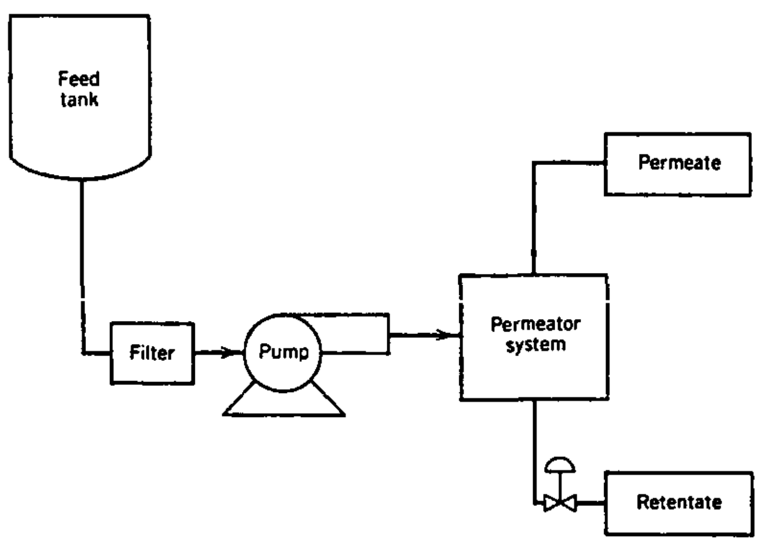

Fig. 6 High pressure reverse osmosis system for alcohol reduction.

Seagrams has used this process to produce Mount Royal Light and V. O. Light, both 54° proof, full-flavored products that are currently in test markets in the United States and Canada, respectively.

Analytical Procedures

Analytical results of distilled spirits are expressed either by chemical class or by individual constituent. When these results are expressed by chemical class, the most prevalent constituent within that class is used as the marker, eg, acetic acid for acids, acetaldehyde for aldehydes, and ethyl acetate for esters. Wet chemical

Table 7. Congener Concentrations, mg/100 mL, of Unprocessed, RO Processed, and Water Diluted Whiskeys

Congener	CAS Registry Number	Whiskey sample		
		Unprocessed[a]	RO processed[b]	Water diluted[b]
acetaldehyde	[*75-07-0*]	1.08	1.08	0.72
methanol	[*67-56-1*]	2.48	1.59	1.65
ethyl acetate	[*141-78-6*]	8.76	8.45	5.84
n-propanol	[*71-23-8*]	2.88	2.84	1.92
isobutyl alcohol	[*78-83-1*]	7.00	6.91	4.67
n-butanol	[*71-36-3*]	0.04	0.05	0.03
ethyl proprionate	[*105-37-3*]	0.20	0.19	0.13
acetal	[*1820-50-4*]	0.72	0.32	0.48
isoamyl alcohol	[*123-51-3*]	15.44	15.09	10.29
amyl alcohol	[*71-41-0*]	6.16	5.99	4.11
isoamyl acetate	[*123-92-2*]	0.037	0.036	0.024

[a]80 proof = 40 vol %.
[b]54 proof = 27 vol %.

methods are employed in the determination of results by chemical class, while more advanced and refined techniques are employed in the determination of individual chemical constituents.

Alcoholic beverages are made up primarily of ethanol, congeners, and water. Congeners are vaporized with the alcohol in distillation below 190° proof and are developed during the maturation process by oxidation and other reactions. These components contribute to palatability and create the characteristic appearance, aroma, and taste of a particular spirit. When the spirit is distilled at a lower proof, more congeners are present and the spirits possess more character. Congeners are usually reported either as grams per 100 liters at "as is" proof, or as 100° proof at parts per million or parts per billion.

Distilled spirits are governed by the Bureau of Alcohol, Tobacco, and Firearms regulations. Every bottle of distilled spirits must contain a specified percent of alcohol or proof as stated on the label. Proof is the ethyl alcohol content of a liquid at 15.6°C, stated as twice the percent of ethyl alcohol by volume.

The proof content is determined by the use of a standardized hydrometer with a standardized thermometer. The alcohol content can also be determined by the use of an immersion refractometer, a pycnometer, or a density meter.

In order to determine the true proof of a distilled spirit sample, it must not contain solids. However, if the sample contains less than 600 mg/100 mL of solids, an obscuration factor can be used; it is added to the apparent proof. For example, one hundred milligrams of solids per 100 milliliters reduces the apparent proof by 0.4 of 1 degree of proof. If the sample contains more than 600 milligrams of solids, it must be distilled.

The actual proof must not be more than the stated proof or no less than 0.3 below the stated proof for solids less than 600 mg/100 mL, and no less than 0.5 below the stated proof for solids greater than 600 mg/100 mL. The actual alcohol content must be given to the nearest 0.5%.

The analysis of individual chemical constituents in distilled spirits currently is performed using gas chromatography (gc) and high pressure liquid chromatography (hplc). Although other types of instrumental analyses have yielded much information regarding the chemical constituency of distilled spirits, the combination of gc and hplc has allowed hundreds of different chemical components of distilled spirits to be individually identified and accurately quantified.

The most common chromatogram in the distilled spirits industry is the fusel oil content. This consists of *n*-propyl alcohol, isobutyl alcohol, and isoamyl alcohol. Other common peaks are ethyl acetate, acetaldehyde, and methanol. The gc columns may be steel, copper, or glass packed column or capillary columns. Additional analyses include determinations of esters, total acids, fixed acids, volatile acids, solids or extracts (used to determine obscuration), tannins, and furfural.

The high degree of sensitivity, selectivity, and efficiency of gas chromatography allows the elucidation of a complete profile of the volatile components of distilled spirits. The wide selection of chromatographic columns and techniques, such as gc-ms, gc-ftir, and gc-ms-ftir, has allowed the chemist to routinely identify and quantify individual constituents on a parts-per-billion level. The two most critical variables in the analysis of volatile components of distilled spirits by gas chromatography are the selection of a suitable chromatographic column and of the most appropriate detector.

Gas chromatographic analysis of distilled spirits has historically been performed using glass or metal columns supporting a Carbowax packing. This type of analytical system is still commonly used for quality control and routine measurements. Currently, gc analysis of distilled spirits has moved toward capillary gas chromatographic techniques. A narrow bore-fused silica tube lined with a specific coating serves to effect extremely efficient separation of the constituents of distilled spirits. The most commonly used chromatographic columns in these analyses are the DB-1, the DB-5, and the Carbowax columns.

The most favored gas chromatographic detectors for the analysis of distilled spirits are the flame ionization detector (FID) and the mass spectrometer detector. The flame ionization detector employs a hydrogen flame for the combustion of organic substances to produce electrons and ions that are collected on an anode. The resulting electrical current is proportional to the amount of the material burned. Mass spectrometric detection employs an electron beam to cause fragmentation of the chromatographic effluent. The fragments are collected and compared to the individually specific fragmentation patterns of known compounds.

Hplc techniques are used to routinely separate and quantify less volatile compounds. The hplc columns used to affect this separation are selected based on the constituents of interest. They are typically reverse phase or anion exchange in nature. The constituents routinely assayed in this type of analysis are those high in molecular weight or low in volatility. Specific compounds of interest include wood sugars, vanillin, and tannin complexes. The most common types of hplc detectors employed in the analysis of distilled spirits are the refractive index detector and the ultraviolet detector. Additionally, the recent introduction of the photodiode array detector is making a significant impact in the analysis of distilled spirits.

Advances in the technology of chemical analysis and the ability to analyze for trace amounts of complex compounds now make it possible to combine analytical information with sensory analysis to identify taste characteristics and facilitate process control.

Health and Safety Factors

Ethyl Carbamate. In November 1985, the Canadian Government indicated that it had detected ethyl carbamate [*51-79-6*] (urethane), a suspected carcinogen, in some wines and distilled spirits. Since that time, the U.S. distilled spirits industry has mounted a serious effort to monitor and reduce the amount of ethyl carbamate (EC) in its products. In December 1985, the Canadian Government set limits of 150 ppb in distilled spirits and 400 ppb in fruit brandies, cordials, and liqueurs. The FDA accepted a plan in 1987 from the Distilled Spirits Council of the United States (DISCUS) to reduce ethyl carbamate in whiskey to 125 ppb or less, beginning with all new production in January 1989.

Ethyl carbamate, $C_3H_7NO_2$, is developed naturally during the fermentation of alcoholic beverages. It also appears in foods such as bread and yogurt. Since ethyl carbamate is not easily distilled, its formation most likely involves a distillable precursor. The mechanism of ethyl carbamate formation probably involves cyanate produced from the oxidation of cyanide or from urea-based compounds in the beer. Cyanate reacts with alcohol to form ethyl carbamate as follows:

$$CH_3CH_2OH + HOCN \longrightarrow CH_3CH_2O{-}\overset{\overset{\displaystyle O}{\|}}{C}{-}NH_2$$

There are at least three gc methods employed by the ATF, industry, and the government to detect ethyl carbamate.

Distillers employ a somewhat unique process to make various products and have tailored approaches to control and reduce ethyl carbamate to their own particular process. Some of the methods used are the use of copper packing in the rectifying section of stills, increased frequency of cleaning stills and other equipment, and using a cool-down period in the cleaning procedure. Increased rectification also reduces ethyl carbamate. Keeping the system clean is critical to minimizing ethyl carbamate.

Based on an agreement with the FDA, all distilleries report their ethyl carbamate values by plant on a quarterly basis through DISCUS. Most distilleries always have been under the 125 ppb limit. Others, by controlling their process and by making some of the modifications mentioned above, have been able to reduce the concentration well below the limit (20–50 ppb). Although more remains to be done in the area of ethyl carbamate formation mechanisms, much progress has been made in monitoring, controlling, and significantly reducing ethyl carbamate in distilled spirits.

Packaging

Packaging for distilled spirits intended for domestic distribution is regulated by the Federal Bureau of Alcohol, Tobacco, and Firearms (ATF). This strict supervision establishes acceptable container size, labeling, and sealing requirements, as well as the disclosure of information on the shipping container. Furthermore, local and state distilled spirits' labeling and packaging requirements must also be met.

Several changes have occurred in these areas over the past few years. The addition of a health warning statement for each alcoholic beverage container and the change from proof to percent alcohol by volume has necessitated numerous label changes. Many distillers have also modified, redesigned, or changed their package to eliminate the Federal strip stamp that has long been commonplace for all distilled spirits. Most distillers have opted for a tamper evident closure, which has required changes to bottle molds and glassware, whereas some distillers are utilizing a tamper evident shrinkband to replace the strip stamp. These changes have been implemented primarily to comply with the regulations, lower the cost, and simplify the packaging of beverage alcohol.

The standard legal sizes, as outlined by the ATF for domestic distribution, are 1.75 L (59.2 fluid ounces), 1 L (33.8 fluid ounces), 750 mL (25.4 fluid ounces), 375 mL (12.8 fluid ounces), 200 mL (6.8 fluid ounces), 100 mL (3.4 fluid ounces, which is approved by a limited number of states), and 50 mL (1.7 fluid ounces). Individual states continue to limit the number and size of containers that are distributed within their jurisdiction. In some cases these do not coincide with all sizes available and authorized by the ATF.

In the past few years many changes have occurred in the packaging materials utilized for distilled spirits. Traditionally, distilled spirits have been packed primarily in glass containers of approved ATF sizes. Over the last 5–10 years, plastic containers, primarily poly(ethylene terephthalate) (PET), have been utilized by increasing numbers of distillers. Because of environmental concerns, the last two years have seen a change back to glass on some of these package sizes. However, the 50 mL miniature bottle continues to be primarily packed in PET plastic containers.

Most recently, greater concern has been placed on the increased use of bar codes. The basis for all bar code systems is the Uniform Product Code or UPC. Strict guidelines were developed by the Uniform Code Council and are enforced by the retail outlets. The purpose for the bar codes is for electronic data scanning and computerized pricing at the retail outlets. Recently, more and more emphasis is being placed on the use of bar codes throughout the distribution network. Shipping Container Symbology or SCS bar codes are now being requested on each case of distilled spirits.

A spinoff from the UPC bar code is the European Article Numbering (EAN) bar code system. EAN numbers are based on the UPC bar code guidelines. For distribution outside North America, these bar codes are unique and different in the number of characters per code and the computer data base related to each code.

In addition to the EAN bar code that appears on the consumer salable package, an EAN dispatch bar code has been established, similar to the SCS bar code, for shipping containers. The dispatch bar code on the shipping containers enable distributors to scan individual cases throughout the distribution network to assure that the proper products are being selected and shipped, as well as serving as an inventory control. Eventually, the use of these bar code systems will enable electronic data interchange for ordering and releasing. These systems are already in place in food grocery systems as well as consumer durable goods distribution networks. The distilled spirits industry is challenged with developing economical ways of complying with these new bar code system requirements.

Deposit refund labeling is another requirement recently introduced to the distilled spirits industry. Currently, legislation in Iowa, Vermont, and Maine requires distillers to comply with labeling requirements to disclose the deposit refund value for each state on their label. Other states are considering similar forms of deposit refund legislation for distilled spirits. This will offer another challenge for the packaging of distilled spirits. The environmental concerns extend further than deposit refunds and offer the greatest packaging challenge in the near future. Packaging components that are produced and supplied to distillers should adhere to Environmental Protection Agency (EPA) guidelines. These guidelines limit the intentional use of heavy metals for the production of packaging materials. The use of inks, coatings, and other materials for packaging of distilled spirits must not contain more than 5 ppm of defined heavy metals such as lead, mercury, selenium, chromium, silver, and cadmium. Additionally, no trace amounts of these elements should be found in the product itself. This requirement has nearly eliminated the use of crystal decanters or ceramics that were commonly used for decorative package sales in the past.

The packaging of distilled spirits has become more complex with both the demands for higher efficiencies of the production facilities within the industry, and the request by marketing and sales departments for higher flexibility to provide customers with greater value on products.

The distilled spirits industry has an ongoing development of new products and line extensions using more diverse and less traditional types of packaging materials. In addition, there is a significant thrust on reducing the weight of all glass and plastic packaging to lower the cost of goods and shipping.

Flavor Applications

Flavoring beverage alcohol products presents some interesting challenges since ethyl alcohol itself has flavor. In high proof beverages (ie, over 80°), which contain pure alcohol, and not whiskey or rum that have inherent flavor characteristics, flavors must be added to help smooth out the harshness and singularity of the flavor profile. At proofs below 10°, the ethyl alcohol flavor is often below optimum sensory profiles. Flavors can be used to supplement a desired aroma profile. The same flavor has different sensory characteristics at various proof levels. Higher concentrations of flavor are generally required in higher proof products to compete with the ethyl alcohol flavor. The sources and types of distilled spirits (brandy, whiskey, rum, and spirits) have various analytical profiles that combine differently with specific flavors. It is not unusual to find certain characteristics of a flavor out of balance when applied to a base that contains the same chemicals. When organic-based flavors are applied to an ethyl alcohol base, there is the potential for several chemical reactions. Ethyl alcohol reacts with aldehydes to form the corresponding acetal. This reaction occurs more readily at higher proofs and in acid conditions such as those found in most cordials. This is in many cases a desirable reaction and was described years ago as "marrying."

Since the acetal exists in equilibrium with the aldehyde, it is possible for the aldehyde to be released when water is added in a mixed drink, changing the balance and giving a burst of freshness to a mixed drink. Ethyl esters of terpene alcohols in citrus oils and other botanicals, plus the ethyl esters of fatty and volatile acids, are formed during prolonged exposure to ethyl alcohol. Certain beverage alcohol products that need to contain milk, eggs, or other protein containing materials must be developed carefully and the added flavors must be considered to prevent the precipitation of the protein and separation of the product.

Most flavors that are designed for beverage alcohol products use ethanol as the primary solvent for the flavor. Glycerol [*56-81-5*], propylene glycol [*57-55-6*], and water are other common solvents in liquid flavors. Some beverage alcohol concepts require the addition of an emulsified flavor, either as a vehicle to solubilize the oils in the beverage or as a deliberate attempt to cloud the product. This can best be accomplished at lower proofs with the alcohol breaking the emulsion.

Many beverage alcohol products depend heavily on the addition of compounded flavors, distillates, percolates, and extracts to carry the organoleptic profile of the product. Cordials, liqueurs, and schnapps at various proofs, such as Creme de Cocoa, Peppermint Schnapps, fruit-flavored cordials and schnapps, and spirit coolers are examples. Flavored whiskeys, rums, and brandies often contain blenders and merger flavors which assist in diminishing the flavor variances encountered because of crop and storage conditions. Generally, flavor applications in food and beverages are specific to the type of end product that the flavor is used to enhance. For instance, when flavoring a bakery item, flavors must be able to compensate for high oven temperatures and other inherent flavors present in the food. More strikingly, foods that are extruded must use flavors that can withstand the extreme temperatures used during processing. Likewise, beverage alcohol flavor applications require the consideration of many factors that affect the finished product. These include proof, inherent flavors, added flavors, source of ethanol, and overall composition.

Future Developments

The decline in distilled spirits consumption is likely to continue, but will be somewhat ameliorated by the increased consumer interest in high price premium products and the increased activity in the international markets. For example, Japan is importing significant amounts of American bourbon, a trend that will probably continue.

The trend toward lower proof beverages will also likely continue because of new consumer preferences, cost reduction, and tax savings opportunities. Pressures to improve production efficiencies and lower costs will increase and new technology must play a greater role in this area. Distilled beverages have been produced for several thousands of years and will continue to be consumed in an ever increasing variety of forms and packages.

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BIACETYL. See KETONES.

BILE CONSTITUENTS. See MEAT PRODUCTS.

Dist. Liquors
Sec. #2
Ref. #5

BINARY CYCLES. See POWER GENERATION.

BIOASSAY. See AUTOMATED INSTRUMENTATION; IMMUNOASSAY; MEDICAL DIAGNOSTIC REAGENTS.

BIOCIDES. See INDUSTRIAL ANTIMICROBIAL AGENTS.

BIOCOMPATIBLE MATERIALS. See PROSTHETIC AND BIOMEDICAL DEVICES; SUTURES.

BIODEGRADABLE POLYMERS. See PLASTICS, ENVIRONMENTALLY DEGRADABLE.

BIOGENIC AMINES. See NEUROREGULATORS; OPIODS, ENDOGENOUS.

BIOMASS CHEMICALS. See CHEMURGY; FUELS FROM BIOMASS.

BIOMEDICAL AUTOMATED INSTRUMENTATION. See AUTOMATED INSTRUMENTATION.

BIOPOLYMERS

SURVEY

Biopolymers are the naturally occurring macromolecular materials that are the components of all living systems. There are three principal categories of biopolymers, each of which is the topic of a separate article in the *Encyclopedia:* proteins (qv); nucleic acids (qv); and polysaccharides (see CARBOHYDRATES; MICROBIAL POLYSACCHARIDES). Biopolymers are formed through condensation of monomeric units; ie, the corresponding monomers are amino acids (qv), nucleotides, and monosaccharides, for proteins, nucleic acids, and polysaccharides, respectively. The term biopolymers is also used to describe synthetic polymers prepared from the same or similar monomer units as are the natural molecules.

In addition to being necessary for all forms of life, biopolymers, especially