

In these distillation columns at Schenley's Lawrenceburg distillery, beverage spirits are separated from a fermented mash of grain. Emerging from the still, new whiskey is colorless

and unpalatable, but exact scientific control of processes provides a product that becomes smooth, mellow, and amber colored after storage for several years in charred oak barrels

## Distilled Alcoholic Beverages

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Average yearly production of 175 million proof gallons of distilled beverages, made possible through fermentation processes, provides a sizable market for American grain farmers. In the 20 years since repeal, the alcoholic beverage industry has instituted technological improvements which permit close control of the process and the product

**H**ISTORY AND DEVELOPMENT of distilled alcoholic beverages are difficult to trace in the absence of reliable documentation.

In legend, the innovation of distillation is ascribed to a Greek physician who reportedly noted the condensation of liquid on a plate which had been placed over some heated vegetable to keep it warm while it was served to him. There is, however, no mention of any utilization of his discovery.

There are in existence some documents dating back as far as 800 B.C., mentioning a beverage called arrack, which was supposed to be obtained by distillation from fermented sugar liquor.

Some investigators believe that the old Egyptians knew distillation. If it was practiced by them, it is more than likely that the product was alcohol.

The word alcohol is supposed to be of

Arabian origin; "Al-Cohol" or "Al-Kohal" means a finely powdered or purified substance. Before the word took on its narrower definition, even a finely powdered substance such as wheat flour, would have been considered as "alcoholized." Therefore, the term alcohol was not applied only to distilled spirits, but to all finely divided materials which had been separated from larger particles. Similarly, the word "distill," did not at first mean the separation of a certain vapor from a solution or from another liquid, but merely the separation of a fine powder from larger particles. The word "distill" itself is derived from the Latin "distillo," which means "to drop."

There is hardly a doubt that the presence of alcohol as a combustible ingredient in wine was known to scientists of antiquity such as Aristotle, Hippocrates, and Pliny. Aristotle speaks of a wine

which provides a "spirit." Pliny is much more definite and mentions a "wine which can be ignited." Therefore, the presence of alcohol in certain fermented liquids was most likely known, or at least suspected, but there is no proof or even strong indication that the separation of this flammable ingredient had been successfully carried out in Pliny's time.

The first evidence concerning the separation of alcohol appears toward the end of the 12th century in the writings of an Italian physician named Master Salernus, who died in the year 1167. Somewhat more definite information is given by an ecclesiastic Albertus Magnus, a well known alchemist of his time, who lived in the German city of Regensburg, Bavaria. From then on, distilling must have taken on considerable importance and must have been practiced by many

men in many countries. The main purpose to which the new knowledge was applied was the preparation of healing drafts. Since the physicians of those days were to be found chiefly among the monks, it was natural that the preparation of distilled medicines was largely carried out in monasteries. To this day, the Benedictines and the monks of the Chartreuse have guarded the secrets of the distillation of their brews.

The fascinating historic development of distillation has been expertly described in a book entitled "Short History of the Art of Distillation" by R. J. Forbes, published in 1948 in Leiden, Holland, by E. J. Brill. This "short history" fills 405 pages and has 203 illustrations, and it provides easy and instructive reading for those who are inclined to learn more about the subject.

### Modern Distilled Beverages

Beverages containing alcohol are basically divided into two classes, the first comprising fermented products such as beer and wine, and the second, so-called distilled spirits or alcoholic liquors. Only the latter class is considered here.

Since all alcoholic beverages, and especially distilled spirits, are heavily taxed by federal, state, and local governments, and constitute the source of large incomes of the various government agencies, all these products are under severe and detailed regulations, which are promulgated by the Treasury Department. One of the most vital parts of the government regulations contains the definitions of the various alcoholic beverages in a very detailed chapter, the "Standards of Identity."

The following abbreviated and condensed definition of the governmental standards of identity omits such details as are chiefly important only in the mechanical operation of these laws, and are not essential in a discussion of technical or scientific nature.

Differentiation must be made between products obtained directly from distillation and others obtained by blending or mixing. In the first category belong straight whiskey, brandy, and rum, whereas the so-called blends and the liqueurs form the most important and largest part of the second type of products.

### Products Obtained Directly by Distillation

**Neutral Spirits or Alcohol.** Neutral spirits or alcohol are spirits distilled from any material at or above 190° proof, whether or not such proof is subsequently reduced.

In the United States, and also in Great Britain, the strength of alcoholic beverages is officially stated by "proof." The U. S. Statutes define this standard as fol-

lows: "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 60° F." In other words, the figure for proof is double that of the alcoholic content by volume. In Great Britain, "proof spirit is such as at 51° F. weighs exactly twelve-thirteenths of the weight of an equal bulk of distilled water."

**Whiskey—Straight Whiskey.** Whiskey is the alcoholic distillate that is obtained from a fermented mash of grain (or mixture of grains) and that has the traditional and characteristic taste and aroma associated with whiskey.

The resulting whiskey is designated "rye whiskey," "bourbon whiskey," "wheat whiskey," "malt whiskey," "rye malt whiskey," depending on the predominating type of grain used in the preparation. Not less than 51% of grain of one type must be used in order to justify identifying the product accordingly, and the distillation must be carried out at a proof not exceeding 160. The storage of the distillate must be in charred, new, oak containers.

"Straight whiskey" is defined as "an alcoholic distillate from a fermented mash of grain distilled at not exceeding 160° proof and withdrawn from the cistern room of the distillery at not more than 110° and not less than 80° proof," and it must be aged in charred, new, oak containers for not less than 24 calendar months. But no product may be called simply "straight" in its labeling, and the type of grain utilized must always be stated.

**Brandy.** Brandy is a distillate from the fermented juice of fruit, distilled at less than 190 proof and possessing the characteristics generally attributed to brandy. Without further qualification, the word brandy is always meant to

**Alfred J. Liebmann** has been with the Schenley organization since 1933. His chief fields of



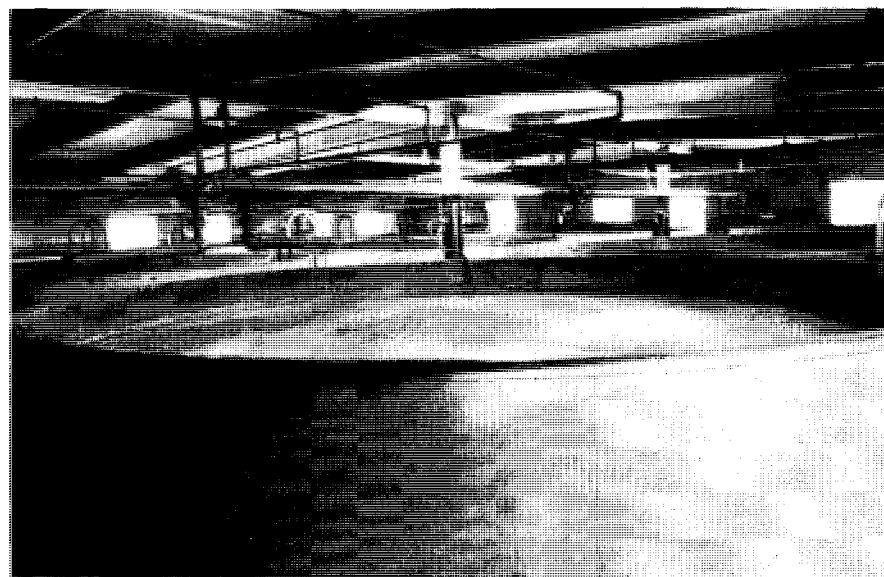
interest are in fermentation, distillation, and antibiotics, although his early career was with manufacturers of electric lamps. Dr. Liebmann was born and educated in Zurich, Switzerland, and came to the U. S. about 1912. During World War II, he served the War Production Board as a

member of the Penicillin Producers Industry Advisory Committee. He has contributed articles to many scientific magazines and is a member on many scientific societies, including the ACS, The Society of American Bacteriologists, and the New York Academy of Sciences.

apply to "grape brandy," or therefore to the distillate obtained from fermented grape juice or wine. If juice from some other fruit has been fermented and distilled, the resulting brandy carries the name of the respective fruit, such as apple brandy and apricot brandy. Still other distinctions are made from geographical origin, such as cognac and cognac brandy, if the product originates in certain provinces of France.

**Rum.** Rum is the distillate from the fermented juice of sugar cane, molasses, or other sugar cane by-products, distilled at less than 190 proof. New England rum is further defined as having been produced in the U. S. and distilled at less than 160 proof. Other rums are distinguished not by type, but by geographic origin, such as Jamaica rum and Virgin Island rum.

**Fermentation room.** A mash normally undergoes 72 to 96 hours fermentation before it is ready for distillation. Vats are filled from overhead pipes.



**Gin.** Gin is a distillate obtained by either an original distillation from a fermented mash or the redistillation of neutral spirits over or with juniper berries or other aromatic botanicals.

### Products Obtained by Blending or Mixing

**Blended Whiskey.** Blended whiskey is a mixture that must contain at least 20% by volume of straight whiskey at 100 proof. In the majority of blended whiskeys, the balance is made up of neutral spirits, but certain blends or blended whiskeys are made up entirely of straight whiskeys, in which case the product will be "a blend of straight whiskeys." If neutral spirits (ethyl alcohol) is used, there must be stated not only the percentage of these neutral spirits—80% or less—but also the name of the commodity from which they have been distilled. The majority of the blends are made with spirits produced from grains, and the designation in this case will be "neutral spirits distilled from grain" or, more briefly, "grain neutral spirits." Some individual states recognize this variety only and require that blends made with spirits from other sources be called "imitation whiskey." The grain shortage during and following World War II resulted in the temporarily increased use of blending spirit produced from sugar cane or molasses ("distilled from cane products"), from grapes or other fruit ("distilled from fruit"), or from potatoes and other tubers ("distilled from vegetables").

In addition to the distinction based on the nature of the grain used in the preparation, there is a distinction based on geographic origin. In this category belong such types as Scotch whiskey, Irish whiskey, and Canadian whiskey. These products are distinctive for the land of origin and are produced in compliance with the laws prevailing there.

**Cordials and Liqueurs.** Cordials and liqueurs are products obtained by mixing or redistilling neutral spirits, brandy, gin, or other distilled spirits with or over fruits, flowers, plants, or pure juices therefrom, or other natural flavoring materials, or with extracts derived from infusions, percolations, or maceration of such materials, and to which sugar or dextrose or both have been added in an amount not less than 2.5% by weight of the finished product. Synthetic or imitation flavoring materials shall not be included.

**Compound Gin.** Compound gin is the product obtained by mixing neutral spirits with distilled gin or gin essence or other flavoring materials customarily used in the production of gin, and deriving its main characteristic flavor from juniper berries and reduced at time of bottling to not less than 80° proof. Mixtures of such products are also included.

### Production

The production of distilled alcoholic beverages is basically divided into two distinct operations—namely, fermentation and distillation.

The fermentation converts the sugar content in the raw material into alcohol, and the distillation separates the alcohol and certain important constituents, characteristic for the desired product, from the unfermentable residue of the raw materials and the nonvolatile fermentation products. In fermentation again, we have to differentiate between saccharine raw materials, which contain directly fermentable sugars (such as glucose, fructose, sucrose), and those which contain compound carbohydrates which are not directly fermentable (such as starch and inulin), and which therefore have first to be converted into a fermentable variety of sugar. (See Table I.)

For many years, practically the only raw material in the production of industrial alcohol was molasses. Only the extraordinary conditions brought about by World War II, especially the threat to shipping and the shortage of tank steamers, caused grains to be used as a source for this basic material. During the critical war period it was urgently needed in the production of munitions, and even more so, in the synthetic rubber industry. For economic reasons, there is very little likelihood that grains will ever be put to this use again.

On the other hand, for beverage purposes, grains are almost exclusively used for alcohol production. While the government definition of "neutral spirits or alcohol" are recognizing "any material" from which they may be distilled, the label requirements demand that in any beverage in which neutral spirits

or alcohol is used, not only the percentage thereof be given, but also the "commodity from which it is distilled."

If molasses or sugar beets are used, this would have to appear on the label, also if potatoes were used, and in the case of synthetic alcohol, it probably would have to be stated whether the basic material was a petroleum by-product or natural gas. Purified alcohol obtained from any of these materials would be chemically identical, or practically so, with pure alcohol made from grains. However, tradition, legend, and perhaps superstition have imbued the consuming public with the idea that neutral spirits from grain was somehow better and preferable. Experience has shown that any product carrying a label indicating the presence of spirits derived from a material other than grains—as for instance, sugar cane or potatoes—met with sales resistance from the consumers.

The chief raw material for the production of beverage alcohol in the United States has been corn. The mash formula consists, as a rule, of just enough malt—about 8%—necessary to convert the starch contained in the corn. Assuming a yield of about 2.75 gallons of alcohol at 190 proof per bushel, the cost of the grains, at present prices, would be about 86 cents per gallon.

### Process of Manufacture

**Fermentation.** The only alcoholic beverages which can be obtained directly from fermentable saccharine materials are brandy and rum. The fermentation can be a natural one, caused by the yeast present on the grapes. In modern and well controlled wine making, yeast cultivated under controlled conditions is, as a rule, added.

**Table I. Materials Available for Making Alcohol by Fermentation**

Class	Saccharine Materials	Starchy Materials
Roots and tubers	Sugar beets	Artichoke (Jerusalem) Cassava Potato Sweet potato
Stems and leaves	Agave Cactus Sorghum Stalks of sugar corn Sugar cane	
Fruits and seeds	Apple Banana Grape Melon Orange Peach Pear Pineapple Prickly pear Tomato Watermelon	Corn Maize Barley Oats Rye Wheat
Trade wastes	Beet molasses Cane molasses Corn cannery refuse	



Whiskey fresh from the distillation columns is pumped into tanks in the cistern room, where it is drained into charred oak barrels for storing and aging. Grain residues, remaining after distillation, are recovered for livestock feeds

In many European countries, distinctive distilled beverages are being produced. The French distill wine into the "Eau de Vie" (water of life) a designation which is being used for all grape brandies not prepared in the two provinces of Charente where the product is named after the capital, Cognac.

Other brandies are made of fruits other than grapes in localities where certain fruits grow in abundance. Kirschwasser, made from cherries, is a famous product from the black forest in Germany and the district of Zug in Switzerland. Others are Slivovitz, a prune brandy, and apricot brandies, mostly from central Europe and Yugoslavia.

Rum is obtained from fermented sugar cane molasses. The quality of the rum is determined to a large extent by the addition of cultivated yeast and also by using in the mash some stillage from preceding operations. The use of such stillage, called dunder, is common in the preparation of the so-called "island rum," especially in Jamaica, where a particularly heavy and highly aromatic product is being made. The quality is further influenced by the distilling operations; while continuous distillation predominates, old fashioned pot stills are still being used in some localities.

By far the most important distilled beverage in the United States is whiskey. As previously mentioned, whiskey is obtained from a fermented mash of grains. The choice of the type of grains determines to a considerable extent the type and character of the end product. Corn is used in high percentage compared to other grains if the whiskey is desired "light" in character. The description "light" for whiskey means that it is relatively low in aromatic substances such as aldehydes and esters, and especially also in heavy bodied materials such as higher alcohols. The other grains commonly

used are rye and the amount of malt necessary for conversion. Both add a certain amount of flavor to the final product due to the aromatic oils contained in the grain.

The lightest whiskey of the bourbon type contains predominantly corn. It is made from a mixture containing about 88% corn, 2% rye, and 10% malt. Before fermentation can take place, the starch content in the grains has to be converted into sugar. This is effected through the action of the enzyme diastase or amylase present in malt, which is germinated grain. As a rule barley malt is used, especially for bourbon. The enzyme is developed in the grain by means of the malting process. The enzymatic activity is of a catalytic character. The conversion never proceeds quantitatively and depends upon a number of conditions during the conversion. In good practice, about 80% of fermentable maltose is obtained. If the conversion process is pressed too strongly toward a high proportion of maltose, it is usually at the expense of quality. This consideration is not of particular importance in the production of spirits, where an effective purification takes place during distillation at high proof, and subsequently rectification. With whiskey as the end product, all the factors during conversion have to be carefully supervised and controlled. Diastase really exerts a dual action, liquefaction and saccharification. The intensity of the conversion depends largely on the completeness of the starch pastification, and this in turn depends on the temperature applied during this process. In order to be effective, the conversion is, as a rule, carried out under pressure in so-called cookers. The malt is added to the cooked mash after it is cooled to optimum temperature for the action of the diastase.

#### Preparation of "Beer"

The alcoholic mixture that ultimately is used for distillation is called "distillers' beer," or just "beer" in America. In England it is designated "wort." Beer is prepared from grain by five steps: grinding, cooking, saccharification (conversion), dilution, and fermentation. The grain is ground fine enough to be made into a homogenous paste. If ground too fine, excessive dusting or difficulties during the process of by-product recovery will result. The bulk of the grind should preferably be between 10 and 30 mesh.

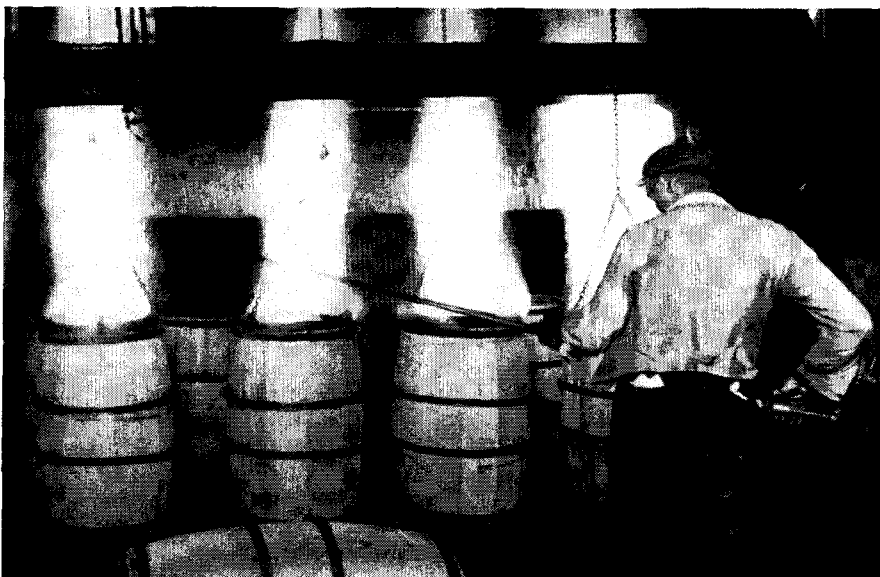
The flour derived from each bushel of grain (56 pounds) is thoroughly mixed with about 24 gallons of water. The mixture is heated under constant agitation by means of steam. This processing step is called "cooking." Cooking causes a pasting of the starch of the flour, enabling it to be converted (during saccharification) more rapidly into fermentable sugar than the starch of the

original kernels. Depending upon the equipment used, there are two types of cooking: at atmospheric pressure, generally carried out in open mash tubs; and "pressure cooking," for which closed pressure tanks are employed. Both open mash tubs and pressure cookers are provided with heating facilities and agitating devices. In open tubs, the mixture of flour and water is heated to 212° F. and kept at that temperature for a period of one half to one hour. In the preparation of rye, the heating is carried only to 160° F. because the distillate may develop an unpleasant flavor if the cooking is carried out at a higher temperature. In pressure cookers, the mixture of water and flour (for bourbon and corn whiskey production—corn flour) is heated to 250° to 275° F.; for spirit production (milo, wheat, and corn flour) the heating is carried up to 300° F. These temperatures in the pressure cooker are generally maintained for no longer than five minutes.

After cooking, the mash is rapidly cooled to between 140° to 144° F., in open mash tubs by means of cooling coils, and in pressure cookers by means of barometric condensers. The entire process of cooking can also be performed as a continuous operation.

Saccharification, or conversion of the starch paste into maltose, is carried out at 140° to 144° F., being initiated by adding ground malt in the form of flour or a slurry. Most of the conversion takes place with constant agitation in the open mash tub or cooker within 20 to 60 minutes. In this interval, 60 to 70% of the starch present in the grain is converted into maltose, while the remainder is converted into dextrins, the major portion of which will subsequently be converted in the next two or three days during fermentation.

The converted mash, also called cooker mash, is then cooled to fermentation temperature and delivered into the fermenters, where it is diluted. The concentration varies from 30 to 36 gallons of liquid per bushel of grain, for the production of spirits or alcohol, to 40 to 45 gallons for making whiskey. For diluting the converted mash, thin screened stillage is used, although in some instances only water is added. (Stillage is the liquid recovered from the still after the alcohol has been removed by distillation.) It is customary to use 10 to 15 gallons (and occasionally more) of thin stillage for every bushel of grain. Frequently a small portion of stillage as well as of malt is added to the cooker or open tub before the cooking process. The volume of water to be added is calculated so that, with the amount of condensate formed from the steam during cooking and the stillage, the desired dilution is obtained. The diluted cooker mash—ready for fermentation—is called sweet beer.

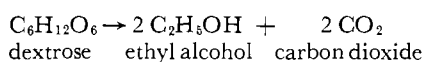


New oak barrels are charred. Red layer just beneath char embodies several soluble wood compounds which react with whiskey, modify harsh congeners, form new compounds, and mature the whiskey

### Fermentation

The process of fermentation converts sugar into alcohol. Alcoholic fermentation is caused by an enzyme complex—zymase—which is introduced by various microorganisms. In the preparation of alcoholic beverages, yeast of the genus *Saccharomyces* is almost exclusively used as the source. It grows in the beer and multiplies rapidly by means of budding. Of the many species of *Saccharomyces*, *S. cerevisiae* is invariably used for controlled alcoholic fermentation. A great variety of strains exist from which the most suitable for a specific purpose can be selected.

Alcoholic fermentation represents a chain of complicated reactions, which can be indicated by the over-all equation:



The numerous side reactions depend on the composition of the wort, the yeast, and the general conditions existing during fermentation, and lead to trace quantities of a number of secondary fermentation products. In the production of alcohol, the secondary products are eliminated during the succeeding processes of distillation and rectification. In the production of alcoholic beverages, formation of the secondary products and their nature determine the characteristics and qualities of the final product; these are formed and retained during the subsequent operations a number of aldehydes and esters (such as ethyl acetate), higher alcohols collectively designated fusel oils, and some fatty acids. The secondary products retained in the final distillate are designated congeners.

Fermentation is initiated by the inoculation of the sweet beer with 2 or 3%

by volume of ripe yeast prepared separately. Fermentation takes place in two, three or four days and proceeds in three distinctive phases. It begins with the so-called prefermentation or incubation period during which the yeast cells multiply, increasing in number from about 6 million to about 100 million or more per milliliter of the liquid. During the second period, the main fermentation, the largest part of the sugar is fermented to alcohol and carbon dioxide. The third phase is the postfermentation period. In this phase the remaining dextrins are converted into fermentable sugar and fermented to alcohol. Fermentation during the first and last phases is very slow; during the main fermentation, as indicated by the violent boiling of the fermenting beer, it is very rapid.

Since alcoholic fermentation is a heat-producing reaction, in modern fermenters this heat is conducted away by cooling coils; the temperature of the fermenting beer is thus kept close to optimum from the beginning until the end of this stage. Depending upon the yeast used, the optimum temperature varies from 82° to 86° F. In fermenters not provided with coolers, the inoculation temperature (called setting temperature) is so selected that the temperature during fermentation will not increase above the maximum limit (around 90° to 92° F.) tolerated by the yeast. The setting temperature in summertime may be as low as 66° F., while in the winter it is generally around 74°.

Preparation of the yeast is similar to the preparation of beer, the only difference being that the mash concentration is higher, that is about 24 to 28 gallons of water per bushel of grain. This concentrated yeast mash is acidified by a 20-hour fermentation with lactic acid

bacteria. Every 100 gallon of this acidified yeast mash is then inoculated with 10 to 30 gallons of yeast previously prepared by stepwise propagation.

### Distillation and Rectification

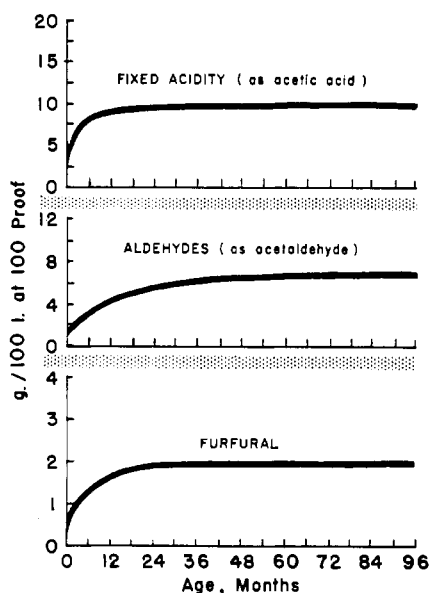
The fermented wort, or distillers beer contains, besides the alcohol and the desirable secondary fermentation products, solid particles originating from the grain, yeast, mineral salts, and such fermentation products as glycerol, lactic acid, succinic acid, tartaric acid, and fatty acids. The alcohol and the other by-products desired in the final product are separated by distillation. A great number of varieties and degrees are possible: single distillation, double distillation ("doubling"), elimination of the various secondary products by means of separate distilling columns, and rectification in multichambered rectifying columns. The equipment used for these purposes has undergone, in the course of many years, a steady development and improvement.

**Pot Stills.** The oldest and most primitive form of still consists of a boiling pot or vessel to which is attached a head that collects the vapors and conducts them to a condenser. The alcoholic vapors pass through the head, which is usually in the form of a retort, and emerge from the condenser as the distillate. The product of the first distillation from a pot still is, as a rule, impure and of about 40 to 50 proof. These so-called "low wines" are then redistilled two or three times until the distillate has reached the desired purity and strength.

Stills are generally made of copper. Pot stills are heated either directly by fire or sparged steam, or indirectly by means of a jacket, coil, or calandria. If a more highly purified distillate is desired, an improved type of pot still can be used. This type of still is equipped with a rectifying head, which acts as a sort of dephlegmator. It condenses a portion of the vapors, which are returned to the still. Improved stills of this type are in use today for the production of certain types of whiskeys and brandies. Pot stills can distill only one batch at a time, and, therefore, are not very economical in operation.

**Continuous Stills.** The first form of continuous still appeared as the so-called Coffey or patent still in England. In the operation of such stills, the preheated wort is fed continuously into the still, which consists of a column of perforated plates. The wort flows downward and is met by steam that ascends through the still from the bottom, causing the wort on the plates to boil and the alcohol and other volatile substances to vaporize. The descending wort is gradually freed of these substances and finally is removed from the bottom of the still.

Modern stills are characterized by



many improvements and refinements that permit not only more economical operation, but also a very close control of the entire operation and, thereby, the final product. One improvement is the development of vacuum distillation, which is being used in some distilleries.

#### By-Products

The beer, once freed from alcohol and other volatile substances, is designated stillage or slop, and contains a number of substances in solution and suspension. These consist in part of the insoluble and undecomposed portions of the grain and of other resulting substances mainly from the conversion and fermentation processes. The solid portions are recovered by screening and drying (usually in steam dryers). The liquid portion, which contains finely suspended and dissolved materials, is first concentrated by vacuum evaporation to the consistency of a sirupy liquid. This concentrate can either be mixed with the previously screened-out solid materials and the two dried together, or it can be dried separately on rotary-drum dryers. The content of the liquid portion in vitamins, especially riboflavin and other components of the B complex, make this material particularly valuable as an ingredient for animal feed mixtures.

#### Production of Specific Types

The type and character of alcoholic beverages is determined, not only by the raw materials used in their preparation, but also by specific measures employed in the various steps of the manufacturing process. For whiskey, the method of distillation, and especially the type of still used, is of importance.

When a heavy bodied whiskey is desired, pot stills are used. Pot stills are, of course, in principle nothing but large sized retorts. However, prac-

tically all whiskey is nowadays distilled in continuous stills, and the operation is carried out at a proof below 160, which limit is prescribed by United States Government regulations for whiskeys which are to be identified by type—such as bourbon or rye. Under observation of this borderline, it is possible to influence the quality of the whiskey by distilling close to the permitted upper limit whenever a light-bodied product is wanted. The consequences of distilling at a high proof is that more condensate is refluxed into the still, thereby deviating more of the heavily flavored material to the beer residue, the “stillage”. If a heavier type is desired, distillation is carried out at a lower proof. Newly distilled whiskey is colorless and to most people, unpleasant in taste and aroma.

#### Maturation

In order to make whiskey palatable and give it the desired appearance, flavor and taste, a very carefully controlled process of maturation is necessary. For this purpose, whiskey is stored in new charred white oak barrels, usually for a period of four to eight years, sometimes longer.

The use of new and very dry wood results, of course, in the absorption of considerable quantities of whiskey by soakage into the wood. This whiskey is lost to production since it is expressly forbidden to sweat or press out this soakage whiskey from the wood. It is, of course, possible to store whiskey in previously used barrels in which the wood is already saturated with whiskey. However, the regulations prescribe that the use of barrels of this type must be stated on the label of any product so prepared. Furthermore no statement of age is permitted for whiskey that has been stored in reused barrels. It is often claimed that reused barrels have certain advantages, but again tradition and legend have prejudiced the public, so that a product with a label indicating a product aged in reused barrels will meet with great sales resistance.

The action of the barrel during maturation is manifold. The char itself has a purifying influence since it will adsorb some of the higher alcohols and fatty acids. Underneath the char is a layer of toasted wood containing a variety of aromatic and flavoring substances, as well as highly pigmented materials. The untoasted wood contains, among other components, quantities of tannins. Of all these ingredients much is gradually extracted by the whiskey to contribute to its character, taste, flavor, and the reddish-brown color.

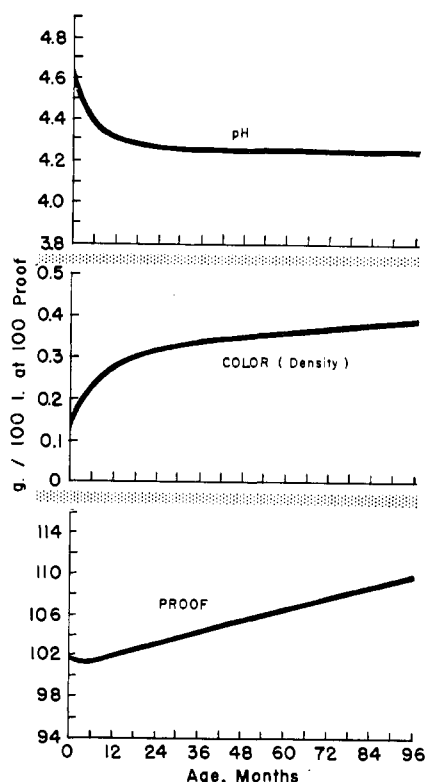
After being filled with whiskey, the barrel is placed on the rack in the storage warehouse. There begins the intricate process of maturation which consists in interreactions between the substances

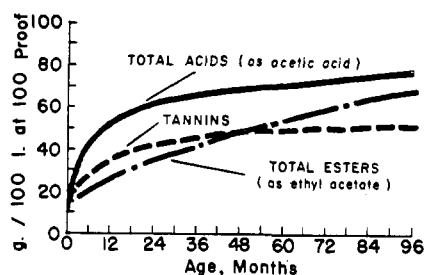
originally in the newly distilled whiskey, the so-called congeners, and those extracted from the wood. These react with each other, and with the oxygen contained in the barrel or entering it by diffusion through the wood.

The barrels are placed in bonded warehouses (constructed of wood, brick, or concrete) and are usually permitted to remain for four or more years under variably controlled conditions of temperature and humidity. The maturing stage ends with the withdrawal of the aged whiskey from the barrels.

Three fundamental environmental factors are intimately related to the development of a whiskey during maturing—temperature, humidity, and ventilation. The barrel, serving primarily as a container, also acts as a semi-permeable membrane and permits the passage of alcohol and water vapors from the interior of the barrel to the outside. This phenomenon is an integral phase of maturing. Under the conditions normally prevailing in storage warehouses—especially as to humidity and temperature—the barrel permits water vapor to escape at a faster rate than alcohol vapor. Consequently, there is a gradual accumulation of alcohol at the expense of water inside the barrel. Thus the proof of the contents rises with age. Many warehouses are constructed and equipped to allow positive control of this phase of the maturing process.

During maturation, whiskey undergoes definite and intended changes in aromatic and taste characteristics. These changes are caused by three major types of reactions occurring continually in the





barrel: extraction of complex wood substances by liquid; oxidation of the original organic substances and of the extracted wood material; and reaction between various organic substances present in the liquid to form new products.

The development of quality, therefore, consists of specific chemical and physical changes in the properties of the liquid, some of which are relatively simple to determine. These properties, or characteristics, are commonly used as a guide to and measure of quality. Experience and observation have shown that abnormalities arising in one or several of the physicochemical characteristics generally will result in abnormalities of the taste characteristics of the liquid.

Several comprehensive investigations of these changes have been carried out, as by Crampton and Tolman (1908), Valaer and Frazer (1936), Liebmann and Rosenblatt (1943), and Liebmann and Scherl (1949) [*Ind. Eng. Chem.*, **41**, 354(1949)]. This last work was begun by the Schenley organization shortly after repeal and was continued over more than 12 years. It involved 469 barrels, each of which was kept under constant observation. One-pint samples were withdrawn at the age of and, one, three, six, and twelve months, zero every six months thereafter up to the age of four, six, or eight years. Methods of determination were those of AOAC.

#### Changes in Whiskey Characteristics

The changes in whiskey which take

place during maturation are chiefly responsible for the progress in quality, and the characteristics of the final product. These changes are illustrated by a number of figures, each representing the development that takes place in one of the constituents.

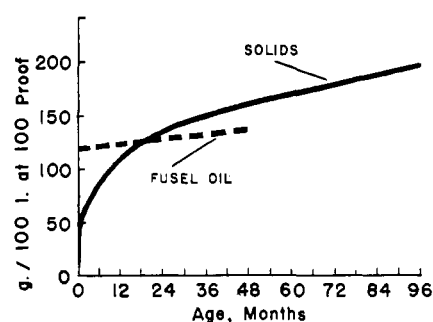
**Whiskey Constituents.** It is evident that for those characteristics which depend for their formation on the extraction of certain substances from the wood of the barrel and their interreaction with the originally present congeners, the development of the characteristics of whiskey is in practically all instances rapid at the beginning. This is indicated by a sharp curve during the first 12 months. Thereafter, there is a change to a linear increase, and from the second year on an asymptotic approach takes place.

The proof at which whiskey is usually bonded (age zero) is determined by the distiller and is customarily set at 102 to 105 proof.

The development of total acidity is largely determined by the extraction of soluble substances from the toasted wood section underneath the completely charred inner surface of the barrel. During the early stages of maturation there occurs an extremely rapid increase, whereupon the rate begins to fall off, and assumes an asymptotic approach which is also typical for solids, tannins, color, and to some extent for pH, although in this case the graph appears inverted, since the pH represents the reciprocal of the hydrogen-ion concentration.

Table II is based on a statistically significant number of examinations. It presents what may well be considered a standard for this type of alcoholic beverage.

Alcoholic beverages occupy an important place in national economics and have often played a prominent part in historical developments.



From an insignificant nine cents per proof gallon in 1790, the federal excise tax was raised step by step until it reached \$1.10, which prevailed all through the interregnum of prohibition, when whiskey was presumed to be strictly a therapeutic article. It could be withdrawn only from existing inventories, excepting the issuance of a few permits for new production. Shortly after repeal the tax was fixed in 1934 at \$2.00, and successively raised in 1938 to \$2.25; in 1940 to \$3.00; in 1941 to \$4.00; in 1942 to \$6.00; in 1944 to \$9.00; and in 1951 to \$10.50. In addition there are federal stamp taxes, and a multiplicity of state and local levies.

In normal years the production of distilled beverages will be about 175 million proof gallons, of which about 90 million gallons would be whiskey. During the fiscal year of 1951 a total of 401 million gallons were produced, a very abnormal increase due to precautionary measures and the distillers' fear of new restrictive measures in case of a war. In 1952 the production was 221 million gallons, of which 103 million gallons was whiskey.

The distilled beverage industry paid in direct excise taxes to the federal government in the first 11 months of the fiscal year of 1952, \$1451 million, or at the yearly rate \$1.6 billion. Next to personal income and corporation taxes this excise tax is, therefore, the largest source of revenue.

Table II. Characteristics of American Whiskies at Various Ages<sup>a</sup>

Age	Age		Total Acids	Fixed Acids	Esters	Aldehydes	Furfural	Fusel Oil	Solids	Color (Density)	Tannins	pH	
	Yr.	Mon.											Proof
0			101.8	5.9	0.8	16.7	1.4	0.2	111	8.7	0.032	0.7	4.92
1			101.4	20.4	3.7	17.2	2.1	1.2	123	44.1	0.156	12	4.62
			101.3	32.2	5.3	18.5	2.8	1.5	131	66.6	0.205	21	4.46
			101.4	42.5	6.6	21.8	3.3	1.6	131	87.7	0.243	28	4.38
1			102.0	53.4	8.3	26.8	4.1	1.7	132	111.1	0.282	35	4.38
			102.5	58.1	9.0	31.1	4.8	1.8	132	127.6	0.308	39	4.29
2			103.1	61.8	9.2	35.5	5.5	1.8	134	137.5	0.328	42	4.29
			103.6	64.1	9.3	38.9	5.8	1.9	136	147.7	0.341	44	4.28
3			104.1	65.8	9.3	41.8	6.0	1.8	135	152.7	0.352	47	4.27
			104.7	67.8	9.4	44.7	6.0	1.9	137	157.7	0.360	48	4.26
4			105.2	69.2	9.4	47.6	6.1	1.8	138	165.9	0.365	48	4.26
			105.5	69.7	9.4	48.0	6.1	1.7	...	166.0	0.367	49	4.26
5			106.0	70.2	9.5	51.9	6.2	1.7	...	173.0	0.368	49	4.26
			106.7	72.0	9.5	55.6	6.3	1.8	...	174.2	0.369	49	4.26
6			107.4	71.6	9.5	57.6	6.5	1.8	...	181.5	0.380	49	4.24
			107.9	74.4	9.6	61.2	7.0	1.8	...	186.0	0.385	50	4.24
7			108.6	76.2	9.7	62.0	7.0	1.8	...	198.6	0.389	50	4.23
			108.9	79.4	9.7	64.4	7.0	2.0	...	198.9	0.413	50	4.22
8			109.3	81.9	9.7	64.8	7.0	2.0	...	209.6	0.449	53	4.20

<sup>a</sup> All figures represent average values and are expressed as grams per 100 liters at 100 proof, except proof (expressed as degrees proof), color (expressed as density), and pH. Source: *Ind. Eng. Chem.*, **41**, 354 (1949).