Emission Factor Documentation for AP-42
Section 9.12.3

Distilled Spirits

Final Report

For U. S. Environmental Protection Agency
Office of Air Quality Planning and Standards
Emission Factor and Inventory Group

EPA Contract 68-D2-0159
Work Assignment No. 4-04

MRI Project No. 4604-04

March 1997

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For U. S. Environmental Protection Agency
Office of Air Quality Planning and Standards
Emission Factor and Inventory Group
Research Triangle Park, NC 27711

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NOTICE

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PREFACE

This report was prepared by Midwest Research Institute (MRI) for the Office of Air Quality Planning and Standards (OAQPS), U. S. Environmental Protection Agency (EPA), under Contract No. 68-D2-0159, Work Assignment Nos. 2-03, 3-01, and 4-04. Mr. Dallas Safriet was the requester of the work.

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1. INTRODUCTION

The document *Compilation of Air Pollutant Emission Factors* (AP-42) has been published by the U. S. Environmental Protection Agency (EPA) since 1972. Supplements to AP-42 have been routinely published to add new emission source categories and to update existing emission factors. AP-42 is routinely updated by EPA to respond to new emission factor needs of EPA, State and local air pollution control programs, and industry.

An emission factor is a representative value that attempts to relate the quantity of a pollutant released to the atmosphere with an activity associated with the release of that pollutant. Emission factors usually are expressed as the weight of pollutant divided by the unit weight, volume, distance, or duration of the activity that emits the pollutant. The emission factors presented in AP-42 may be appropriate to use in a number of situations, such as making source-specific emission estimates for areawide inventories for dispersion modeling, developing control strategies, screening sources for compliance purposes, establishing operating permit fees, and making permit applicability determinations. The purpose of this report is to provide background information from test reports and other information to support revisions to AP-42 Section 9.12.3, Distilled and Blended Liquors (formerly incorporated into Section 6.5, Fermentation).

This background report consists of five sections. Section 1 includes the introduction to the report. Section 2 gives a description of the distilled spirits industry. It includes a characterization of the industry, a description of the different process operations, a characterization of emission sources and pollutants emitted, and a description of the technology used to control emissions resulting from these sources. Section 3 is a review of emission data collection (and emission measurement) procedures. It describes the literature search, the screening of emission data reports, and the quality rating system for both emission data and emission factors. Section 4 details how the revised AP-42 section was developed. It includes the review of specific data sets and a description of how candidate emission factors were developed and a summary of changes to the AP-42 section. Section 5 presents the AP-42 Section 9.12.3, Distilled Spirits. Supporting documentation for the emission factor development is presented in the Appendices.

2. INDUSTRY DESCRIPTION

The section gives a brief review of trends in the distilled spirits industry and describes the process of whisky production. Emission information is only available for fermentation and aging. Sources of volatile organic compounds (VOC), principally ethanol, are discussed, and a brief description of emission control technology is given.

2.1 INDUSTRY CHARACTERIZATION[1-4]

The fermentation industry includes the production of malt beverages (beer); wines; brandy and brandy spirits; distilled spirits; and the secondary products of all of these industries. The most commonly produced distilled spirits for beverage purposes include whiskies, gins, vodkas, rums, and brandies.[a] Whiskies are produced from fermented grain mashes and aged. Vodkas are produced from fermented grain mashes, but are not aged. Gins generally are produced from the fermented product, grain neutral spirits (GNS), to which either botanical extracts and/or flavors are added to the GNS and bottled, or dried botanicals (e.g., juniper berries) are added to the GNS to extract their oils and then distilled. Rums are made from fermented sugar cane products, such as molasses. Gins and rums may be aged in barrels. Brandies are distilled from wine or other fermented fruit juices, and are generally aged in barrels. Distilled spirits production (e.g., whisky, vodka, or gin) may produce secondary products, such as distillers dried grains used as livestock feed.

Distilled spirits are produced throughout the United States (see Table 2-1). The data presented in Table 2-1 represent production of distilled spirits as reported to the Bureau of Alcohol, Tobacco, and Firearms (BATF), U. S. Department of the Treasury. The classification of distilled spirits (SIC 2085) includes the production of distilled spirits for both beverage purposes and medicinal purposes; quantities for both of these purposes are included in the "alcohol and spirits" column of Table 2-1. Establishments engaged in manufacturing alcohol for industrial purposes are classified under SIC 2869; quantities of ethanol produced from grain for industrial purposes may also be included in Table 2-1. In Table 2-1, the production quantities for vodka are no longer reported separately by the BATF but are included in the larger category of "alcohol and spirits."

The remainder of this document is concerned primarily with the emissions resulting from the production of distilled spirits for beverage purposes. Over the last several years, the distilled spirits industry has experienced large decreases in sales. United States distilled spirits sales peaked in 1981 at approximately 189 million 9-liter cases and decreased to approximately 137 million 9-liter cases in 1994, a decline of almost 28 percent.

2.2 PROCESS DESCRIPTION[4-5]

Distilled spirits can be produced by a variety of processes. Typically, whisky production utilizes malted grains which are mashed and fermented to produce an alcohol/water solution that is distilled to concentrate the alcohol. This is not necessarily true for production of other distilled spirits, such as vodka, rum and brandy. The concentrated alcohol is usually aged in wooden barrels to provide natural color and impart flavor and aroma. Recognizing that not all distillers employ identical techniques and materials, this

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[a]Brandies are discussed in AP-42, Section 9.12.2, Wines and Brandy.

TABLE 2-1. PRODUCTION OF DISTILLED SPIRITS--1995[a,b]

State	Whisky[c]		Brandy	Rum	Gin	Alcohol & spirits	
	160° and under	Over 160°				190° and above	Under 190°
CA	789	0	9,089,118	0	0	15,682,949	785,878
FL	0	0	1,860,633	918,372	0	4,366,642	(88,444)
IL	0	0	0	0	2,399,822	817,619,465	3,928,243
IN	833,937	3,496,625	0	0	8,237,141	10,007,598	774,646
IA	0	0	0	0	1,341,305	429,460,453	4,336,322
KY	45,755,633	396,505	0	0	0	10,367	293,990
MI	0	0	0	0	0	0	470,141
MN	0	0	0	0	0	2,945,614	0
OH	0	0	0	0	0	866,647	0
TN	16,894,626	0	0	0	0	77,943,406	0
TX	0	0	0	0	0	36,069,118	139,225
VA	78,593	0	0	0	0	935,098	0
Other[d]	39,780	0	6,061	0	1,786,200	78,398,481	1,486,938
TOTAL	63,603,358	3,893,130	10,955,812	918,372	13,764,468	1,474,305,838	12,126,939

Source: Reference 3.

[a] Represents gross production (original plus redistillation) minus the products used in redistillation. Vodka production quantities are no longer reported separately; they are incorporated into a larger category of "alcohol and spirits."

[b] All quantities in proof gallons. Proof gallon is a U.S. gallon of proof spirits or the alcoholic equivalent thereof, i.e., a U.S. gallon containing 50 percent of ethyl alcohol (ethanol) by volume (Reference 4).

[c] Gross production of whisky includes bourbon, light, corn, and other whisky in new barrels.

[d] Includes Connecticut, Georgia, Kansas, Maryland, Massachusetts, Missouri, New Hampshire, New Jersey, New York, North Carolina, Oregon, Pennsylvania, Puerto Rico, Washington, and Wisconsin.

section attempts to provide a generic description of distilled spirits (distillery) operations. The focus of this discussion will be on Bourbon whisky production. Processes for other distilled spirits will differ from Bourbon whisky production.

Under the standards of identity set forth by the BATF, whisky refers to an alcoholic distillate from a fermented mash of grain produced at less than 190° proof ethanol (95 percent by volume) in such a manner that the distillate possesses the taste, aroma, and characteristics generally attributed to whisky, stored in oak containers (except that corn whisky need not be so stored), and bottled at no less than 80° proof, and also includes mixtures of such distillates for which no specific standards of identity are prescribed.[b] (See Reference 6). Types of whisky and classes and types of other distilled spirits also are defined in BATF standards of identity.[6] Figure 2-1 provides a simple diagram of a typical whisky production process.

In the distilled spirits industry, there are two terms commonly used to describe the volume of the spirits: "proof gallons" and "wine gallons." The term "proof gallon" refers to a U. S. gallon of proof spirits, or the alcoholic equivalent thereof, containing 50 percent of ethyl alcohol by volume. Since excise taxes are paid on the basis of proof gallons, this term is synonymous with tax gallons. The term "wine gallon" refers to a measure of the actual volume regardless of the proof of the spirits.[4]

2.2.1 <u>Grain Handling and Preparation (Milling)</u>

Distilleries utilize premium cereal grains, such as hybrid corn, rye, malted barley, and wheat, to produce the various types of whisky and other distilled spirits. United States distilleries purchase malted grain instead of performing the malting process onsite. The grains have particular specifications, especially with regard to the elimination of grain with objectionable odors which may have developed in the field or during storage, handling, or drying at the elevators.

Grain receiving, handling, and cleaning are potential sources of particulate matter (PM) emissions. Grain is generally received in either hopper railcars or trucks. Grain handling is the transfer from the unloading pit by pneumatic conveyor system, auger system, and bucket elevators to and from the grain storage silos. Although it usually has been subjected to a cleaning process at the elevator, the grain may be subjected to additional cleaning, which may include a series of vibrating screens that sift out foreign materials and magnetic separators used to remove any ferromagnetic items. Dust collectors and air jets may be used to remove light materials and aid in the control of PM emissions.

Milling, which breaks the outer cellulose protective wall around the kernel and exposes the starch to the cooking and conversion process, can be accomplished by several milling methods. For example, hammer mills use a series of hammers rotating at 1,800 to 3,600 rpm within a close-fitting casing. These hammers shear the grain to a meal that is removed through a screen with different mesh sizes for various types of grain. Cage mills use a series of counter rotating bars at high speed to grind the grain by impact. Roller mills use a series of close tolerance serrated rollers to crush the grain. Distillers require an even grind, generally with a particle size as small as can be physically handled by the facility.

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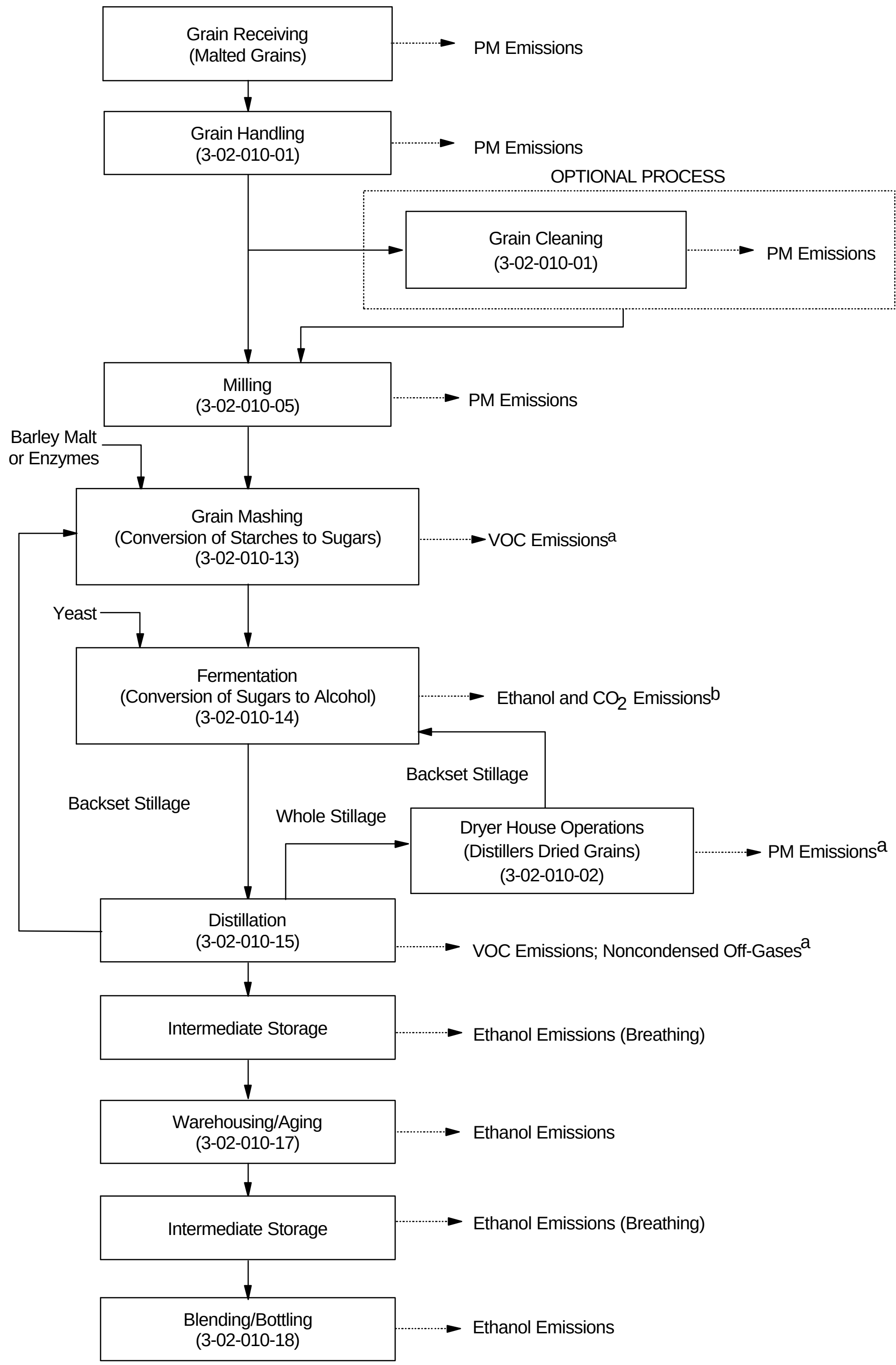
[b]In the United States, 100° proof equals 50% ethanol content by volume at 15.6°C (60°F). In Canada and the United Kingdom, 87.7° proof equals 50% ethanol by volume at 10.6°C (51°F).



[a] Processes require heat. Emissions generated (e.g., CO, CO_2, NO_x, SO_2, PM, and VOCs) will depend on the source of fuel.
[b] Other compounds can be generated in trace quantities during fermentation including ethyl acetate, fusel oil, furfural, acetaldehyde, sulfur dioxide, and hydrogen sulfide. Acetaldehyde is a hazardous air pollutant (HAP).

Figure 2-1. Whisky production process.

2.2.2 Mashing

The mashing process consists of cooking (gelatinization) of the grain in water to solubilize the starches from the kernels and converting (saccharification) of the starch to "grain sugar" (primarily glucose and maltose). In general, cooking can be carried out at or above atmospheric pressure in either a batch or continuous process. During mashing, trace VOC emissions may result from constituents in the grain. Small quantities of malted barley are sometimes added prior to grain cooking. After partial cooling, conversion of the starch to sugar is accomplished by adding barley malt and/or enzymes (from other sources) to the cooked grain at approximately 63°C (145°F). The mash then passes through a noncontact cooler to a fermenter. Between the mashing and fermentation, the process generally is closed during cooling, with no emissions. Distillers may vary mashing procedures, but generally conform to basic principles, especially in the maintenance of sanitary conditions.

2.2.3 Fermentation

Fermentation, which usually lasts 3 to 5 days for whisky, involves the use of a yeast to convert the grain sugars into ethanol and carbon dioxide (CO_2). The converted grain mash is cooled prior to entering the fermenter or tank and inoculated with yeast. It is common practice to dilute the hot grain mash to its final solids concentration by adding backset stillage and/or water. Backset is liquid stillage which is screened or centrifuged from the distillation "beer still bottoms." The use of backset provides water conservation, nutrient supplements, pH adjustment of the fermentation, and some flavor components (e.g., sour mash).

The fermentation process varies slightly for the production of other distilled spirits. For instance, rum fermentations takes 1 to 2 days. In rum production, black strap molasses is the source of fermentable sugars and is stored in tanks prior to fermentation. The black strap molasses also is not "mashed" (i.e., cooked) prior to being diluted with water to obtain the proper concentration of fermentable sugars.

Congeners are flavor compounds which are produced during fermentation, as well as during the aging process. These congeners include trace aldehydes, esters, and higher alcohols (i.e., fusel oils). Lactic acid bacteria (*lactobacillus*) may simultaneously ferment within the mash and contribute to the overall whisky flavor profile. On rare occasions *lactobacillus* may provide some pH control. On other occasions, the addition of sulfuric acid, though rarely used, may result in trace hydrogen sulfide emissions from the fermentation tank.

In whisky production, significant increases in the amount of yeast consumed occur during the first 30 hours of fermentation, when over 75 percent of the carbohydrate (sugar) is converted to ethanol and carbon dioxide. Many fermentation vessels are equipped with agitation and/or cooling means that facilitate temperature control. Fermentation vessels may be constructed of wood or metal and may be open or closed top.

The final fermented grain alcohol mixture, called "beer," is agitated to resuspend its solids and may be transferred to the "beer well" storage vessel for holding until it is pumped to the "beer still." Distillers use mechanical or air agitation during transfer and storage to prevent settling of solids. In the instance of air agitation, trace amounts of aldehydes may be produced. The beer passes from the beer well through a preheater where it is warmed by the alcohol vapors leaving the still and then enters the still for distillation. The beer still vapors condensed in the preheater generally are returned to the beer still as reflux.

2.2.4 Distillation

The distillation process separates and concentrates the alcohol products from the fermented grain mash. In addition to the alcohol and congeners, the fermented mash contains solid grain particles, yeast cells, water-soluble proteins, mineral salts, lactic acid, fatty acids, and traces of glycerol and other trace congeners. Although many distillation processes exist, the most common systems used in the United States are the continuous beer still, with or without a doubler unit. Other distillation processes include the continuous multicolumn extractive and rectifying systems, and the batch rectifying pot still and condensing unit. Whisky stills are usually made of copper, especially in the rectifying section, although stainless steel may be used in some stills.

In a general whisky distillation process using a beer still, the whisky separating column consists of a cylindrical shell having three sections: stripping, entrainment removal, and rectifying. The stripping section contains approximately 14 to 21 perforated plates, spaced 56 to 61 cm (22 to 24 inches) apart. The fermented mash is introduced at the top of the stripping section and descends from plate to plate until it reaches the base where the stillage is discharged. Steam is introduced at the base of the column, and the vapors from the bottom of the still pass up through the perforations in the plates. Whisky stills are usually fitted with entrainment removal sections that consist of a plate above the stripping plate to remove fermented grain particles entrained in the vapor. Distillation columns operate under reflux (sealed) conditions and most vapors are condensed and collected, although small amounts of noncondensable gases will be emitted to the atmosphere. The rectifying section contains several bubble cap or valve rectifying plates in the top section of the still that produce distillates (ethanol) up to 190° proof.

The diameter of the still, the number of stripping and rectifying plates, capacity of any doubler, and proof of distillation are factors that can contribute characteristics to a particular whisky. The doubler is a type of pot still that is used to redistill the distillate from the beer still to enhance and refine the flavors desired in a specific whisky. Following distillation, the whisky, at high proof, is pumped to stainless steel tanks and diluted with demineralized water to the desired alcohol concentration prior to filling into oak barrels.

The distillation of other spirits, such as rum, is similar. Tennessee Whisky utilizes a different process than Bourbon, in that the distillate is passed through sugar maple charcoal in mellowing vats prior to dilution with demineralized water.

2.2.5 Grain and Liquid Stillage ("Dryer House Operations")

At most distilleries, after the removal of alcohol, still bottoms (known as whole stillage) are pumped from the distillation column to a dryer house. Whole stillage may be sold, land applied (with appropriate permitting), sold as liquid feed, or processed and dried to produce distillers dried grains (DDG). The DDG consists of proteins, fats, minerals, vitamins, and fibers which are concentrated three-fold by the removal of the grain starch in the mashing and fermentation process. Distillers' secondary products are divided into four groups: DDG, distillers dried solubles (DDS), DDG with solubles (DDG/S), and condensed distillers solubles (CDS).

Solids in the whole stillage are separated using centrifuges or screens. The liquid portion “thin stillage” may be used as a backset or may be concentrated by vacuum evaporation. The resultant syrup may be recombined with the solid portion or dried separately. This remaining mixture is then dried using one of a variety of types of dryers (usually steam-heated or flash dryers). The majority of DDG are used in animal feed, although increasing quantities are being sold as food ingredients for human consumption due to its nutrient and fiber content.

2.2.6 Warehousing/Aging

In the aging process, both the charred oak barrel in which beverage alcohol is stored and the barrel environment are key to producing distilled spirits of desired quality and uniqueness. The aging process gives whisky its characteristic color and distinctive flavor and aroma. Variations in the aging process are integral to producing the characteristic taste of a particular brand of distilled spirits. Aging practices may differ from distillate to distiller, and even for different products of the same distiller.

Ambient atmospheric conditions, such as temperature and humidity, as well as seasonal variation, are important factors in the aging process. Aging practices vary considerably--some distillers, for example, keep their warehouse windows open during certain months to promote interaction of the aging whisky with outdoor atmospheric conditions. An EPA report observed that the aging process, in particular, depends upon the interaction of whisky in oak barrels with ambient air and particularly the temperature, humidity, and ventilation promoted by the different types of warehouse construction utilized in the industry.[5] While each distiller alters the barrel environment to produce a product with the distinctive characteristics of its brand, the fundamentals of the natural aging process are inviolate. The various distillers control the barrel environment differently by operating their warehouses in different manners; all of these variations illustrate the number of differing aging philosophies and traditions.[5]

Ethanol emissions are a natural and integral consequence of creating the distinctive qualities of various whisky production and aging embodied in the federal law. In producing Bourbon whisky, for example, ethanol from the raw beverage alcohol is unavoidably released because the wooden barrels, in which it is aged, are porous to ethanol vapors. Bourbon is typically aged for 4 years. (Not all distilled spirits are aged the same; for example, rum may be aged from 3 months to more than 1 year.)

In keeping with federal regulations and because of constituents of the barrel imparted to Bourbon in the aging process, only new charred oak barrels can be used in Bourbon production. Charred white oak barrels encourage reactions within the whisky and between the whisky and the wood to produce the desired whisky flavor. White oak is used because it is one of the few woods that holds liquids while allowing breathing (gas exchange) through the wood. These barrels used to age Bourbon are typically reused for aging other whiskies and other distilled spirits products, such as cognac, Scotch whiskey, and brandies. Most whisky barrels are reused for approximately 20 to 30 years for aging other whiskies and distilled spirits that utilize barrel aging.

When whisky ages, the alcohol extracts and reacts with constituents in the barrel wood, producing its distinctive color, taste and aroma. Constituents in the wood are transferred to the bulk liquid in the barrel by simple diffusion, by convection currents in the bulk liquid, and by temperature cycling. As the barrel heats up, the gas above the liquid increases in pressure and forces liquid into the barrel wood. When the barrel cools and the gas pressure drops, the liquid flows out of the wood into the bulk liquid, carrying wood constituents with it. The distinctive qualities of whisky are added during aging as trace substances called congeners which occur through (1) extraction of organic substances from the wood and their transfer to the whisky, (2) oxidation of the original substances and of the extracted wood material, and (3) reaction between various organic substances present in the liquid to form new products. The amber color develops and the taste of the whisky mellows during aging as the concentration of congeners increases. Similar reactions between the barrel liquid and barrel constituents characterize aging of other distilled spirits, such as brandy and rum.

In aging or maturation, the rate of extraction of wood constituents, transfer, and reaction depend on both ambient conditions such as temperature and humidity and the concentrations of various whisky constituents. For instance, higher temperatures increase the rate of extraction, transfer by diffusion, and reaction. Diurnal and seasonal temperature changes also cause convection currents in the liquid and pressure changes in the gas affecting transfer. The rate of diffusion will depend upon the difference in concentrations of constituents in the wood, liquid, and air blanketing the barrel. The rates of reaction will increase or decrease with the concentration of constituents. Thus, changes in the airflow around the barrel would change the alcohol concentration around the barrel and impact the diffusion rate. All of these variables are integral to a particular product brand which will have its own unique taste, color, and aroma. According to the 1978 EPA report, when ventilation was artificially increased, the quality of the product was greatly impaired.

In the aging process, both the oak barrel in which the beverage is stored and the barrel environment are key to producing distilled spirits of desired quality and uniqueness. The oak barrels used for aging distilled spirits play a significant role in determining the final flavor and aroma of the beverage. Newly distilled whisky is colorless with a strong, harsh and unpalatable odor. The new whisky distillate undergoes many types of physical and chemical changes in the aging process that impart the distinctive color, taste and aroma of the whisky and gives 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 compounds with each other, and oxidation produced by diffusion to ambient atmosphere. As whisky ages, the alcohol grain distillate (containing grain flavors) extracts wood flavors and color from the barrel. These congeners (oxidation products) are produced by chemical reaction induced by simple diffusion, by convection currents in the bulk liquid, and by diurnal and seasonal temperature cycling. As the barrel heats up, the gas in the headspace above the liquid increases in pressure and forces the liquid into the wood. When the barrel cools and the gas pressure drops, the liquid flows out of the wood into the bulk liquid, carrying wood constituents with it. These constituents give whisky its distinctive color, taste, and aroma. The amber color develops and the taste of the whisky mellows as it undergoes the aging cycle. Ethanol and water vapor result from the breathing phenomenon of the white oak barrels and are emitted during the aging process. As the staves become saturated with whisky, ethanol is emitted to the atmosphere as an ethanol/water vapor mixture. This phenomenon of the wood acting as a semipermeable membrane is complex and not well understood. Figure 2-2 shows a simplified illustration of the mechanisms of the whisky aging process.

The barrel environment is extremely critical in whisky aging and varies considerably by distillery and warehouse and even by location of the barrel within a warehouse. Ambient atmospheric conditions, such as seasonal variation in temperature and humidity, have a great effect on the aging process. For instance, higher temperatures in the aging warehouse increase the equilibrium rate of extraction, rate of transfer by diffusion, and rate of reaction. Furthermore, diurnal and seasonal temperature changes affect transfer rates by creating convection currents in the liquid and pressure changes in the gas. For these reasons, distillers may selectively open warehouse windows during certain months to promote interaction of the barrels with outdoor atmospheric conditions. Furthermore, the equilibrium concentrations of the various whisky components depend heavily on the air flow around the barrel. All of these variables are utilized by each distiller to produce its distinctive brand with its own unique taste, color, and aroma.

Distillers utilize various warehouse designs, which include single- or multistory buildings constructed of metal, wood, brick, or masonry. Most warehouses have no climate control systems and rely on natural ambient temperature and humidity changes to drive the aging process; in a few warehouses, temperature is adjusted in the wintertime. However, no whisky warehouses have the capability of controlling humidity, which varies with natural climatic conditions.

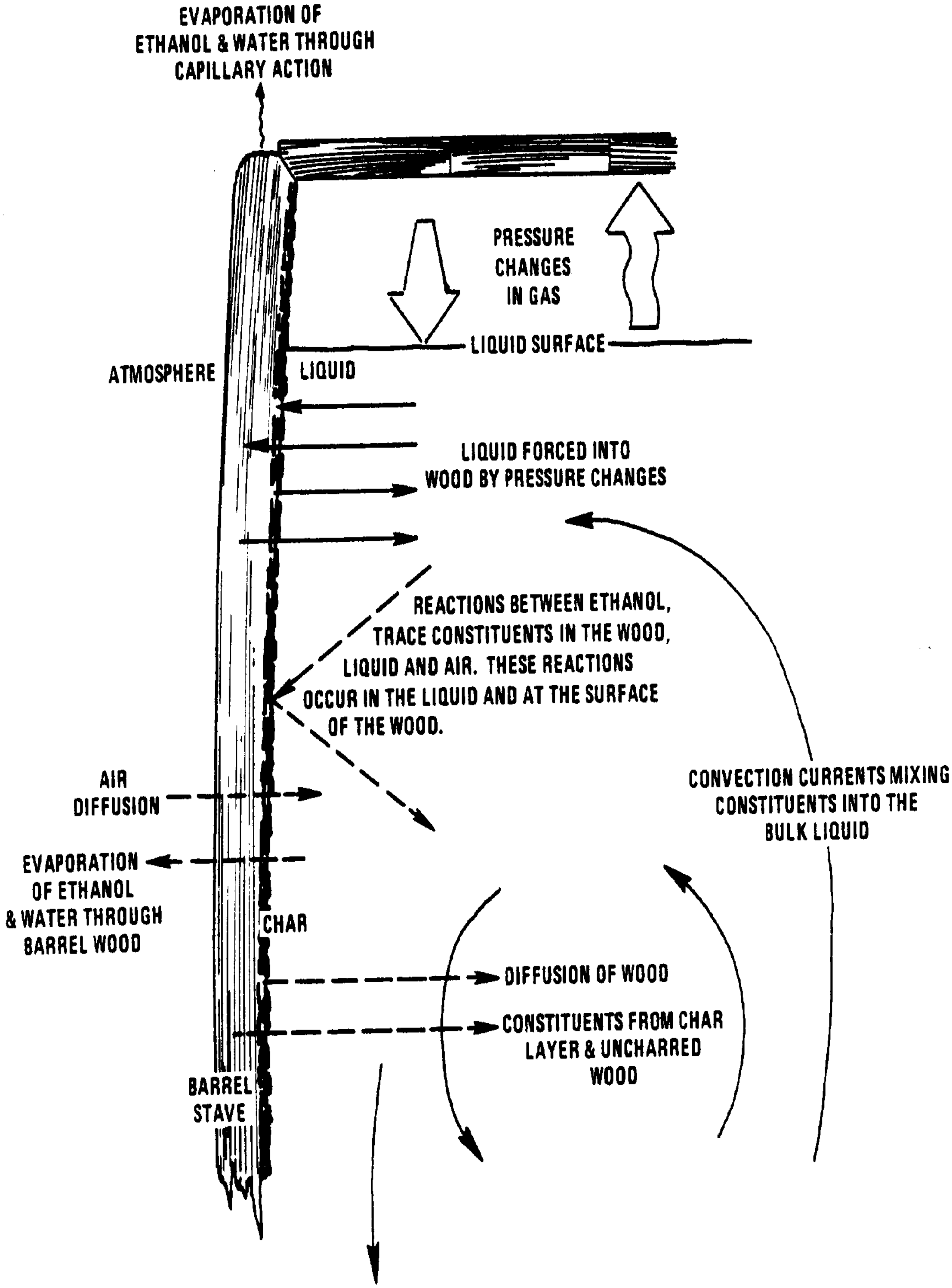


Figure 2-2. Mechanisms of whisky aging.[5]

2.2.7 Blending/Bottling

After the whisky has completed its desired aging period, it is dumped or pumped from barrels into stainless steel tanks and reduced in proof to the desired alcohol concentration by adding demineralized water. The diluted whisky is processed and filtered. Following a filtration process the whisky is pumped to a tank, proof adjusted, and bottled.

Due to their value and salability, used barrels are not generally stored but either refilled with other whiskies or bung sealed and sold to manufacturers of Scotch Whiskey, Canadian Whiskey, rum, brandy, Tequila, or wines.

New bottles are unloaded from cases and put on a conveyor belt, where they are air cleaned, filled, capped, and labeled. At the end of the conveyor belt, the final product is put into cases, which are sealed, labeled, and shipped to distributors.

2.3 EMISSIONS[4-5]

The principal emission from the production of distilled spirits is ethanol, and occurs primarily during aging/warehousing. In addition to ethanol, other volatile compounds produced in trace quantities during aging may include acetaldehyde (a HAP), ethyl acetate, glycerol, fusel oil, and furfural. A comparatively small source of ethanol emissions also results from fermentation. Carbon dioxide is also produced during fermentation; in addition, trace quantities of ethyl acetate, isobutyl alcohol, and isoamyl alcohol are also produced. Particulate matter emissions may result from the grain receiving, grain handling, grain cleaning, milling and grain drying processes; data for those emissions are contained in Section 9.9.1, Grain Elevators and Processes. Whisky production emissions are indicated by process in Figure 2-1. Other emissions, including SO_2, CO_2, CO, NO_x, VOC, and PM, may be generated by fuel combustion from power production in a typical distilled spirits plant.

The emissions from evaporation from the barrel during aging are not constant. During the first 6 to 18 months, the evaporation rate from a new barrel is low because the dry wood must become saturated (known as "soakage") before evaporation from the barrel begins. After saturation, the evaporation rate is greater, but then decreases as evaporation lowers the liquid level in the barrel. The lower liquid level decreases the surface area of the liquid in contact with the wood and thus reduces the surface area subject to evaporation. Loss rates are also affected by temperature and relative humidity. Higher temperatures expand whisky volume, force more whisky into the wood, and increase emission rates. Higher relative humidity reduces water vaporization from the barrel, reducing the emission rate. In addition, humidity affects the barrels themselves; barrels with an initial high wood moisture content shrink as relative humidity decreases, causing increased vaporization from the barrel. This shrinkage also can result in leaks, which are another potential source of emissions.

Minor VOC emissions may be generated when the whisky is drained or pumped from the barrels for blending and bottling, but no emission data are available. In addition, some residual whisky remains in used barrels as both a surface film ("heel") and within the wood ("soakage"). Much of the alcohol in this residue would eventually evaporate if the barrel is left exposed to the atmosphere for a sufficient time. For economic reasons, many distillers collect as much residual whisky as possible by using various processes, such as rinsing with water and vacuum methods.

2.4 EMISSION CONTROL TECHNOLOGY[5]

With the exception of devices for controlling PM emissions, there are few emission controls at distilleries. Grain handling and processing emissions are controlled through the use of cyclones, baghouses, and other PM controls (see AP-42 Section 9.9.1). There are no control technologies for VOC emissions from fermenters because the significant amount of grain solids that would be carried out of the fermenters by vapor entrainment could render systems, such as carbon adsorption, inoperable. Add-on air pollution control devices for whisky aging warehouses are not used because of the anticipated adverse impact that such systems would have on product quality. For economic reasons, distillers ensure that barrel construction is of high quality to minimize leakage, and processes are operated to give the highest finished product alcohol yield. If feasible without impairment of product quality, ethanol recovery would require the use of a collection system to capture gaseous emissions in the warehouse and to process the gases through a recovery system prior to venting them to the atmosphere or recirculating them through the warehouse.

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3. GENERAL DATA REVIEW AND ANALYSIS PROCEDURES

3.1 LITERATURE SEARCH AND SCREENING

Data for this investigation were obtained from a number of sources within the Office of Air Quality Planning and Standards (OAQPS) and from outside organizations. The AP-42 background files located in the Emission Factor and Inventory Group (EFIG) were reviewed for information on the industry, processes, and emissions. The Factor Information and Retrieval (FIRE), Crosswalk/Air Toxic Emission Factor Data Base Management System (XATEF), and VOC/PM Speciation Data Base Management System (SPECIATE) data bases were searched by SCC code for identification of the potential pollutants emitted and emission factors for those pollutants. A general search of the Air CHIEF CD-ROM also was conducted to supplement the information from these data bases.

Information on the industry, including number of plants, plant location, and annual production capacities, was obtained from the *Census of Manufactures* and other sources. A search of the Test Method Storage and Retrieval (TSAR) data base was conducted to identify test reports for sources within the distilled spirits industry. The EPA library was searched for additional test reports. Publications lists from the Office of Research and Development (ORD) and Control Technology Center (CTC) were also searched for reports on emissions from the distilled spirits industry. In addition, the distilled spirits trade association, Distilled Spirits Council of the United States (DISCUS), was contacted for assistance in obtaining information about the industry and emissions.

To screen out unusable test reports, documents, and information from which emission factors could not be developed, the following general criteria were used:

1. Emission data must be from a primary reference:

a. Source testing must be from a referenced study that does not reiterate information from previous studies.

b. The document must constitute the original source of test data. For example, a technical paper was not included if the original study was contained in the previous document. If the exact source of the data could not be determined, the document was eliminated.

2. The referenced study should contain test results based on more than one test run. If results from only one run are presented, the emission factors must be down rated.

3. The report must contain sufficient data to evaluate the testing procedures and source operating conditions (e.g., one-page reports were generally rejected).

A final set of reference materials was compiled after a thorough review of the pertinent reports, documents, and information according to these criteria.

3.2 DATA QUALITY RATING SYSTEM[1]

As part of the analysis of the emission data, the quantity and quality of the information contained in the final set of reference documents were evaluated. The following data were excluded from consideration:

1. Test series averages reported in units that cannot be converted to the selected reporting units;

2. Test series representing incompatible test methods (i.e., comparison of EPA Method 5 front half with EPA Method 5 front and back half);

3. Test series of controlled emissions for which the control device is not specified;

4. Test series in which the source process is not clearly identified and described; and

5. Test series in which it is not clear whether the emissions were measured before or after the control device.

Test data sets that were not excluded were assigned a quality rating. The rating system used was that specified by EFIG for preparing AP-42 sections. The data were rated as follows:

A—Multiple test runs that were performed using sound methodology and reported in enough detail for adequate validation. These tests do not necessarily conform to the methodology specified in EPA reference test methods, although these methods were used as a guide for the methodology actually used.

B—Tests that were performed by a generally sound methodology but lack enough detail for adequate validation.

C—Tests that were based on an unproven or new methodology or that lacked a significant amount of background information.

D—Tests that were based on a generally unacceptable method but may provide an order-of-magnitude value for the source.

The following criteria were used to evaluate source test reports for sound methodology and adequate detail:

1. <u>Source operation</u>. The manner in which the source was operated is well documented in the report. The source was operating within typical parameters during the test.

2. <u>Sampling procedures</u>. The sampling procedures conformed to a generally acceptable methodology. If actual procedures deviated from accepted methods, the deviations are well documented. When this occurred, an evaluation was made of the extent to which such alternative procedures could influence the test results.

3. <u>Sampling and process data</u>. Adequate sampling and process data are documented in the report, and any variations in the sampling and process operation are noted. If a large spread between test results cannot be explained by information contained in the test report, the data are suspect and are given a lower rating.

4. Analysis and calculations. The test reports contain original raw data sheets. The nomenclature and equations used were compared to those (if any) specified by EPA to establish equivalency. The depth of review of the calculations was dictated by the reviewer's confidence in the ability and conscientiousness of the tester, which in turn was based on factors such as consistency of results and completeness of other areas of the test report.

3.3 EMISSION FACTOR QUALITY RATING SYSTEM[1]

The quality of the emission factors developed from analysis of the test data was rated using the following general criteria:

A—Excellent: Developed from A- and B-rated source test data taken from many randomly chosen facilities in the industry population. The source category is specific enough so that variability within the source category population may be minimized.

B—Above average: Developed only from A- or B-rated test data from a reasonable number of facilities. Although no specific bias is evident, it is not clear if the facilities tested represent a random sample of the industries. The source category is specific enough so that variability within the source category population may be minimized.

C—Average: Developed only from A-, B- and/or C-rated test data from a reasonable number of facilities. Although no specific bias is evident, it is not clear if the facilities tested represent a random sample of the industry. In addition, the source category is specific enough so that variability within the source category population may be minimized.

D—Below average: The emission factor was developed only from A-, B-, and/or C-rated test data from a small number of facilities, and there is reason to suspect that these facilities do not represent a random sample of the industry. There also may be evidence of variability within the source category population. Limitations on the use of the emission factor are noted in the emission factor table.

E—Poor: The emission factor was developed from C- and D-rated test data, and there is reason to suspect that the facilities tested do not represent a random sample of the industry. There also may be evidence of variability within the source category population. Limitations on the use of these factors are footnoted.

The use of these criteria is somewhat subjective and depends to an extent upon the individual reviewer. Details of the rating of each candidate emission factor are provided in Section 4.

REFERENCE FOR SECTION 3

1. *Procedures for Preparing Emission Factor Documents*, Second Revised Draft Version, EPA-454/R-95-___, Office of Air Quality Planning and Standards, U. S. Environmental Protection Agency, Research Triangle Park, NC, September 1995.

4. REVIEW OF SPECIFIC DATA SETS

4.1 INTRODUCTION

This section describes the data evaluated and methodology used to develop pollutant emission factors for the manufacture of distilled spirits. In general, the information presented in Section 9.12.3, Distilled Spirits, is new to Chapter 9 of AP-42. The section narrative presented in the current AP-42, Section 6.5 (Fourth Edition), only briefly discusses distilled spirits processes. In this new section, the distilled spirits production process is discussed with emphasis on the whisky-aging process and associated emissions.

4.2 REVIEW OF SPECIFIC DATA SETS

The literature search yielded two documents (References 1 and 2) from which emission factors could be developed. A review of these two documents is given below; full citations for these references are given at the end of this section. Pertinent excerpts from these references are provided in the Appendices. In addition, other references were identified in the literature search or by the industry.

4.2.1 Reference 1

This reference is a 1974 study of emissions from grain fermentation units at a U.S. whisky distillery. It consists of two parts: a 1974 journal article titled "Gaseous Emissions from Whisky Fermentation Units" and an undated preliminary paper with the same title and authors reporting the same data. The results provide the basis for a VOC emission factor from whisky fermentation tanks. Appendix A provides a copy of both references.

Emission source tests were conducted on four closed, steel fermentation vats at an unnamed integrated whisky distillery. Each vat held approximately 121,000 L (32,000 gal) of grain slurry, which yielded 5.14 proof gallons per bushel of grain. Chemical analysis indicated that fermentable sugars in the grain slurry were converted to CO_2, ethyl alcohol, and other VOCs; CO_2 and ethyl alcohol were produced in equivalent molecular quantities. Although carbon dioxide was the bulk constituent of the gas stream, ethyl alcohol and other VOCs also were emitted in the gas stream.

The tests were conducted by sealing off all effluent vents except for the emergency vent. Concurrent velocity and temperature measurements were taken at the emergency vent while sampling. Samples were collected by drawing headspace vapor through charcoal-filled glass tubes at 10-hour intervals. The charcoal sections were analyzed individually by extraction with carbon disulfide and injection into a gas chromatograph equipped with hydrogen flame ionization detectors. The chromatographic results detected six VOCs in the vat emissions; ethyl alcohol represented 99.6 percent of the total VOCs detected. The remaining compounds were: ethyl acetate, *n*-propyl alcohol, isobutyl alcohol, isoamyl acetate, and isoamyl alcohol. Isoamyl acetate and *n*-propyl alcohol were present in trace quantities and could not be quantified.

An emission factor based on quantity of emissions/quantity of grain fermented was developed. The authors' calculations were not given and, therefore, cannot be verified. The test was based on a new methodology conducted at one distillery and lacks sufficient data for confirmation of emission factors. This reference was given a rating of D.

4.2.2 Reference 2

Reference 2 is a 1978 EPA document which discusses the process by which alcohol is emitted from whisky barrels during aging and gives a detailed description of whisky warehouses and operations. Control technologies also are discussed, including ethanol capture and potential reuse, but it is recognized that the utilization of any control technology in a whisky aging warehouse potentially would have an adverse impact on product quality.

Four sets of data were used to estimate emission factors. The first set was U.S. Internal Revenue Service data;[c] distilleries report stocks, withdrawals, and losses to the BATF, which uses the data for taxation purposes. The data used were for the years 1974, 1975, and 1976. The emission factor derived from this data set includes both evaporation and soakage losses because the alcohol loss calculation is based on initial whisky stocks less withdrawals. The estimated emission factors range from 2.99 kg/bbl/yr (6.6 lb/bbl/yr) to 3.27 kg/bbl/yr (7.2 lb/bbl/yr) with an average of 3.15 kg/bbl/yr (6.9 lb/bbl/yr). This emission factor was calculated by subtracting the amount of distilled spirits taken from storage for consumption from the original amount of distilled spirits stored. The other three data sets were from individual distillers, emissions from whisky in bonded warehouses, and losses based on age distribution of bonded whisky in Kentucky in 1975. The emission factor developed from the individual distillers data set was 3.65 kg/bbl/yr (8.0 lb/bbl/yr). For emissions from whisky in bonded warehouses, the emission factor was 3.02 kg/bbl/yr (6.6 lb/bbl/yr). The emission factor developed based on the age distribution data was 3.46 kg/bbl/yr (7.6 lb/bbl/yr). The average emission factor based the three data sets was 3.38 kg/bbl/yr (7.4 lb/bbl/yr). This emission factor includes both evaporative losses and losses due to soakage.

The original calculations for this reference were not available to review. The data were rated D. Pertinent excerpts from the reference are presented in Appendix B.

4.2.3 Reference 3

Reference 3 is a 1992 letter from the Commonwealth of Kentucky adopting an ethanol evaporative emission factor of 7.6 lb/bbl/yr for the aging process. This value was based upon information received from EPA based on Reference 2. Because the emission factor was based on the same data presented in Reference 2, this reference was not used in Section 4.3.2. Reference 3 does not contain actual emission measurements for the industry and is graded D. Appendix C contains a copy of Reference 3.

4.2.4 Reference 4

This report discusses a waste minimization assessment for an unidentified Bourbon distillery that annually produces approximately 5 million gallons of Bourbon and 16,000 tons of distillers dried grains. Annual ethanol emissions (lb/yr) were estimated for five different emission sources but no information was presented for the method used to estimate these emission levels. No descriptions of the production process or any details of the emissions were provided because of facility confidentiality issues.

The data quality are rated D. No data from this reference were used to develop emission factors. An EPA research brief and report cover page are provided in Appendix D.

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[c]The reference refers to these as IRS data, although the publication cited was the Bureau of Alcohol, Tobacco, and Firearms (BATF), U.S. Treasury Department.

4.2.5 Reference 5

Reference 5 is a compilation of regauged tax gallon (RTG) data over a series of aging periods for Bourbon, corn whisky, and light whisky developed by Seagram Americas. The data represent measured whisky volumes (in proof gallons) from barrels after varying stages of the aging process. Based on these data, average total ethanol losses were calculated over an aging time between 4 and 10.5 years for each of the three types of whisky. The average total ethanol losses include both evaporation losses and soakage losses. Calculated total ethanol losses were 3.3 kg/bbl/yr (7.3 lb/bbl/yr) for Bourbon, 3.1 kg/bbl/yr (6.8 lb/bbl/yr) for corn, and 3.9 kg/bbl/yr (8.5 lb/bbl/yr) for light whisky; the average total ethanol loss for the three types is 3.4 kg/bbl/yr (7.5 lb/bbl/yr).

Soakage losses were calculated for each of the three types based on the reported data; the soakage value for Bourbon was confirmed by Seagrams based on actual weight measurements. The average total proof gallon loss, excluding soakage, should be an estimate of losses due to evaporation. The average total ethanol losses due to evaporation were 2.7 kg/bbl/yr (6.0 lb/bbl/yr) for Bourbon, 3.0 kg/bbl/yr (6.5 lb/bbl/yr) for corn, and 3.7 kg/bbl/yr (8.2 lb/bbl/yr) for light whisky; for the three types, the average total ethanol loss due to evaporation is 3.1 kg/bbl/yr (6.9 lb/bbl/yr).

The original data and calculations for this reference were not available to review. The data were rated D. Appendix E contains the data submitted by Seagram Americas and the pertinent calculations for this reference.

4.2.6 Reference 6

Reference 6 is a compilation of whisky loss data over a series of aging periods for Bourbon and corn whisky developed by Jim Beam Brands. The data represent measured whisky losses determined as the difference between proof gallons (PG) entered minus the proof gallons regauged for tax purposes when emptied. Based on these data, average total ethanol losses were calculated over an aging time between 4.7 and 10.5 years for Bourbon whisky and 3.9 and 8.4 years for corn whisky. The average total ethanol losses include both evaporation losses and soakage losses. Calculated total ethanol losses were 4.2 kg/bbl-yr (9.3 lb/bbl/yr) for Bourbon and 3.4 kg/bbl/yr (7.5 lb/bbl/yr) for corn whisky; the average total ethanol loss for the two types is 3.8 kg/bbl/yr (8.4 lb/bbl/yr).

Soakage loss for Bourbon was calculated based on the reported data. The average total PG loss, excluding soakage, should be an estimate of losses due to evaporation. For Bourbon whisky, the total ethanol loss due to evaporation was 3.1 kg/bbl/yr (6.8 lb/bbl/yr).

The original data and calculations for this reference were not available to review. The data were rated D. Appendix F contains the data submitted by Jim Beam Brands and the pertinent calculations for this reference.

4.3 DEVELOPMENT OF CANDIDATE EMISSION FACTORS

Candidate emission factors for the fermentation and for aging are developed below. An alternative estimation method for losses during aging is also presented. No data were available for ethanol or VOC emissions from any source other than fermentation and aging. No data were available for particulate (PM) emissions from grain receiving, handling, cleaning, and milling, and dryer house operations. Emission factors for grain receiving, handling, and cleaning may be found in AP-42 Section 9.9.1, Grain Elevators and Processes.

4.3.1 Whisky Fermentation

The candidate emission factors for four VOCs in whisky fermentation vats (Table 4-1) were taken directly from Reference 1. Distillers report that bushel weights may vary between distilled spirits operations therefore introducing a potential source of error in the application of the emission factor. Because the emission factor was based upon D-rated test data, the emission factor is rated E.

TABLE 4-1. EMISSION FACTORS FOR WHISKY FERMENTATION VATS

EMISSION FACTOR RATING: E

	Emission factor	
VOC	g/m^3 (ppm)	lb/1,000 bu grain input
Ethyl acetate	0.59	0.046
Ethyl alcohol	182.2	14.15
Isobutyl alcohol	0.051	0.004
Isoamyl alcohol	0.17	0.013
Total VOCs	183	14.21

Source: Reference 1 (see Appendix A).

4.3.2 Whisky Aging

A summary of references 2, 5, and 6 for ethanol emissions during the whisky aging process is shown in Table 4-2. Full citations for these references are given at the end of this section. Pertinent excerpts from these references are provided in the Appendices B, E, and F. References 3 and 4 did not contain appropriate emissions data and were not used for emission factor development.

An average ethanol emission factor for total losses during whisky aging was calculated based on the four data sources cited in Table 4-2. The candidate emission factor for total ethanol loss during whisky aging is 3.45 kg/bbl/yr (7.6 lb/bbl/yr). Because the emission factor was based upon D-rated test data, the emission factor is rated E.

An average ethanol emission factor for evaporation losses (total losses minus soakage) during whisky aging was calculated based on the two data sources cited in Table 4-2. The candidate emission factor for ethanol evaporation loss during whisky aging is 3.1 kg/bbl/yr (6.9 lb/bbl/yr). Because the emission factor was based upon D-rated test data, the emission factor is rated E.

TABLE 4-2. SUMMARY OF ETHANOL EMISSION DATA FOR WHISKY AGING

Source	Type of loss	No. of data sets	Data rating	Emission factor range, kg/bbl/yr (lb/bbl/yr)	Average emission factor, kg/bbl/yr (lb/bbl/yr)	Ref. No.
BATF reports	Total[a]	3	D	3.0-3.3 (6.6-7.2)	3.2 (6.9)	2
Distillery data	Total	3	D	3.0-3.7-(6.6-8.0)	3.4 (7.4)	2
Seagrams America	Total	3	D	3.1-3.9 (6.8-8.5)	3.4 (7.5)	5
	Evaporation[b]	3	D	2.7-3.7 (6.0-8.2)	3.1 (6.9)	5
Jim Beam Brands	Total	2	D	3.4-4.2 (7.5-9.3)	3.8 (8.4)	6
	Evaporation	1	D	NA	3.1 (6.8)	6

[a]Total loss incorporates all losses including soakage.
[b]Evaporation loss is defined as total loss minus soakage loss.

Alternatively an ethanol emission factor for total losses during aging and for evaporative losses can be calculated based on annual emissions per barrel in proof gallons (PG). This calculation method is derived from the gauging of product that a distiller is required to perform by the federal government for federal revenue protection purposes. This method measures the difference in the amount of product when the barrel was filled and when the barrel was emptied. Fugitive evaporative emissions, however, are not the sole difference between these two amounts. During the aging period, product soaks into the barrel, test samples are drawn, and other losses (e.g., spillage, leakage) may occur. Soakage only applies to new barrels. Soakage and other losses not volatilized are not evaporative emissions, and thus are subtracted from total product losses. Average annual ethanol emissions per barrel per year is obtained as follows:

1. Divide the total annual proof gallons (PG) sent to aging by the number of barrels filled to obtain the original PG per barrel;

2. Divide the total annual PG emptied by the number of barrels emptied to give regauged PG, which is the amount of ethanol recovered after the entire aging process;

3. Subtract the regauged PG from the original PG to give the total quantity of ethanol per barrel lost (TQL) during the aging process;

4. Total ethanol evaporative emissions, in PG, are obtained by adjusting the TQL for non-volatilized losses such as soakage and samples withdrawn for quality control; and

5. Total evaporative emissions are divided by the number of years of aging to obtain the average annual evaporative emissions, in PG, per barrel.

The annual emissions in proof gallons are then converted to pounds of ethanol per barrel per aging year by dividing by two (2) and multiplying by 6.6097 lb per gallon for 100 percent ethanol at 15.6°C (60°F).

There are a number of methods to calculate barrel soakage. Soakage is the ethanol that soaks into and saturates the new barrel wood during the aging process. This ethanol is retained in the barrel wood when the product is emptied from the barrel and will only be released to the atmosphere at a source if the barrel is not reused within a reasonable period of time. Since barrels generally are put back into service immediately for aging various other products, the differences in losses between new Bourbon barrels and reused barrels can closely approximate the amount of soakage that occurs during the life of a barrel. One estimation method involves determining total ethanol losses per barrel, based on steps 1 through 5 above, for new and reused barrels. For new barrels, total ethanol losses include soakage losses but not for reused barrels. The difference between total ethanol losses for new barrels and for reused barrels can be used as an estimate of soakage losses. With this method, it is important that entry proofs of both new and used barrels be close to the same strength and that the barrels are stored under similar warehouse conditions. There is no exclusive method to calculate soakage and factors such as entry proof, individual barrel characteristics, differences in the water content of the wood, and differences in aging practices, can impact the amount of soakage. In addition, the method for estimating soakage may differ between distillers.

4.4 SUMMARY OF CHANGES TO AP-42 SECTION

4.4.1 Section Narrative

The previous AP-42 section incorporated distilled spirits production into an overall section entitled "Fermentation" but no process description or process flow diagram was provided. This new section provides a description of the current production practices and a process flow diagram for a typical whisky production facility.

4.4.2 Emission Factors

The previous AP-42 section presented emission factors based on outdated production processes. This new section replaces the existing emission factors with data consistent with current practices in the distilled spirits industry.

REFERENCES FOR SECTION 4

1. Carter, R. V., and B. Linsky, "Gaseous Emissions from Whiskey Fermentation Units," *Atmospheric Environment*, 8:57-62, January 1974; also a preliminary paper of the same title by these authors (undated).

2. *Cost and Engineering Study-Control of Volatile Organic Emissions from Whiskey Warehousing*, EPA-450/2-78-013, Emissions Standards Division, Chemical and Petroleum Branch, Office of Air Quality Planning and Standards, U. S. EPA, Research Triangle Park, NC, April 1978.

3. Written communication from J. E. Hornback, Department For Environmental Protection, Commonwealth of Kentucky, Frankfort, KY, to H. E. O'Daniel, Jr., Kentucky Distillers Association, Springfield, KY, September 18, 1992.

4. Fleischman, M., et al., "Waste Minimization Assessment to a Bourbon Distillery", EPA/600/5-95/002, Risk Reduction Engineering Laboratory, U. S. Environmental Protection Agency, Cincinnati, OH, April 1995.

5. Written communication from R. J. Garcia, Seagrams Americas, Louisville, KY, to T. Lapp, Midwest Research Institute, Cary, NC, March 3, 1997. RTG's versus age for 1993 standards.

6. Written communication from L. J. Omlie, Distilled Spirits Council of the United States, Washington, DC, to T. Lapp, Midwest Research Institute, Cary, NC, February 6, 1997. Ethanol emissions data from Jim Beam Brands Company.