AP42 Section:	9.12.3
Title:	Emission Factor Documentation for AP-42 Section 9.12. Distilled Spirits Final Report March 1997 APPENDICES ONLY

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
Research Triangle Park, NC 27711

Attn: Mr. Dallas Safriet (MD-14)

Appendices only

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

MRI Project No. 4604-04

March 1997

APPENDIX A

EXCERPTS FROM REFERENCE 1

(Carter and Linsky, 1974)

Atmospheric Environment Vol. 8, pp. 57–62. Pergamon Press 1974. Printed in Great Britain.

GASEOUS EMISSIONS FROM WHISKEY FERMENTATION UNITS

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(*First received* 18 *September* 1972 *and in final form* 24 *July* 1973)

Abstract—Source testing for air pollutants was conducted on grain fermentation units in a whiskey distillery. Odorants were adsorbed on activated carbon and analyses were made by gas chromatography. Six compounds found in the predominately carbon dioxide gas stream were ethyl acetate, ethyl alcohol, *n*-propyl alcohol, isobutyl alcohol, isoamyl alcohol and isoamyl acetate. Ethyl alcohol comprised more than 99 per cent (by volume) of the organic concentration. Experimental data indicated that instantaneous organic contaminant concentrations were functions of time in the batch-type fermenting process. Results are presented in tabular and graphical form and may be used to estimate emissions from similar processes. Commentary is included as to the reliability and accuracy of the data.

INTRODUCTION

There is a growing awareness of atmospheric pollutants that cause unpleasant or offending odors. Odors are generally detected by the human sense of smell at levels below the detectable limits of most portable and much of the usual laboratory instrumentation. This report describes and quantifies odor producing gaseous emissions from fermenting units at whiskey distilleries with reasonably sensitive sampling and analytical procedures. Although fermenting procedures vary somewhat throughout the industry, the basic method of whiskey production is common to all. Adequate process description is available (Benton, 1960; Rose, 1961). During the batch type process, fermentable sugars are converted to carbon dioxide and ethyl alcohol in equal molecular quantities. Other volatile organic compounds are also formed, some of which are released to the atmosphere with carbon dioxide as the carrier.

Several researchers have studied alcoholic beverages as the product of distillation, but few have published information defining the vapors from fermentation (Smith, 1952; Komoda *et al.*, 1968; Kahn, 1969). This paper reports emission data from four similar fermenting vats in an integrated whiskey distillery.

EXPERIMENTAL METHODS

Samples of effluent were collected from closed steel vats containing approximately 121 120 l. of grain slurry, each of which yielded 5.14 proof gallons‡ of ethyl alcohol per bushel of grain.

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‡ A proof gallon is a standard U.S. gallon of 231 in^3 (3786 cm^3) at a temperature of 15.6°C and at 100 proof. 100 proof is equal to 50 per cent by volume.

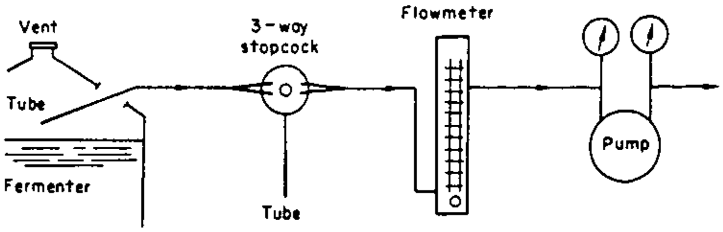

Fig. 1. Sampling apparatus.

Emission parameters were measured at the top vents while vapor samples were collected at the side hatch openings both at intervals of approximately 5 h (Fig. 1). Contaminants were adsorbed onto R.C. type Barneby Cheney activated charcoal contained in 5 mm i.d. glass tubes. The charcoal was separated into 2.5 cm sections by plugs of medium fine pyrex glass wool.

Charcoal sections were removed from the sampling tubes and analyzed individually. Extraction was accomplished with the addition of carbon disulfide, shaking and overnight desorption. Aliquots of the supernatant (5 μl) were injected into a Varian Aerograph, Model 2100 gas chromatograph equipped with hydrogen flame ionization detectors. Two 6.1 mm × 3.2 m stainless steel columns with 10 per cent FFAP stationary phase on 80/100 mesh acid washed DMCS Chromosorb W solid support were employed to separate the components. Standard mixtures were similarly analyzed. Additional information about various aspects of the sampling and analytical techniques used may be found in publications by Brooman and Edgerly (1966), Fraust and Hermann (1966) and White *et al.* (1970).

Table 1. System operating conditions

A. Gas chromatograph
  1. Nitrogen carrier flow at 45 701·kg m^{-2}
  2. Hydrogen flow at 17 577 kg m^{-2}
  3. Air flow at 35 155 kg m^{-2}
  4. Injector-detector temperature, 200°C
  5. Electrometer attenuation, 32 *x*
  6. Range, 10^{-11} AmV^{-1}
  7. Oven temperature program:
    a. Initial temperature, 90°C
    b. 15°C rise for 6 min
    c. Final temperature, 180°C for 3 min

B. Recorder-integrater
  1. Attenuation, 1 *x*
  2. Peak width at 1/2 height, 10 s
  3. Slope sensitivity, 2–5
  4. Digital baseline corrector rate, maximum
  5. Recorder chart speed, 102 cm h^{-1}

RESULTS

Investigation determined that six organic compounds were present in the vat gas effluent. These compounds were ethyl acetate, ethyl alcohol, *n*-propyl alcohol, isobutyl alcohol, isoamyl acetate and isoamyl alcohol. Figure 2 shows a typical sample chromatogram. Detailed gas chromatographic analyses were performed on all six compounds with

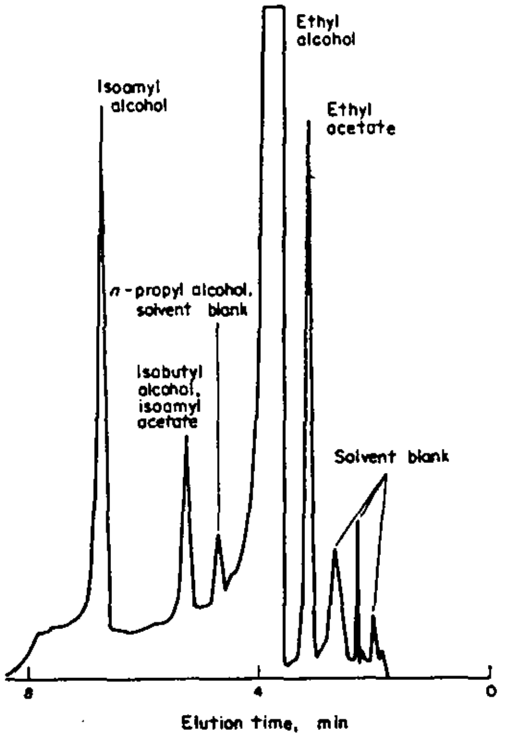

Fig. 2. Chromatogram, charcoal No. 1.

the exception of the isoamyl acetate and *n*-propyl alcohol. The analytical results yielded for these compounds were not reproducible because of analytical interferences.

It appeared that a general trend was established over the fermenting period as to contaminant concentration in the vat gas (Fig. 3). Statistical analysis of variance tests on the data showed that a significant increase in concentration with time did exist at the 0.005 confidence level. Additionally, no significant variation was noticed between vats at the 0.05 confidence level. Based on this statistical evidence, the data from all vats were grouped together and considered as one "average" source. A quadratic least squares curve fitting equation was used to adjust lines of best fit through the measured data. This enabled a better definition of a specific compound's concentration at any given time.

Total volume measured emission data could not be fitted satisfactorily with mathematical curve fitting techniques. The measured data was plotted in Fig. 4 and an approximate curve entered by hand. Total volume emission values for individual vats were found by plotting each data set separately (not shown). The active fermentation period was separated into 150 min intervals in order to arrive at figures representing the total volume emitted during that interval. These values were summed to arrive at data as shown in Table 2.

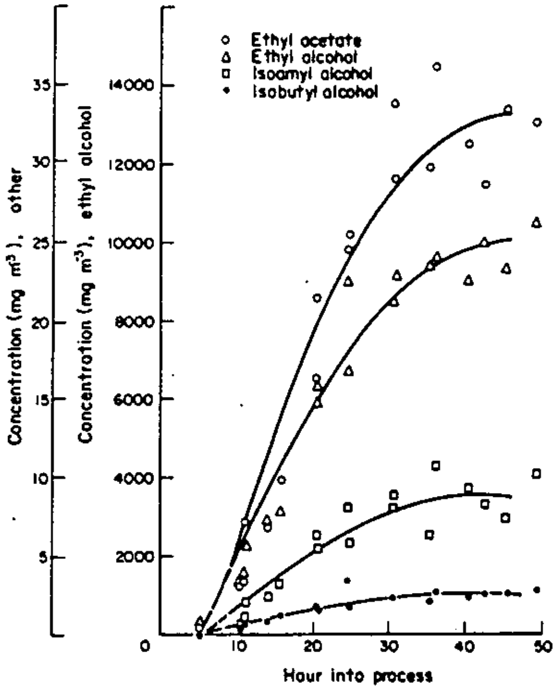

Fig. 3. Organic concentration.

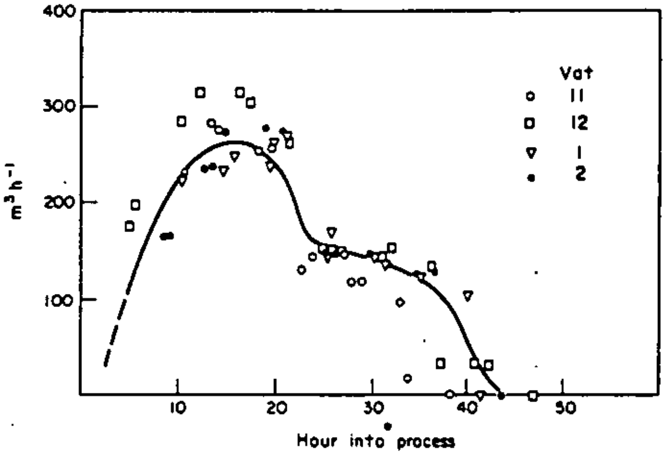

Fig. 4. Measured exit gas (composite).

Table 2. Total volume of emitted gas (21°C, 760 mm Hg)

Vat number	11	12	1	2
Volume (m³)	5215	5805	5770	6140

The volume emitted during a specified time interval was correlated with the average concentration of each compound during that same interval. Considering the measured data, the total amount of organic compound emitted from the process per unit of raw material was calculated. Table 3 shows the weight of compound emitted per volume of grain input taken over the entire fermentation process.

Table 3. Organic emissions from whiskey fermentation vats*

	Vat number				
Compound	11	12	1	2	Ave.
Ethyl acetate	0.499	0.594	0.627	0.654	0.593
Ethyl alcohol	156.7	181.5	190.9	199.5	182.2
Isobutyl alcohol	0.044	0.051	0.053	0.055	0.051
Isoamyl alcohol	0.141	0.167	0.175	0.183	0.166

* Expressed as g emitted per m^3 of grain input.

SUMMARY AND CONCLUSIONS

There is evidence in the literature that not all of the organic compound is desorbed from activated charcoal by carbon disulfide (Brooman *et al.*, 1966; Carter, 1971; Fraust *et al.*, 1966). From information available on the compounds studied in this report, as much as 30 per cent of the total may remain on the charcoal after the desorption procedures employed in this project were completed. For some compounds however, all is desorbed and none remains as residual. Therefore, it must be cautioned that the quantitative data as measured and presented were lower than what actually existed at the emission point by 0–30 per cent. The limited number of data on desorptive efficiencies make it improper to improve experimental accuracy by means of an adjustment factor.

The results showed that at least six organic compounds were present in the gas stream in measurable quantities: ethyl acetate, ethyl alcohol, isopropyl alcohol, *n*-propyl alcohol, isoamyl alcohol and isoamyl acetate. Other compounds were detected by the chromatograph but were present in trace amounts only.

Odors present in the vicinity of a distillery, while not scientifically documented at this time, are thought to be the result of gaseous organic compounds emanating from cooking, fermenting and drying process operations of whiskey production. The experimental results contained in Table 3 may be used to estimate contaminant emissions from whiskey fermentation. Although the emission rates are subjected to variations from different process types and alcohol yields, one may generally assume a linear relationship between input materials (grain) and organic substance emissions.

Acknowledgements—Initial investigation was supported by the Kentucky Air Pollution Control Commission, Frankfort, Kentucky. Efforts were completed under an Office of Air Programs, Environmental Protection Agency training grant at West Virginia University, Morgantown, West Virginia. The authors wish to thank Lowell D. White, Charles V. Cooper, Richard E. Kupel and the staff of the Bureau of Occupational Safety and Health, Public Health Service at Cincinnati, Ohio, for their assistance in the development of collection and analytical techniques.

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PAPER

GASEOUS EMISSIONS FROM
WHISKEY FERMENTATION UNITS

Subject Area: Ambient Air
and Source Measurements

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INTRODUCTION

There is a growing awareness of atmospheric pollutants that cause unpleasant or offending odors. Odors are generally detected by the human sense of smell at levels below the detectable limits of most portable and much of the usual laboratory instrumentation. Such contaiminants are too often assumed to be non-toxic below that of the initial sensory response. It is the opinion of the authors that regulations specifying the number of dilutions which produce no subject response are inadequate. Control measures are often applied to an odor source without adequate knowledge of physical and behavorial properties of each contaminant.

This report therefore attempts to describe and quantify gaseous pollutant emissions from fermenting units at whiskey distilleries with reasonably sensitive sampling and analytical methods and procedures. Although fermenting procedures vary somewhat throughout the industry, the basic method of whiskey production is common to all. Adequate process descriptions may be found in several references[6]. During the process, fermentable sugars are converted to carbon dioxide and ethyl alcohol in equal molecular quantities. Other volatile organic compounds are also formed, some of which are released to the atmosphere with carbon dioxide as the carrier.

Several researchers have studied alcoholic beverages as the product of distillation but few have published information defining the vapors from fermentation. This paper reports emission data from four similar fermenting vats in an integrated whiskey distillery. Samples of effluent were collected from closed steel vats containing approximately 32,000 gallons of grain slurry each which yielded 5.14° proof gallons* of ethyl alcohol per bushel of grain.

> *A proof gallon is a standard U.S. gallon of 231 cubic inches at a temperature of 60° F. and at 100° proof (100° proof is equal to 50% alcohol by volume.)

COLLECTION OF SAMPLES

All effluent vents were sealed off with the exception of an emergency vent ten inches in diameter located on the top center of each vat. Velocity and temperature measurements were made at these emergency openings while the tube samples described below were taken through the more accessible side hatches.

Measurements of effluent temperature and exit velocity (determined with an Alnor velometer) were made at approximately five hour intervals. Head space vapor samples were collected in charcoal filled glass tubes at 10 hour intervals. Five millimeter ID glass tubing was cut into ten inch sections and the ends fire-polished. R C type Barneby Cheney activated charcoal was added to the tubes and loosely packed into one inch sections, each separated by one-half inch plugs of medium fine Pyrex glass wool. The charcoal was pre-conditioned by overnight heating at 200°C and returned to room temperature in a dessicator. The finished tube was flame sealed to prevent contamination prior to sampling. The number of charcoal sections desired dictated the length of the tube. Initial sampling indicated that six-one inch sections of charcoal were sufficient to prevent sample penetration.

The sampling apparatus shown in Figure 1 consisted of two sampling tubes, a 3-way glass stopcock value, a Brooks flowmeter #448-225, heavy walled Tygon tubing and a Gast vacuum pump equipped with pressure gages.

The flow meter and velometer were calibrated according to the manufacturers recommended procedures.

~~Stopcock in Sampling Position~~

~~Key to Numbers~~

~~1. Fermenter~~
~~2. 3-way Stopcock Value~~
~~3. Flowmeter~~
~~4. Vacuum Pump~~
~~5. Used Sampling Tube~~
~~6. Sampling Tube~~
~~7. Side Hatch~~
~~8. Emergency Vent~~

~~Figure 1. Sampling Apparatus~~

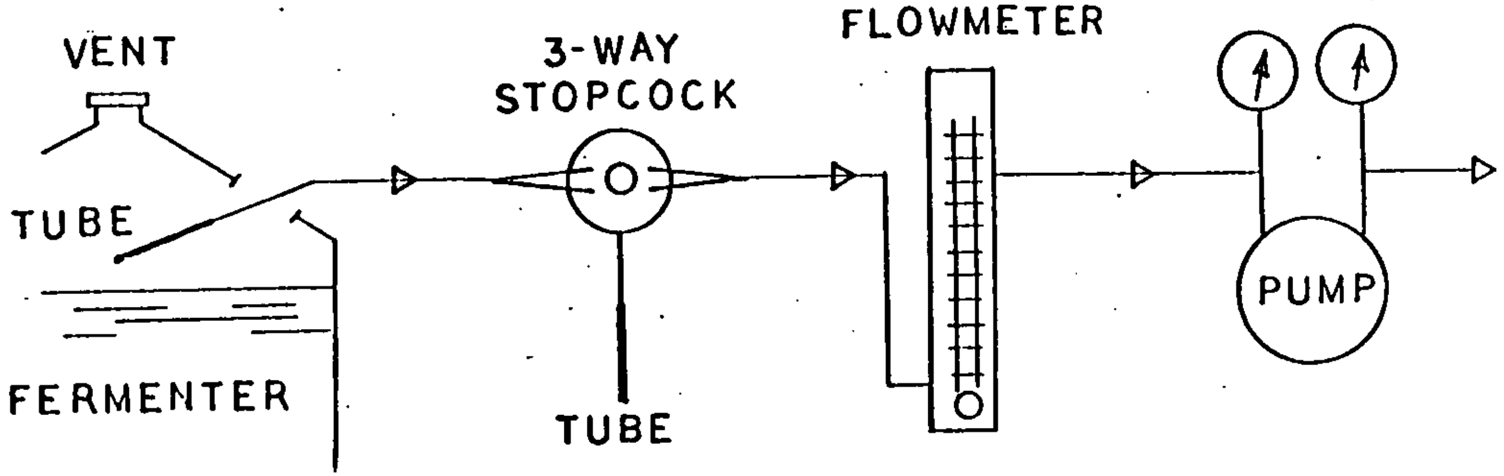

FIGURE 1. SAMPLING APPARATUS

The sampling procedure was identical in all cases. Vat effluent was drawn through the charcoal tube at the rate of 3.1 liters per minute for periods varying from one to five minutes. All of the sample entered the sampling tube directly, thereby, avoiding any chance of contamination by the sampling apparatus. The tube was removed from the Tygon tubing and sealed with parafilm.

ANALYTICAL PROCEDURE

The six charcoal sections from a sampling tube were analyzed individually. Each section was placed in 15 x 125 mm Kimax culture tubes and exactly 1.5 ml carbon disulfide was added with a volumetric delivery pipette. (Carbon disulfide presents a considerable fire hazard and, as a gas, is harmful to breathe. All transfers were made in a hood.) Culture tubes were equipped with Teflon lined caps to minimize the escape of harmful gases. The carbon disulfide and activated charcoal were shaken initially and again just before the analysis was performed. The mixture was allowed to remain undisturbed overnight (8 to 12 hours) to maximize desorption. A 10 μl Hamilton syringe was used to extract and inject 5 μl aliquots of the supernatant into the gas chromatograph. A Varian Aerograph, Model 2100 gas chromatograph was equipped with hydrogen flame ionization detectors. A 20 ft. x 1/8 in. stainless steel column with 10% FFAP stationary phase on 80/100 mesh acid washed DMCS Chromasorb W solid support was employed to separate the components. Table 1 shows the specific operating conditions and detailed description of the analytical system.

TABLE 1. SYSTEM OPERATING CONDITIONS

A. Gas Chromatograph:

1. Nitrogen carrier flow @ 65 psig, 25 cc/min
2. Hydrogen flow @ 25 psig, 25 cc/min
3. Air flow @ 50 psig, 300 cc/min
4. Injector-detector temperature, 200°C
5. Electrometer attenuation, 32x
6. Range, 10^{-11} amps/mv
7. Oven temperature program:
    a. Initial temperature, 90°C
    b. 15°C rise for 6 minutes
    c. Final temperature, 180°C for 3 minutes

B. Recorder-Integrater Operating Conditions

1. Attenuation, 1x
2. Peak width at 1/2 height, 10 seconds
3. Slope sensitivity, 2 to 5
4. Digital baseline corrector rate, maximum
5. Recorder chart speed, 40 in/hr

Standard curves were established for each day of analytical operation at the conditions described. The curves were prepared by plotting the average number of integrater digital counts (representative of peak area) verses standard concentrations. Standard mixtures were prepared daily (because of the rapid evaporation of the components and solvent) at different concentrations in order to encompass that of the unknown. Standard mixtures were injected at regular intervals during the analyses to minimize errors that arise from instrument drift and gas tank pressure changes.

Instrument responses to the unknown samples were related to those of the known standard concentrations. From the data, the concentration of each component in the fermentation vat headspace can be calculated. The calculations were based on sample sizes adjusted to 70°F and 760 mm Hg pressure. For the remainder of this report, 70°F temperature and 760 mm Hg pressure will be referred to as "reference conditions."

An equation was devised to express the relationship between instrument response and the source concentrations.

$$C = \frac{1500\rho A}{L}$$

where C = source concentration (mg/M^3)

ρ = organic compound liquid density (gm/ml)

A = compound concentration in sample solution (ppm by volume)

1500 = conversion factor for unit balance

L = sample size (liters at reference conditions)

Conversion to parts per million by volume (P) at reference conditions may be accomplished as shown.

$$P = \frac{24.13}{M} C$$

small w small m

Where M = Molecular weight ($\frac{gms}{mole}$)

24.13 = Conversion factor for unit balance

Investigation determined that six organic compounds were present in the vat gas effluent. These compounds were ethyl acetate, ethyl alcohol, n-propyl alcohol, isobutyl alcohol, isoamyl acetate and isoamyl alcohol. Figure 2 shows a typical sample chromatogram. It was determined that 3 to 7 liter samples at reference conditions provided sufficient

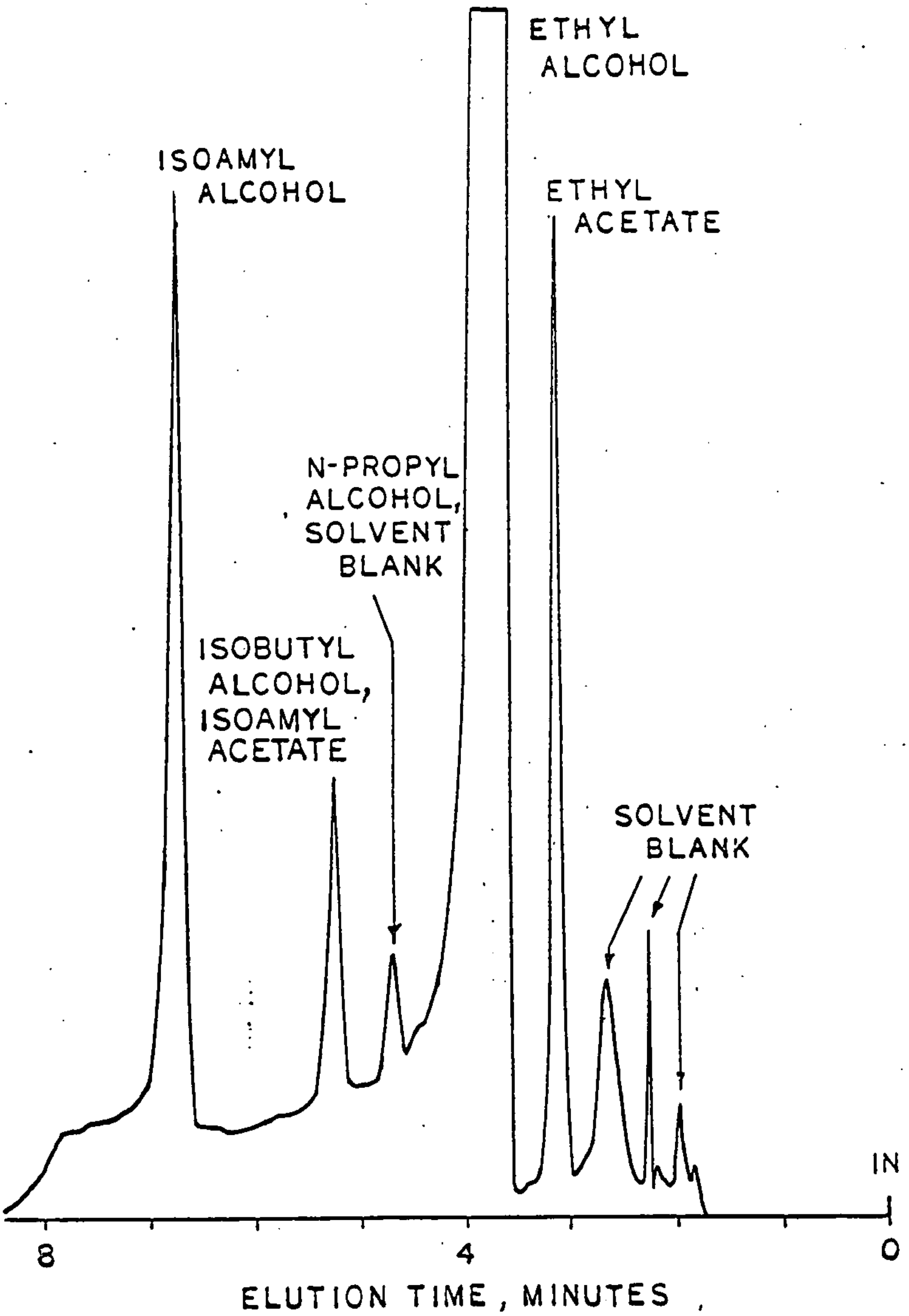

FIGURE 2. CHROMATOGRAM, CHARCOAL SECTION #1

quantities of substance to cause instrument response within detectable and maximum system operating limits. [~~Reference conditions for this report are 70°F and 760 mm mercury.~~] Sample of 7 liters or larger allowed organic substance penetration through the collecting tube, if taken during the latter half of the fermentation period when the highest concentrations were present.

Detailed gas chromatographic analyses were performed on all six compounds with the exception of isoamyl acetate, which was present in only trace quantities. An analytical system could not be devised by the investigator to separate the isoamyl acetate from isobutyl alcohol to an extent allowing separate peak integration by the digital integrator. Because of its low concentration and the relatively large interference with the correct integration of isobutyl alcohol, calculations were made to substract the acetate response from the combined response. Operating on an expanded temperature program, the relative peak areas were determined and a conversion factor devised to relate isobutyl alcohol concentration to the total response.

n-Propyl alcohol was also present in the vat gas in minute concentrations, but was eluted simultaneously with a contaminant common to the unexposed charcoal tubes which served as "blank" samples. Data obtained for n-propyl alcohol was not considered reproducible and did not indicate process trends characteristic of the other compounds when analyzed statistically.

Each section from 21 samples was analyzed individually in duplicate. If there was not satisfactory agreement between values, additional gas chromatograph injections were made and recorded. Data from each of the six sections were summed to yield the concentration of four compounds.

EXPERIMENTAL RESULTS

It appeared that a general trend was established over the period of fermentation as to compound concentration in the vat gas (Table 2). Statistical analysis of variance tests on the data (excepting period A values) showed that there was a significant increase in concentration with time at the .005 confidence level. Additionally, no significant variation was noticed between vats at the .05 confidence level.

TABLE 2. MEASURED ORGANIC CONCENTRATION
(mg/M^3)

COMPOUND	VAT	TIME PERIOD (hours)				
		A	B	C	D	E
Ethyl Acetate	11	3.02	9.82	25.5	29.9	33.3
	12	N	7.27	16.4	33.9	31.3
	1	N	3.35	21.5	29.1	32.7
	2	0.427	6.75	24.6	36.2	28.6
Ethyl Alcohol	11	1303.	3102.	6737.	9420.	9319.
	12	N	2214.	5896.	8526.	9009.
	1	N	1522.	6299.	9164.	10,585.
	2	269	2873.	8513.	9626.	10,005.
Isobutyl Alcohol	11	0.49	1.075	1.66	2.018	2.61
	12	N	0.86	1.84	2.21	2.46
	1	N	0.507	1.49	2.49	2.95
	2	0.0	0.860	3.47	2.76	2.55
Isoamyl Alcohol	11	0.95	3.07	5.92	6.36	7.31
	12	N	2.15	6.14	8.15	9.28
	1	N	1.09	5.44	8.80	10.2
	2	0.0	2.34	8.00	10.7	8.15

Key to Symbols

A = 0.0 to 10.25 hours
B = 10.50 to 15.5 hours
C = 20.17 to 24.83 hours
D = 30.50 to 36.25 hours
E = 40.25 to 49.25 hours
~~ppm = parts per million by volume~~
N = no sample taken
mg/M^3 = MILLIGRAMS PER CUBIC METER

Based on this statistical evidence, the data from all vats were grouped together and considered as one "average" source. A quadratic least squares curve fitting equation was used to adjust lines of best fit through the measured data (Figures 2 to 5)[8]. This enabled a better definition of a specfic compound's concentration at any given time.

Total volume measured emission data could not be fitted satisfactorily with mathematical curve fitting techniques. The measured data was plotted in Figure 6 and an approximate curve entered by hand. Emission values for individual vats were found by plotting each data set separately (not shown). The active fermentation period was separated into 150 minute intervals in order to arrive at figures representing the total volume emitted during that interval. These valued were summed to arrive at data as shown in Table 3.

Table 3. Total Volume of Emitted Gas
(Adjusted to Reference Conditions)

Vat	11	12	1	2
Volume (M^3)	5215.	5805.	5770.	6140.

It was now possible to arrive at the total amount of organic compound emitted from the process per unit of raw material. A scheme was devised to enumerate emission data for each compound found in the vat gas. For example, the volume emitted during a specified time interval was correlated with the concentration of each compound during that same interval. By applying this relation to all time intervals, the total quantity of each organic compound emitted was obtained for the entire fermentation period.

Table 4. Organic Emissions from Whiskey Fermentation Vats *

	Vat Number				
Compound	11	12	1	2	ave.
Ethyl Acetate	17.58	20.94	22.11	23.06	20.91
Ethyl Alcohol	5523.	6396.	6729.	7029.	6419.
Isobutyl Alcohol	1.54	1.81	1.85	1.94	1.79
Isoamyl Alcohol	4.97	5.89	6.18	6.45	5.87

*** Expressed as grams emitted per 1000 bushels of grain input.**

CONCLUSION

Gaseous effluent from grain fermentation was found to contain 4 organic compounds in significant quantities, ranging from 1.54 to 7029 grams per 1000 bushels of grain input in a gas stream of more than 98 percent carbon dioxide. Emission factors for the organic compounds as sampled and analyzed are presented in Table 4. These air pollutant emission factors can be applied to fermentation units in other distilleries for generalized emission figures where source testing is not carried out. Process information, sampling and analytical procedures are presented to provide a base for planning and evaluating future source testing by others.

There is evidence in the literature that not all of the organic compound is desorbed from activated charcoal by carbon disulfide [1,2,9]. From information available on the compounds studied in this report, as much as 30 percent of the total may remain on the charcoal after the desorption procedures employed in this project. For some compounds however, all is desorbed and none remains as residual. It must be cautioned that the quantitative data as measured and presented are lower than what actually exists at the emission point from the source by 0 to 30 percent. Further testing with improved procedures might be expected to more accurately determine the real emission concentrations of individual compounds.

ACKNOWLEDGEMENTS

Initial investigation was supported by the Kentucky Air Pollution Control Commission,Frankfort, Kentucky. Efforts were completed under an Office of Air Programs, Environmental Protection Agency training grant. The authors wish to thank Lowell D. White, Charles V. Cooper, Richard E Kupel and the staff of the Bureau of Occuptional Safety and Health, Public Health Service at Cincinnati, Ohio, for their assistance in the development of collection and analytical techniques.

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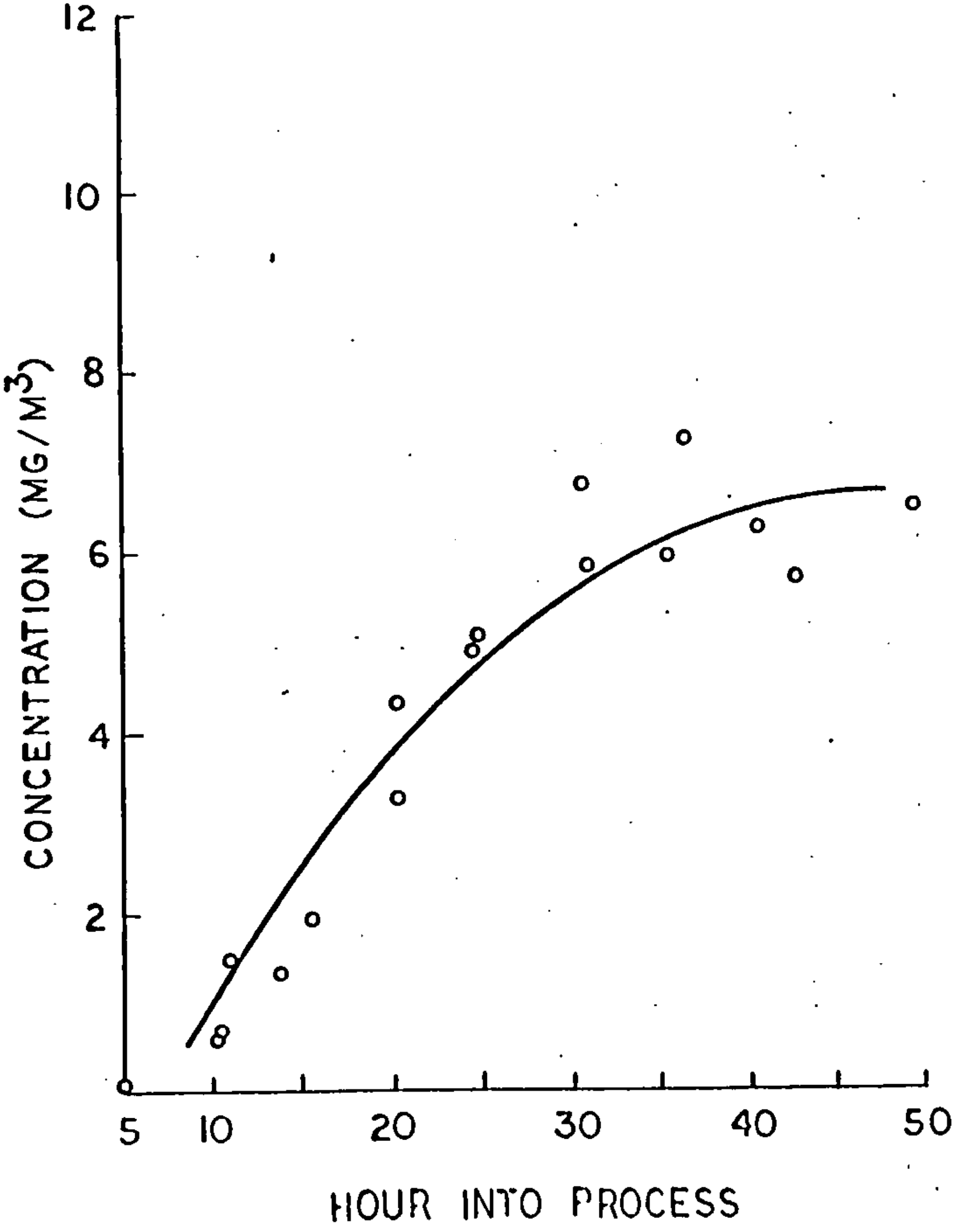

CONCENTRATION (MG/M³)
12
10
8
6
4
2
5
10
20
30
40
50
HOUR INTO PROCESS

FIGURE 7

7

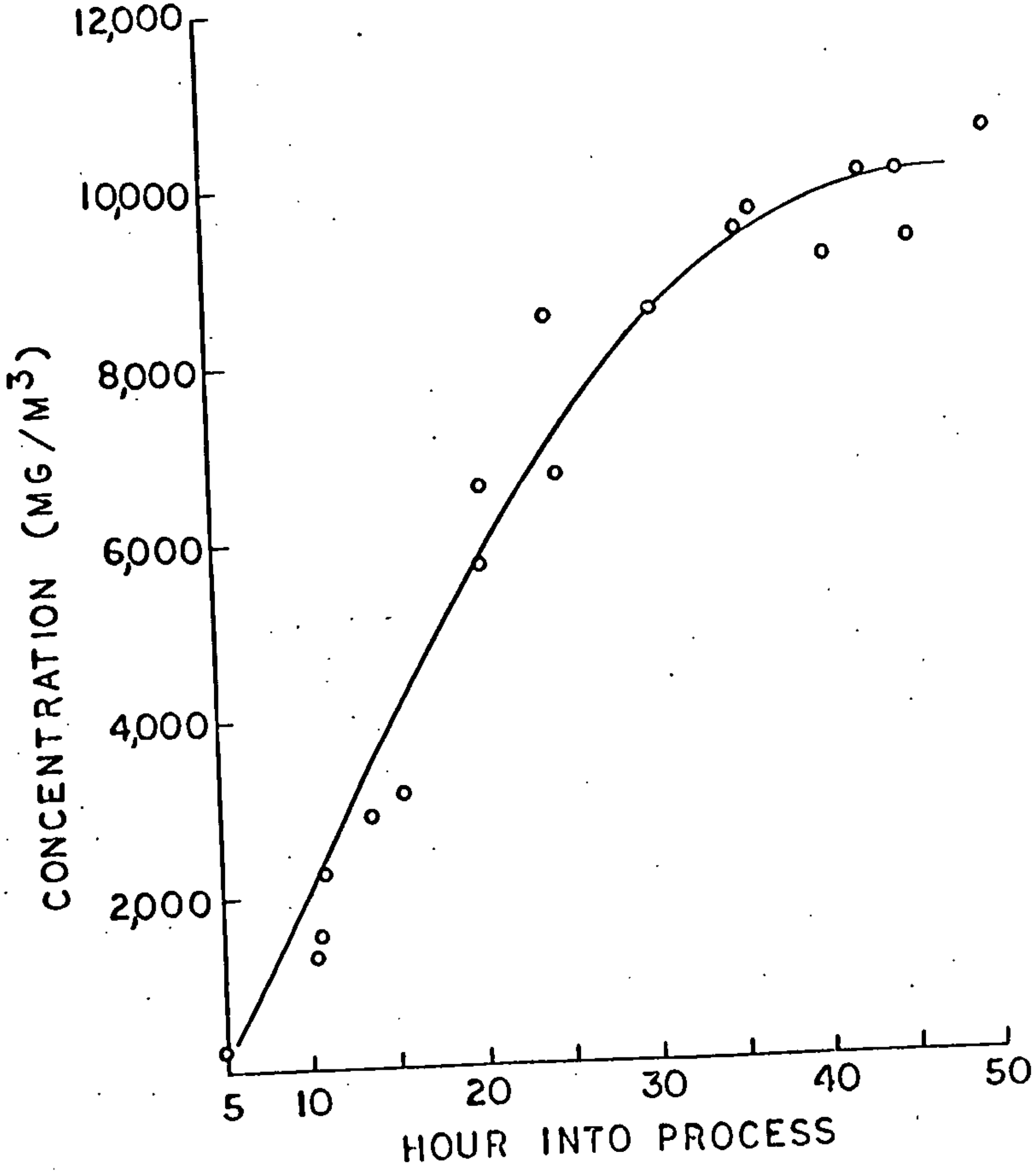

FIGURE 4. ETHYL ALCOHOL

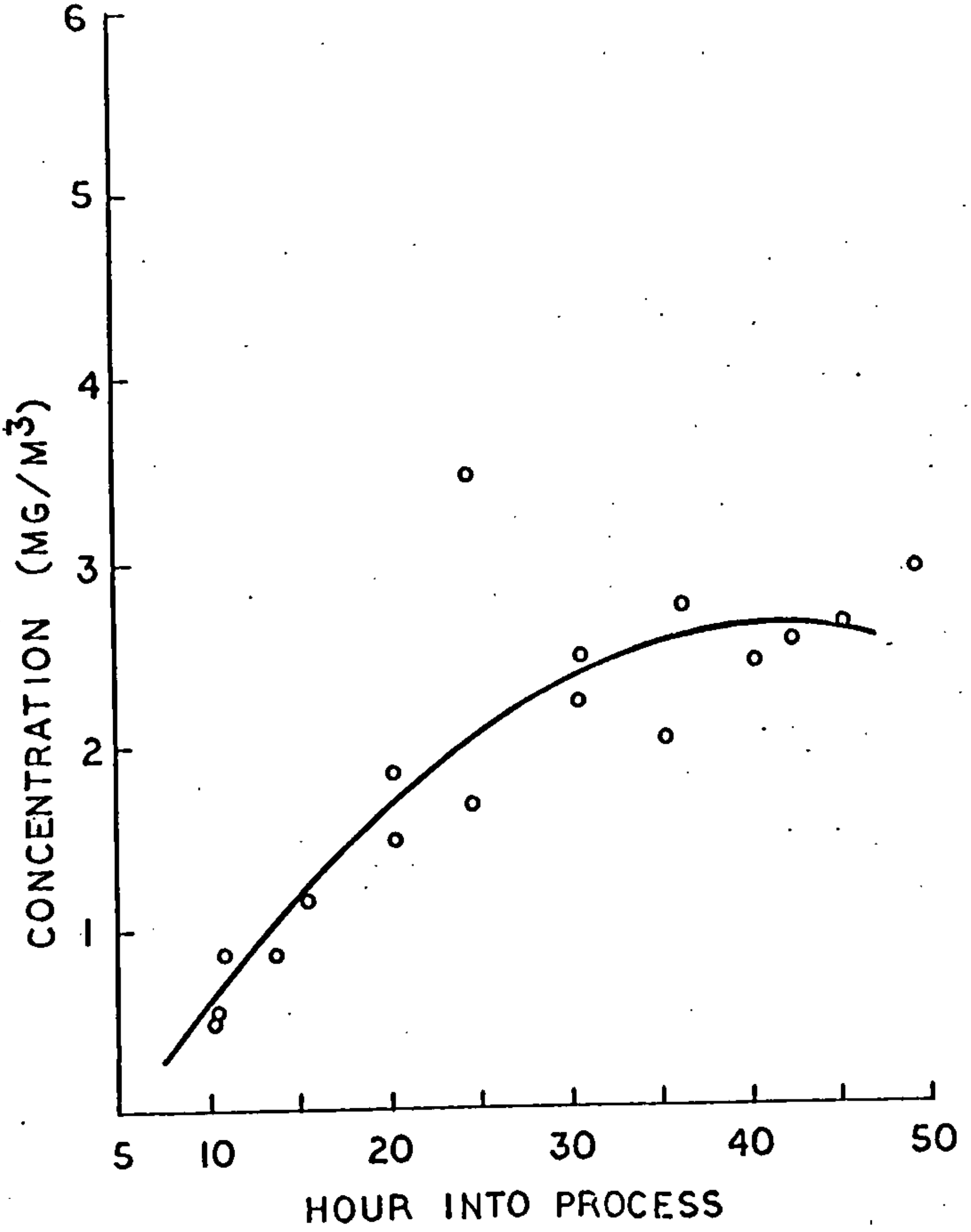

FIGURE 5. ISOBUTYL ALCOHOL

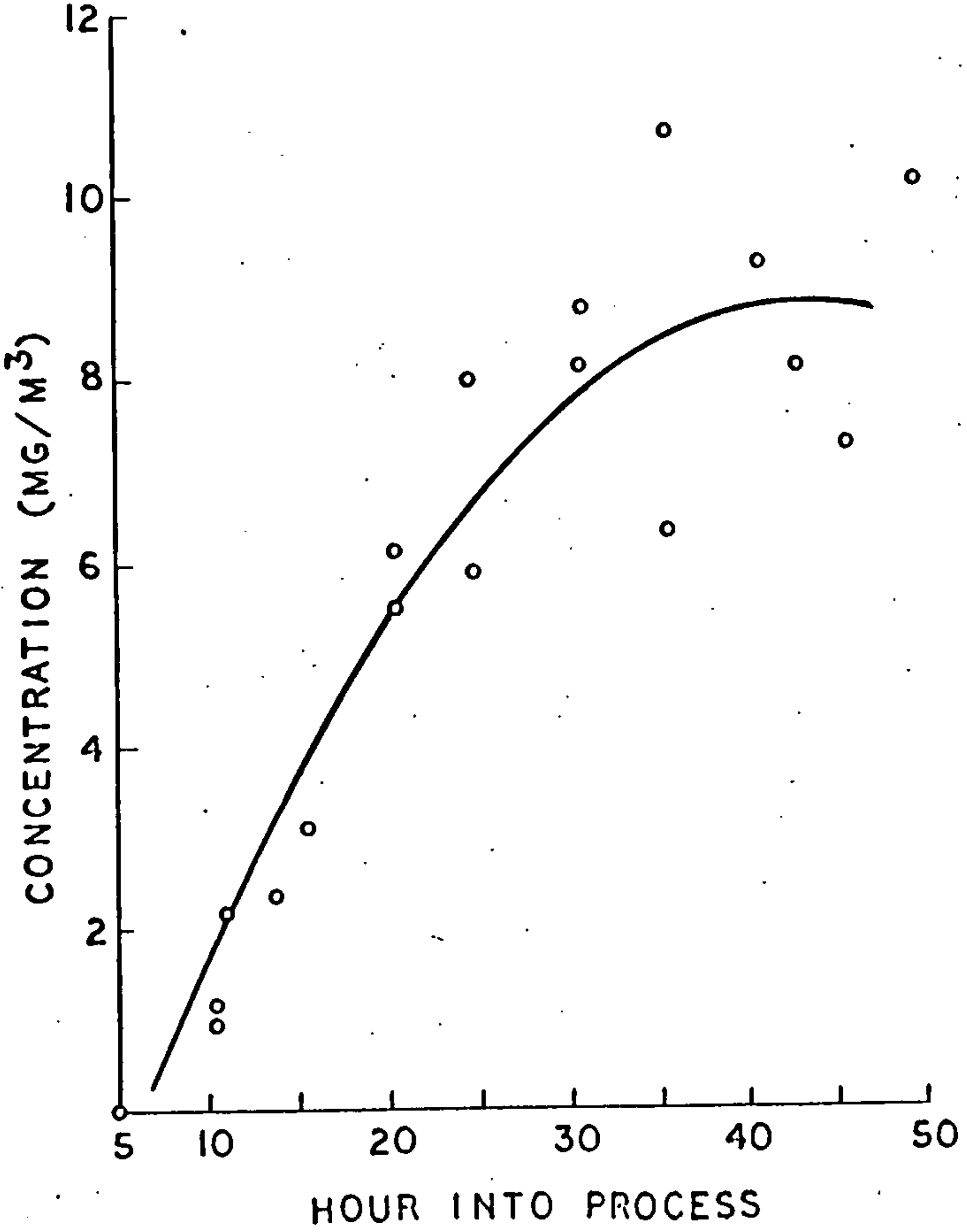

FIGURE 6. ISOAMYL ALCOHOL

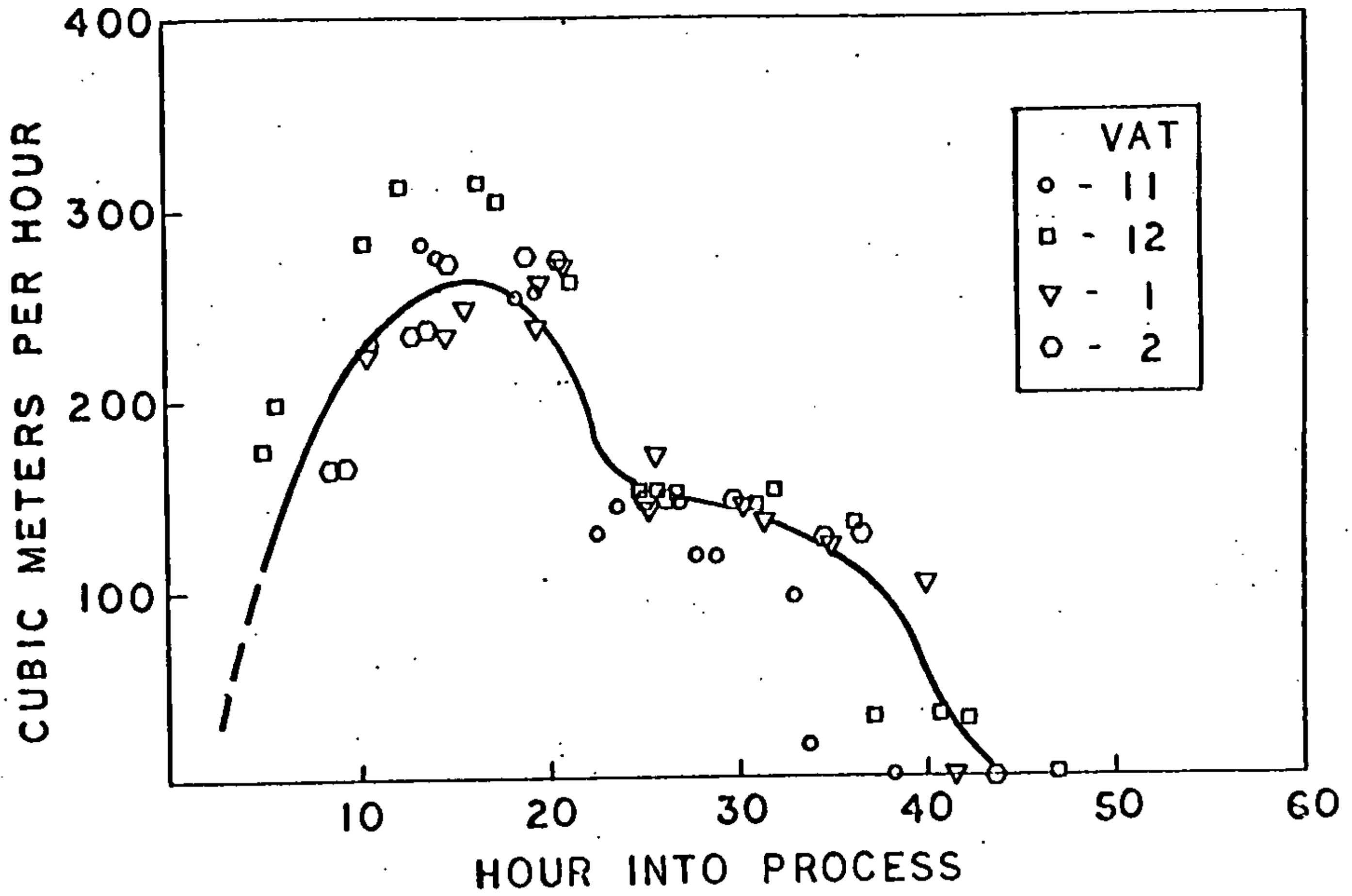

FIGURE 7. MEASURED EXIT GAS (COMPOSITE)

APPENDIX B

EXCERPTS FROM REFERENCE 2

(EPA, 1978)

EPA-450/2-78-013

April 1978

COST AND ENGINEERING STUDY - CONTROL OF VOLATILE ORGANIC EMISSIONS FROM WHISKEY WAREHOUSING

U.S. ENVIRONMENTAL PROTECTION AGENCY
Office of Air and Waste Management
Office of Air Quality Planning and Standards
Research Triangle Park, North Carolina 27711

The second emission, evaporation after barrel emptying, occurs when the saturated barrels are stored after emptying. The amount and location of these emissions depend on the use that the distillers find for the barrels. A significant fraction are stored outside for lengthy periods during which much of the alcohol evaporates. Even if further use is found for the barrels, the bound alcohol will still evaporate if the barrels are stored long enough before reuse. Potential end uses for used barrels are aging Scotch, Canadian whiskies and American light whiskies, and as fuel or for decorative purposes. Federal law prohibits the use of used barrels in bourbon and American blended whiskey.

3.2 WHISKEY WAREHOUSING EMISSION FACTORS

Two sources of data are available to develop emissions factors for whiskey warehousing - aggregate loss data from IRS publications and individual loss data from specific distillers.

3.2.1 Emission Factors from IRS Data

The aggregate loss data from IRS publications are presented in Table 3-1.[1,2] Shown on this table are data on whiskey withdrawals, losses and stocks for 1974, 1975, and 1976, along with emission factors calculated from this data. Withdrawals represent whiskey removed from storage for consumption. Losses represent the difference between the original and withdrawn amounts, i.e. that amount of whiskey lost due to evaporation and barrel soakage, plus theft, spills, etc. Average stocks represent an average of the amount of whiskey held in storage for that year and the previous five.

Three emission factors were developed from this data. Emission Factor I represents the fraction of whiskey production lost and equals .2 proof gallons lost for each proof gallon whiskey produced. This factor was computed by dividing total losses by total production (losses plus withdrawals). Emission Factor II represents the loss rate based on stored whiskey and equals .038 proof gallons lost for each proof gallon in storage each year. This factor was computed by dividing total losses by average stocks. The number of proof gallons in stock was taken to be the average of the number of proof gallons in stock for that year and the previous five. The 6-year average stock was used since losses recorded for a given year represent losses on barrels emptied that year. These losses actually occurred not only during that year, but in previous years while the barrel was in storage. Six years is an approximation of the period of barrel storage - some of the losses for a given year come from barrels stored eight years and more, whereas some stored six years ago have already been emptied for four year old whiskey. Emission Factor III represents a weight loss rate per barrel per year and equals 3.2 kg ethanol/per barrel each year. This factor was computed by multiplying Emission Factor II by 55 proof gallons per barrel and 1.5 kg ethanol per proof gallon. It is important to note that the above figures include losses for both evaporation during storage and soaking into the barrel.

3.2.2 Emission Factors from Individual Distiller Data

The loss rate data from individual distillers and from experiments cover two areas, barrel soakage losses and evaporation losses during storage. These are discussed below.

The data available on barrel soakage losses are presented in Table 3-2.[3,4,5, The table shows the available data on total liquid soakage vs. aging time, plus a best fit equation for this data. The table indicates a rapid saturation of the barrel during the first year, followed by a constant, but slow, increase in weight during subsequent years. It should be noted that the data are for liquid soakage, i.e., both water and ethanol. Work by Boruff and Rittschof[7] indic that the proof of the liquid in the barrel wood is approximately the same as subsequent years. This is in agreement with the theory discussed early. This variation in the incremental loss rate means that the age mix of the barrels in storage will affect the emission rate. Since barrels of different age have different evaporative loss rates, the total emissions will be determined by the fraction of barrels at each age.

Three different barrel age distributions were used to calculated emission factors: (1) the age distribution of bonded whiskey in Kentucky at the end of 1975;[14] (2) an age distribution based on fluctuating market from year to year; and (3) the age distribution based on distillers producing mainly four year old whiskey. Table 3-5 presents the barrel age distribution for the three cases and the respective emission factors of 2.55 kg/barrel-yr for case one, 2.74 kg/barrel-yr for case two, and 2.89 kg/barrel-yr for case three. These emission factors were calculated by multiplying the fraction of the barrels at a given age by the incremental loss for that age in Table 3-5. The four distille producing primarily four and six year old whiskey used in case three are Jim Beam, Clermont, Kentucky; Jim Beam, Beam, Kentucky; Brown-Foreman, Louisville, Kentucky; and Fleischmann, Owensboro, Kentucky.[15]

The above emission factors represent evaporative losses during storage only. To determine overall emission factors, losses due to barrel soakage must be included. This loss is computed by assuming that the number of barrels emptied in a year equals the number of barrels one year old, and that the average barrel has a soakage equivalent to a five year old barrel. This figure is 4.2 kg ethanol barrel. The overall emission factor is therefore:

Aging + Soakage = Total Emissions

case one) 2.55 + 4.2 (.112) = 3.02
case two) 2.74 + 4.2 (.172) = 3.46 kg/barrel-yr
case three) 2.89 + 4.2 (.181) = 3.65

In the preceding discussion, the variations in evaporative loss rate during aging were averaged together to develop a single emission factor.

Table 3-6. SUMMARY OF EMISSION FACTORS
WHISKEY WAREHOUSING

Source	Figure	Description
IRS Publication	.20 proof gallons lost/proof gallons produced*	represents fraction of production lost
	.038 proof gallons lost/proof gallons storage-yr*	represents fraction of storage lost per year
	3.2 kg ethanol/barrel-yr*	represents amount of ethanol lost per barrel in storage per year
Individual Distiller Data & Experiments	3.8 kg ethanol soakage/barrel (4 years	represents amount of ethanol lost per barrel due to soakage into wood. The figure is for a barrel stored 4 years.
	3.02,3.46,3.65 kg ethanol/barrel-year	represents amount of ethanol lost due to both evaporation during storage and soakage for various barrel age distributions
	2.3,3.6 kg ethanol/barrel-yr	represents the range of ethanol loss during storage caused by differing methods of warehouse operation; does not include soakage loss

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*These figures include all types of loss - evaporation during storage, soakage into the barrel, plus leakage, theft,etc.

APPENDIX C

LETTER FROM COMMONWEALTH OF KENTUCKY
(REFERENCE 3)

(Hornbeck, 1992)

PHILLIP J. SHEPHERD
SECRETARY

BRERETON C. JONES
GOVERNOR

COMMONWEALTH OF KENTUCKY
NATURAL RESOURCES AND ENVIRONMENTAL PROTECTION CABINET
DEPARTMENT FOR ENVIRONMENTAL PROTECTION
DIVISION FOR AIR QUALITY
316 St. Clair Mall
Frankfort, Kentucky 40601

September 18, 1992

Mr. H. Edward O'Daniel, Jr., President
Kentucky Distillers Association
110 W. Main Street
Springfield, Kentucky 40069

Ed
Dear Mr. ~~O'~~Daniel:

The Kentucky Division for Air Quality, after reviewing its Emissions Inventory System (EIS) database, has made the following changes regarding distilleries:

(1) The Division has determined that some distillery operations were not required to have an operating permit and thus were exempted from paying an emissions fee *(Please see Table A enclosed)*. If the exempted distilleries had been subject to the emissions fee the cost per ton of pollutant would have been $9.44 instead of $9.51 per ton. However, due to a lower overall emission tonnage caused by the no permit exemptions, a cost savings estimated at $15,353.87 was incurred by the distilling industry.

(2) After consulting with the U.S. Environmental Protection Agency (EPA), the Division changed the emission factor for the whiskey aging process in its EIS database from 10 to 7.6 lbs. of VOC emitted per barrel stored *(Please see Tables B and C enclosed)*. If the old emission factor had been utilized then the cost per ton would have been $9.39. However, due to the lower overall emission tonnage caused by the emission factor change, a cost savings estimated at $24,561.17 was incurred by the distilling industry.

Additionally, enclosed is a copy of an U.S. EPA publication regarding VOC emissions from whiskey warehouses that you requested. If you have questions regarding any of the information provided, please contact me or Mr. Martin Luther, of my staff, at (502) 564-3382.

Sincerely,

John E. Hornback

John E. Hornback, Director

JEH/mrl

Enclosures

TABLE A.

PERMIT FEES GENERATED USING 1991 KYEIS ACTUAL EMISSIONS DATA
SOURCES 25 TONS OR AGGREGATE 25 TONS OF EMISSIONS OR GREATER
POLLUTANTS SUBJECT TO THE FEE ARE NO2, PM10, SO2, AND VOC
DISTILLERIES NOT CHARGED - NO PERMIT REQUIRED

OBS	CONAME	AQCR	COUNTYID	PLANTID	SO2 TONS	NO2 TONS	PM10 TONS	VOC TONS	TOTAL TONS
1	HEAVEN HILL DISTILLERIES	104	3000	0034	0.000	0.000	0.000	577.600	577.600
2	BOULEVARD DISTILLERS	102	1980	0002	0.000	0.000	0.000	332.728	332.728
3	BOULEVARD DISTILLERS	102	0060	0007	0.000	0.000	0.000	255.873	255.873
4	MAKER'S MARK DISTILLERY	104	2580	0003	0.000	0.000	0.932	227.487	228.419
5	GLENMORE DISTILLERIES CO	077	0920	0009	0.000	0.000	0.000	209.228	209.228
6	JAMES B BEAM DISTILLNG CO	102	4140	0010	0.000	0.000	0.000	113.240	113.240
					-----	-----	-----	--------	--------
					0.000	0.000	0.932	1716.156	1717.088

TABLE B.

PERMIT FEES GENERATED USING 1991 KYEIS ACTUAL EMISSIONS DATA
SOURCES 25 TONS OR AGGREGATE 25 TONS OF EMISSIONS OR GREATER
POLLUTANTS SUBJECT TO THE FEE ARE NO2, PM10, SO2, AND VOC
BILLED DISTILLERIES TONNAGE WITH OLD EMISSION FACTOR

OBS	CONAME	AQCR	COUNTYID	PLANTID	SO2 TONS	NO2 TONS	PM10 TONS	VOC TONS	TOTAL TONS
1	HEAVEN HILL DISTILLERIES	104	3000	0005	0.011	1.980	0.550	2500.049	2502.590
2	JIM BEAM BRANDS CO	104	3000	0014	180.142	125.328	9.188	1993.103	2307.761
3	JIM BEAM BRANDS CO	104	0440	0005	72.543	38.822	5.327	2137.297	2253.989
4	JOSEPH SEAGRAM & SONS INC	104	0440	0004	0.108	0.050	0.003	1944.956	1945.117
5	AGE INTERNATIONAL, INC.	102	1320	0009	247.495	125.680	20.644	1303.778	1697.597
6	BARTON BRANDS LTD	104	3000	0020	134.452	66.006	10.284	1174.271	1385.013
7	BOULEVARD DISTILLERS	102	0060	0003	88.986	27.689	4.899	780.414	901.988
8	JAMES B BEAM DISTILLNG CO	102	1320	0003	34.774	15.703	1.352	733.083	784.912
9	MEDLEY DISTILLING CO	077	0920	0042	363.339	47.257	3.197	216.887	630.680
10	JAMES B BEAM DISTILL CO	102	4140	0009	0.000	0.000	0.000	582.500	582.500
					1121.850	448.515	55.444	13366.338	14992.147

TABLE C.

PERMIT FEES GENERATED USING 1991 KYEIS ACTUAL EMISSIONS DATA
SOURCES 25 TONS OR AGGREGATE 25 TONS OF EMISSIONS OR GREATER
POLLUTANTS SUBJECT TO THE FEE ARE NO2, PM10, SO2, AND VOC
DISTILLERIES CHARGED FOR FISCAL YEAR 1992-1993 $9.51 PER TON
THE NEW EMISSION FACTOR WAS USED TO OBTAIN THE $9.51 CHARGE

16:49 FRIDAY, SEPTEMBER 11, 1992

OBS	CONAME	AQCR	COUNTYID	PLANTID	SO2 TONS	NO2 TONS	PM10 TONS	VOC TONS	TOTAL TONS	TOTAL COST
1	JIM BEAM BRANDS CO	104	3000	0014	180.142	125.320	9.188	1947.844	2262.502	21516.40
2	HEAVEN HILL DISTILLERIES	104	3000	0005	0.011	1.980	0.550	1900.049	1902.590	18093.63
3	JIM BEAM BRANDS CO	104	0440	0005	72.543	38.822	5.327	1626.191	1742.883	16574.82
4	JOSEPH SEAGRAM & SONS INC	104	0440	0004	0.108	0.050	0.003	1478.167	1478.328	14058.91
5	AGR INTERNATIONAL, INC.	102	1320	0009	247.495	125.680	20.644	991.001	1384.820	13169.64
6	BARTON BRANDS LTD	104	3000	0020	134.452	66.006	10.284	892.526	1103.268	10492.08
7	BOULEVARD DISTILLERS	102	0060	0003	88.986	27.689	4.899	593.961	715.535	6804.74
8	JAMES B BEAM DISTILLNG CO	102	1320	0003	34.774	15.703	1.352	557.163	608.992	5791.52
9	MEDLEY DISTILLING CO	077	0920	0042	363.339	47.257	3.197	164.891	578.684	5503.27
10	JAMES B BEAM DISTILL CO	102	4140	0009	0.000	0.000	0.000	442.700	442.700	4210.08
					========	=======	======	=========	=========	=========
					1121.850	448.515	55.444	10594.493	12220.302	116215.09

APPENDIX D

EXCERPTS FROM REFERENCE 4

(EPA, 1995)

United States
Environmental Protection
Agency

Research and Development

Risk Reduction
Engineering Laboratory
Cincinnati, OH 45268

EPA/600/S-95/002 April 1995

ENVIRONMENTAL RESEARCH BRIEF

Waste Minimization Assessment for a Bourbon Distillery

Marvin Fleischman*, Michael Parris*,
Eric W. Daley**, and Gwen P. Looby

Abstract

The U.S. Environmental Protection Agency (EPA) has funded a pilot project to assist small and medium-size manufacturers who want to minimize their generation of waste but who lack the expertise to do so. Waste Minimization Assessment Centers (WMACs) were established at selected universities and procedures were adapted from the EPA *Waste Minimization Opportunity Assessment Manual* (EPA/625/7-88/003, July 1988). That document has been superseded by the *Facility Pollution Prevention Guide* (EPA/600/R-92/088, May 1992). The WMAC team at the University of Louisville performed an assessment at a plant that manufactures bourbon whiskey and distiller dried grains as a byproduct from corn, rye, and malt. The grains are milled, mixed together, and cooked. Then the resulting mixture is allowed to ferment. After fermentation, the mixture is processed in a distillation column. The distillate is diluted to proper proof and placed in charred barrels for aging. After an appropriate storage period, the barrels are emptied and the contents are shipped in tank trailers. The team's report, detailing findings and recommendations, indicated that large quantities of CO_2 and ethanol are vented from the plant and that significant cost savings could be achieved through CO_2 and ethanol recovery.

This Research Brief was developed by the principal investigators and EPA's Risk Reduction Engineering Laboratory, Cincinnati, OH, to announce key findings of an ongoing research project that is fully documented in a separate report of the same title available from University City Science Center.

Introduction

The amount of waste generated by industrial plants has become an increasingly costly problem for manufacturers and an additional stress on the environment. One solution to the problem of waste generation is to reduce or eliminate the waste at its source.

University City Science Center (Philadelphia, PA) has begun a pilot project to assist small and medium-size manufacturers who want to minimize their generation of waste but who lack the in-house expertise to do so. Under agreement with EPA's Risk Reduction Engineering Laboratory, the Science Center has established three WMACs. This assessment was done by engineering faculty and students at the University of Louisville's WMAC. The assessment teams have considerable direct experience with process operations in manufacturing plants and also have the knowledge and skills needed to minimize waste generation.

The waste minimization assessments are done for small and medium-size manufacturers at no out-of-pocket cost to the client. To qualify for the assessment, each client must fall within Standard Industrial Classification Code 20-39, have gross annual sales not exceeding $75 million, employ no more than 500 persons, and lack in-house expertise in waste minimization.

The potential benefits of the pilot project include minimization of the amount of waste generated by manufacturers and reduction of waste treatment and disposal costs for participating plants. In addition, the project provides valuable experience for graduate and undergraduate students who participate in the program, and a cleaner environment without more regulations and higher costs for manufacturers.

---

* University of Louisville, Department of Chemical Engineering

** University City Science Center, Philadelphia, PA

Methodology of Assessments

The waste minimization assessments require several site visits to each client served. In general, the WMACs follow the procedures outlined in the EPA *Waste Minimization Opportunity Assessment Manual* (EPA/625/7-88/003, July 1988). The WMAC staff locate the sources of waste in the plant and identify the current disposal or treatment methods and their associated costs. They then identify and analyze a variety of ways to reduce or eliminate the waste. Specific measures to achieve that goal are recommended and the essential supporting technological and economic information is developed. Finally, a confidential report that details the WMAC's findings and recommendations (including cost savings, implementation costs, and payback times) is prepared for each client.

Plant Background

The plant produces bourbon whiskey and distiller dried grains from corn, rye, and malt. It operates three shifts/day to produce approximately 5 million gal of bourbon and over 16,000 tons of distiller dried grain/yr.

Manufacturing Process

The basic raw materials—corn, rye, and malt—are milled in hammer mills and fed to cookers. Water and setback (thin stillage from the drying of spent grain after the alcohol and large solids have been removed) are added and the resulting mixture is cooked. During cooking, the starch in the corn and rye is converted to sugar. After the conversion has taken place, the mixture (known as mash) is pumped to a fermenter where yeast is added to complete the conversion of sugar to alcohol. Upon completion of the fermentation cycle, the mash (or beer) is pumped to an intermediate tank called the beer well.

The contents of the beer well are pumped to the distillation column where the alcohol is steam stripped from the beer. The steam stripper distillate is condensed and pumped to the doubler for final distillation. Distillate from the doubler is condensed and pumped to the barrel-filling operations; spent grain is pumped to the dry house for processing.

At the barrel-filling facility, the distillate is diluted to proper proof with demineralized water. Barrels (of charred new white oak) are filled with the diluted distillate and transported to the warehouse for aging.

During the storage period (a minimum of four years), the material in the barrel goes through a maturation or aging process by which the distillate is transformed into a bourbon. When the product in the barrel is determined to be of proper quality, the barrel is transported to the dumping area. There the contents of the barrel are poured through steel screens for removal of solids. The product is then pumped to one of two storage tanks from which it is loaded into tank trailers for shipment.

Spent grain from the distillation operations is processed into distiller dried grain (an animal feed additive) in the dry house. The spent grain is processed in centrifuges where the solids are concentrated and the excess water (centrate) is removed. The concentrated solids are fed to an air dryer and the centrate is pumped to an evaporator where the dissolved solids are concentrated. The viscous discharge (syrup) from the evaporator is mixed with a portion of the dried grain stream as it is recycled back to the dryer. The portion of the dried grain stream not used as recycle is conveyed to one of two storage silos if the moisture content is satisfactory.

An abbreviated process flow diagram for this plant is shown in Figure 1.

Waste Management Opportunities

The type of waste currently generated by the plant, the source of the waste, the waste management method, the quantity of the waste, and the annual waste management cost for each waste stream identified are given in Table 1.

Table 2 shows the opportunities for waste minimization that the WMAC team recommended for the plant. The minimization opportunity, the type of waste, the possible waste reduction and associated savings, and the implementation cost along with the payback time are given in the table. The quantities of waste currently generated by the plant and possible waste reduction depend on the production level of the plant. All values should be considered in that context.

It should be noted that the economic savings of the minimization opportunity, in most cases, results from the need for less raw material and from reduced present and future costs associated with waste treatment and disposal. Other savings not quantifiable by this study include a wide variety of possible future costs related to changing emissions standards, liability, and employee health. It also should be noted that the savings given for each opportunity reflect the savings achievable when implementing each waste minimization opportunity independently and do not reflect duplication of savings that would result when the opportunities are implemented in a package.

Additional Recommendations

In addition to the opportunities recommended and analyzed by the WMAC team, several additional measures were considered. These measures were not completely analyzed because of insufficient data, minimal savings, implementation difficulty, or a projected lengthy payback. Since one or more of these approaches to waste reduction may, however, increase in attractiveness with changing conditions in the plant, they were brought to the plant's attention for future consideration.

- Seal the grain leaks found throughout the conveying operations.
- Install a shutoff mechanism that provides a tighter seal at the discharge of the milled grain hoppers in order to reduce grain losses.
- Control ethanol emissions from storage tanks.

This research brief summarizes a part of the work done under Cooperative Agreement No. CR-814903 by the University City Science Center under the sponsorship of the U.S. Environmental Protection Agency. The EPA Project Officer was **Emma Lou George**.

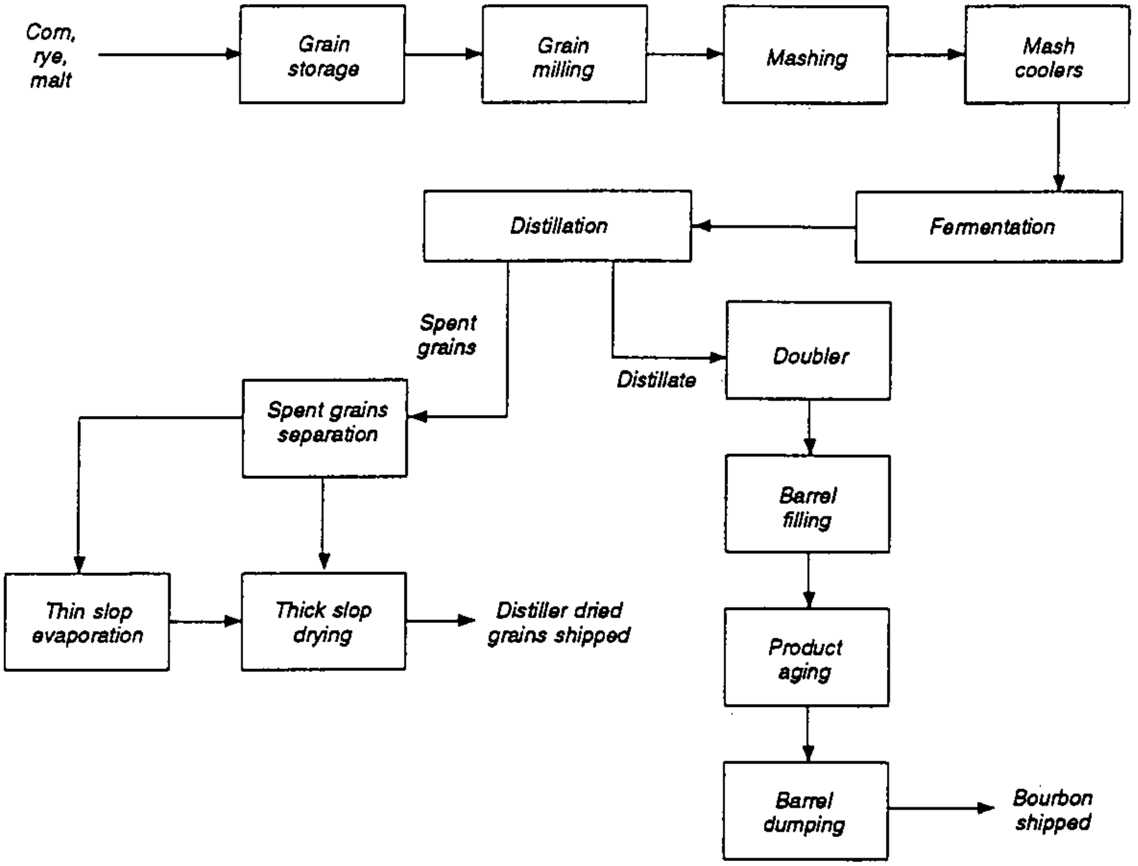

Figure 1. Abbreviated process flow diagram for bourbon whiskey production.

Table 1. Summary of Current Waste Generation

Waste Generated	Source of Waste	Waste Management Method	Annual Quantity Generated (lb/yr)	Annual Waste Management Cost[1]
Grain losses	Raw material handling	Shipped offsite to landfill	24,000	$6,000
Carbon dioxide	Fermentation	Vented from plant	25,680,000	0
— Ethanol	Fermentation	Vented from plant	205,920	43,680
Wastewater	Fermentation tank cleaning	Treated in onsite WWTP; discharged to creek	846,600	—[2]
— Ethanol	Distillation	Vented from plant	228,100	48,000
Spent caustic	Cleaning of distillation equipment	Treated in onsite WWTP; discharged to creek	830,000	—[2]
—Spilled ethanol	Barrel filling	Evaporates	1,040	660
Char	Charring of new barrels	Shipped offsite to landfill	480 ft^3/yr	40
— Ethanol	Warehoused barrels	Vented from plant	3,181,080	1,020,540
Char/bungs	Emptying of barrels and filtering of contents	Mixed with coal; incinerated in onsite boiler	60,000	0
Filter pads	Filtering of barrel contents	Shipped offsite to landfill	3,200 pads/yr	40
Scrubber solids	Dry house air scrubber	Shipped offsite to landfill	3,000	1,090
Scrubber water	Dry house air scrubber	Treated in onsite WWTP; discharged to creek	149,400	—[2]
Spent caustic	Dry house cleaning	Treated in onsite WWTP; discharged to creek	415,000	—[2]
Slop	Dry house	Sold to farmers	640,000 gal/yr	-7,000 (revenue received)
Distiller dried grain losses	Truck loading, equipment leakage	Shipped offsite to landfill	200,000	17,000
Wastewater	Boiler feedwater treatment	Treated in onsite WWTP; discharged to creek	4,191,500	—[2]
Wastewater	Boiler blowdown	Treated in onsite WWTP; discharged to creek	24,000,000	—[2]
Spent liquids	Demineralizer regeneration	Treated in onsite WWTP; discharged to creek	664,000	—[2]
Ashes	Coal-fired boiler	Shipped offsite to landfill	129,600 ft^3/yr	17,600
— Ethanol	Storage tanks	Vented from plant	11,780	5,230
Spent oils	Equipment lubrication	Shipped offsite	275 gal/yr	125
Spent mineral spirits	Parts cleaning	Shipped offsite for recycle	235 gal/yr	1,460
Cloth filters	Dust collectors	Shipped offsite to be cleaned; returned for reuse	110 filters/yr	380

[1] Includes waste treatment, disposal, and handling costs and applicable lost raw materials value.

[2] Cost is included in the annual WWTP operating cost of $118,300/yr.

Table 2. Summary of Recommended Waste Minimization Opportunities

Minimization Opportunity	Waste Reduced	Annual Waste Reduction		Net Annual Savings	Implementation Cost	Simple Payback (yr)
		Quanity (lb/yr)	Per cent			
Install air ducting from the storage hopper vents to the inlet of an existing pulsating dust collector in order to minimize grain losses from the vent lines.	Grain losses	4,800	20	$1,200	$3,000	2.5
Modify the metal funnel apparatus used during grain unloading from the railcars in order to reduce grain losses.	Grain losses	1,200	5	300	500	1.7
Install a recovery plant to recover CO_2 and ethanol from the fermenters. The recovered liquefied CO_2 can be sold to a distributor or an end-user as a food and beverage grade product. Recovered ethanol can be sold to a fuel alcohol facility or used as supplemental fuel onsite.	Carbon dioxide (from fermentation)	20,160,000	79	223,600[1]	930,000	4.2[2]
	Ethanol (from fermentation)	161,700	79			
Install a refrigerated water-cooled condenser on the vent discharges from the distillation process in order to recover ethanol. Use the recovered ethanol onsite as a supplemental fuel.	Ethanol (from distillation)	228,100	100	19,800	39,000	2.0[2]
Recover ethanol emissions from the barrel warehouse using activated carbon adsorption. Redistill the recovered ethanol and sell the product obtained.	Ethanol (from warehousing)	2,900,000	90	495,380	2,050,000	4.1[2]
Replace the loading apparatus currently used for loading the distiller dried grains with a telescoping loading nozzle in order to reduce grain losses.	Distiller dried grain losses	2,000,000	100	17,000	45,000	2.7

[1] Exact savings may vary from this estimate depending on the exact price obtained for the CO_2.

[2] A much shorter payback will be realized should a carbon emissions tax and/or a VOC emissions tax or permit for ethanol be required in the future.

University of Louisville

Waste Minimization Assessment Center

Report No. 17-9

3624 Market Street
Philadelphia, PA 19104
(215) 387-2255

APPENDIX E

LOSSES DURING WHISKY AGING--SEAGRAMS (REFERENCE 5)

(Garcia, 1997)

(502) 244-2404
FAX (502) 244-2416

Seagram Americas
ENGINEERING SERVICES
BARKLEY BUILDING • SUITE 200 • 12700 SHELBYVILLE ROAD • LOUISVILLE, KY 40243-1599

FACSIMILE TRANSMISSION

DATE: ______ FAX MESSAGE NO.: ______

NO. OF PAGES (INCLUDING COVER SHEET:) 2

TO: DR. T. LAPP
MRI

FROM: R. J. GARCIA
SEAGRAM

TRANSMITTING FROM: (502) 244-2416

HELP/TALK-TO NO.: (502) 244-2418

PLEASE CONFIRM RECEIPT BY RETURN TRANSMISSION OF THIS SHEET.

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COMMENTS:

RTG'S VERSUS AGE
FOR 1993 STANDARDS

Age	105 Proof New Char RTG'S	110 Proof Corn/Hi Con RTG'S	140 Proof Lt. Whiskey RTG'S
0	55.886	56.131	69.543
1.5	50.505	52.602	65.145
2.0	49.605	51.613	63.911
2.5	48.706	50.524	62.676
3.0	47.806	49.634	61.442
3.5	46.907	48.645	60.208
4.0	46.007	47.656	58.973
4.5	45.108	46.667	57.739
5.0	44.208	45.677	56.504
5.5	43.309	44.688	55.270
6.0	42.409	43.699	54.036
6.5	41.518	42.709	52.801
7.0	40.610	41.720	51.507
7.5	39.711	40.731	50.332
8.0	38.811	39.741	49.098
8.5	37.912	38.752	47.864
9.0	37.012	37.763	46.629
9.5	36.113	36.783	45.395
10.0	35.213	35.784	44.161
10.5	34.314	34.795	42.926
11.0	33.414	33.805	41.592
11.5	32.515	32.816	40.457
12.0	31.615	31.827	39.223
12.5	30.716	30.837	37.989

	Soakage OPG's/BBL
N Char	2.683
Ch/Hicon	0.561
Lt Wky	0.695

Evaporation Loss %	
N. Char	3.219%
Ch/Hicon	3.525%
Lt Wky	3.550%

SEAGRAMS

I. TOTAL LOSSES (Evaporation and soakage)

Data in proof gallons (pg); one proof gallon contains 50 percent ethanol by volume.

	Bourbon (new char)		Corn		Light whisky	
Aging period, yr	Total loss, pg/bbl	Loss, pg/bbl/yr	Total loss, pg/bl	Loss, pg/bbl/yr	Total loss, pg/bbl	Loss, pg/bbl/yr
4.0	9.879	2.470	8.475	2.119	10.570	2.643
4.5	10.778	2.395	9.464	2.103	11.804	2.623
5.0	11.678	2.336	10.454	2.091	13.039	2.608
5.5	12.577	2.287	11.443	2.081	14.273	2.595
6.0	13.477	2.246	12.432	2.072	15.507	2.585
6.5	14.368	2.210	13.422	2.065	16.742	2.576
7.0	15.276	2.182	14.411	2.059	18.036	2.577
7.5	16.175	2.157	15.400	2.053	19.211	2.561
8.0	17.075	2.134	16.390	2.049	20.445	2.556
8.5	17.974	2.115	17.379	2.045	21.679	2.550
9.0	18.874	2.097	18.368	2.041	22.914	2.546
9.5	19.773	2.081	19.348	2.037	24.148	2.542
10.0	20.673	2.067	20.347	2.035	25.382	2.538
10.5	21.572	2.054	21.336	2.032	26.617	2.535

Bourbon: $\frac{30.831}{14} = 2.202$ pg/bbl/yr $\qquad \frac{2.202}{2} \times 6.6097 = 7.28$ lb/bbl/yr

Corn: $\frac{28.882}{14} = 2.063$ pg/bbl/yr $\qquad \frac{2.063}{2} \times 6.6097 = 6.82$ lb/bbl/yr

Light whisky: $\frac{36.035}{14} = 2.574$ pg/bbl/yr $\qquad \frac{2.574}{2} \times 6.6097 = 8.51$ lb/bbl/yr

Average = 7.28 + 6.82 + 8.51 = 7.5 lb/bbl/yr

Note: One gallon of 100 percent ethanol weighs 6.6097 pounds

II. Soakage

Assumes that soakage occurs within the first 18 months after the initial filling of the barrel and that the rate of evaporation during the aging period of years 0 to 1.5 are the same as during the years of 1.5 to 3.0.

Bourbon (new char):

Year 0 =	55.886 pg/bbl	Year 1.5 =	50.505
Year 1.5 =	50.505	Year 3.0 =	47.806
	5.381 pg/bbl lost		2.699 pg/bbl lost

5.381 - 2.699 = 2.682 pg/bbl lost due to soakage

Corn whisky:

Year 0 =	56.131	Year 1.5 =	52.602
Year 15 =	52.602	Year 3.0 =	49.634
	3.529 pg/bbl lost		2.968 pg/bbl lost

3.529 - 2.968 = 0.561 pg/bbl lost due to soakage

Light whisky:

Year 0 -	69.543	Year 1.5 =	65.145
Year 1.5 =	65.145	Year 3.0 =	61.442
	4.398 pg/bbl lost		3.703 pg/bl lost

4.398 - 3.703 = 0.695 pg/bbl lost due to soakage

III. <u>Evaporation Losses</u> (Total losses - soakage)

Soakage:	bourbon =	2.682 pg/bbl
	corn =	0.561 pg/bbl
	light whisky =	0.695 pg/bbl

	Bourbon			Corn			Light whisky		
Aging period, yr	Total loss, pg/bbl	Minus soakage, pg/bbl	Loss, pg/bbl/yr	Total loss, pg/bbl	Minus soakage, pg/bbl	Loss, pg/bbl/yr	Total loss, pg/bbl	Minus soakage, pg/bbl	Loss, pg/bbl/yr
4.0	9.879	7.197	1.799	8.475	7.914	1.979	10.570	9.875	2.469
4.5	10.778	8.096	1.799	9.464	8.903	1.978	11.804	11.109	2.469
5.0	11.678	8.996	1.799	10.454	9.893	1.979	13.039	12.344	2.469
5.5	12.577	9.895	1.799	11.443	10.882	1.979	14.273	13.578	2.469
6.0	13.477	10.795	1.799	12.432	11.871	1.979	15.507	14.812	2.469
6.5	14.368	11.686	1.798	13.422	12.861	1.979	16.742	16.047	2.469
7.0	15.276	12.594	1.799	14.411	13.850	1.979	18.036	17.341	2.477
7.5	16.175	13.493	1.799	15.400	14.839	1.979	19.211	18.516	2.469
8.0	17.075	14.393	1.799	16.390	15.829	1.979	20.445	19.750	2.469
8.5	17.974	15.292	1.799	17.379	16.818	1.979	21.679	20.984	2.469
9.0	18.874	16.182	1.799	18.368	17.807	1.979	22.914	22.219	2.469
9.5	19.773	17.091	1.799	19.348	18.787	1.978	24.148	23.453	2.469
10.0	20.673	17.991	1.799	20.347	19.786	1.979	25.382	24.687	2.469
10.5	21.572	18.890	1.799	21.336	20.775	1.979	26.617	25.922	2.469

Bourbon: $\frac{1.799}{2} \times 6.6097 = 5.95$ lb/bbl/yr

Corn: $\frac{1.979}{2} \times 6.6097 = 6.54$ lb/bbl/yr

Light whisky: $\frac{2.470}{2} \times 6.6097 = 8.16$ lb/bbl/yr

Average = 5.95 + 6.54 + 8.16 = 6.9 lb/bbl/yr

APPENDIX F

LOSSES DURING WHISKY AGING--JIM BEAM (REFERENCE 6)

(Omlie, 1997)

EMPIRICAL DATA
AND
CALCULATIONS
OF
LOSSES AND EMISSION RATES
DURING WHISKEY AGING PROCESS

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Director Technical Services

August 24, 1992

Table III

EMISSION LOSSES DURING WHISKEY AGING PROCESS

09/03/1992 -- Summary Only
BOURBON WHISKEY

weighted average based on aging months

125° whisky

YRS		(A) Total P.G.Loss	(B) P.G. Entry	(C) Total Barrels	(D) Barrel Months	(E) Total Barrel Yrs.	(F) P.G. Loss/ Bar. Yr.	(G) %Loss/ Bar.Yr	
4.71	53 ≤ ## < 58 months 56.5	2807.20	12998.70	193	18.83	908.26	3.0907	4.5890%	Total for Period
5.15	58 ≤ ## < 63 months 61.8	538799.40	2288654.90	34179	514.92	176077.81	3.0600	4.5698%	Total for Period
5.38	63 ≤ ## < 68 months 64.6	812916.00	3296827.50	48926	792.70	263200.57	3.0886	4.5836%	Total for Period
5.72	68 ≤ ## < 73 months 68.6	12831.50	52603.90	789	11.43	4509.28	2.8456	4.2680%	Total for Period
6.43	73 ≤ ## < 78 months 77.1	130806.40	444919.60	6565	96.30	42205.19	3.0993	4.5732%	Total for Period
6.77	78 ≤ ## < 83 months 81.2	602239.00	2100996.10	32013	466.52	216525.36	2.7814	4.2380%	Total for Period
6.98	83 ≤ ## < 88 months 83.7	114216.20	370911.60	5417	97.73	37792.48	3.0222	4.4138%	Total for Period
7.94	93 ≤ ## < 98 months 95.3	63.20	260.00	4	15.88	31.75	1.9903	3.0621%	Total for Period
8.50	98 ≤ ## < 103 months 102.0	14531.20	43485.40	625	50.55	5312.62	2.7352	3.9312%	Total for Period
9.42	103 ≤ ## < 108 months 105.8	4708.50	13320.00	200	8.82	1763.34	2.6702	4.0093%	Total for Period
9.65	113 ≤ ## < 118 months 115.8	41145.80	108486.00	1722	58.04	16615.38	2.4764	3.9307%	Total for Period
10.07	118 ≤ ## < 123 months 120.8	46886.30	119252.00	1894	110.70	19063.43	2.4595	3.9062%	Total for Period
10.52	## ≥ 123 months 126.2	61691.30	154397.20	2471	299.74	25977.67	2.3748	3.8006%	Total for Period
		2,383,642.00	9,007,112.90	134,998	2,542.15	809,983.14	2.9428	4.4107%	Summary Total

DO NOT USE

INCORRECT

Table IV

EMISSION LOSSES DURING WHISKEY AGING PROCESS

09/03/1992 -- Summary Only
CORN

125' whisky

	(A) Total P.G.Loss	(B) P.G. Entry	(C) Total Barrels	(D) Barrel Months	(E) Total Barrel Yrs.	(F) P.G. Loss/ Bar. Yr.	(G) %Loss/ Bar.Yr	
① ## < 48 months 47.1	1155.90	7524.00	114	3.92	447.26	2.5844	3.9158%	Total for Period
② 48 ≤ ## < 53 months 50.8	2785.20	19308.20	293	8.42	1240.48	2.2453	3.4071%	Total for Period
③ 58 ≤ ## < 63 months 60.2	15132.40	91731.80	1392	55.10	6987.75	2.1656	3.2862%	Total for Period
④ 63 ≤ ## < 68 months 64.4	14446.00	73195.90	1111	48.28	5958.69	2.4244	3.6798%	Total for Period
⑤ 98 ≤ ## < 103 months 101.3	5712.00	21771.50	339	25.33	2862.76	1.9953	3.1068%	Total for Period
	39,231.50	213,531.40	3,249	141.05	17,496.95	2.2422	3.4116%	Summary Total

↑ incorrect average
should be 2.283

JIM BEAM BRANDS

1. The aging periods in Tables III and IV represent a weighted average number of months based on the number of barrels emptied and the actual aging period for each barrel. Not all barrels were aged for the same length of time.

2. Soakage occurs primarily in new char barrels but may occur to a much lesser extent in used barrels. Soakage is a one-time event that occurs in new char barrels during the initial 6 to 18 months of aging. After that time, an equilibrium is established between the wood and the distilled spirit such that little additional soakage occurs. A much lesser amount of soakage occurs in used barrels because the majority of the soakage occurred during the initial use of the barrel.

3. The difference in proof gallon (pg) loss between new and used barrels over the approximately same aging period using whiskies of about the same proof may provide an indication of the loss due to soakage. For the data sets in Tables III and IV, both the bourbon and corn whisky were 125 proof. The data in Tables III and IV cannot be used to estimate soakage unless it is assumed that the evaporation rate for bourbon and corn whisky are the same over the same aging period.

4. To estimate soakage loss, selected data were used from Tables III and IV to establish an aging period of approximately the same duration. For Bourbon whisky in Table III, the span in aging is 4.71 years through 10.52 years. The data for aging periods from 8.82 years through 10.52 years were deleted, which results in an aging period of 4.71 years through 8.50 years. In Table IV, pg loss data are available for corn whisky during a span in aging of 3.93 years through 8.44 years. If the first data set in Table IV is deleted, the aging period becomes 4.23 years through 8.44 years. This is comparable to the aging span for Bourbon whisky in Table III.

I. SOAKAGE

Bourbon: Aging period = 4.7 - 8.5 years
Data from Table III
Data for 95.3 months (7.94 years) of aging were considered to be an outlier and were not used in this calculation.

Aging			
Months	Years	Total loss, pg	Total barrels
56.5	4.71	2,807.2	193
61.8	5.15	538,799.4	34,179
64.6	5.38	812,916.0	48,926
68.6	5.72	12,831.5	789
77.1	6.43	130,806.4	6,565
81.2	6.77	602,239.0	32,013
83.7	6.98	114,216.2	5,417
102.0	8.50	14,531.2	625
TOTAL		2,229,146.9	128,707

Average loss = 17.320 pg/bbl

Corn: Aging period = 4.2 - 8.4 years
Data from Table IV

Aging		Total loss, pg	Total barrels
Months	Years		
50.8	4.23	2,785.2	293
60.2	5.02	15,132.4	1,392
64.4	5.37	14,446.0	1,111
101.3	8.44	5,712.0	339
TOTAL		38,075.6	3,135

Average loss = 12.145 pg/bbl

Estimated soakage:
New barrels (bourbon) = 17.320 pg/bbl
Used barrels (corn) = 12.145 pg/bbl
Soakage 5.175 pg/bbl

II. TOTAL LOSSES (Evaporation plus soakage)

1. Corn whisky -- Corn whisky is aged in used barrels so the primary losses are due to evaporation although some soakage losses probably occur. Data are from Table IV.

Aging		Total loss, pg	Total barrels	Loss, pg/bbl-yr
Months	Years			
47.1	3.93	1,155.9	114	2.580
50.8	4.23	2,785.2	293	2.247
60.2	5.02	15,132.4	1,392	2.166
64.4	5.37	14,446.0	1,111	2.421
101.3	8.44	5,712.0	339	1.996

Average loss = 2.282 pg/bbl-yr

$$\frac{2.282}{2} \times 6.6097 = 7.5 \text{ lb/bbl-yr}$$

2. Bourbon whisky -- Bourbon whisky is aged in new barrels so total losses during aging are due to a combination of soakage and evaporation. Data are from Table III. Data for the aging period 95.3 month (7.94 years) were considered to be an outlier and were not used in the calculation.

Aging		Total loss, pg	Total barrels	Barrel loss, pg/bbl	Loss, pg/bbl-yr
Months	Years				
56.5	4.71	2,807.2	193	14.545	3.088
61.8	5.15	538,799.4	34,179	15.764	3.061
64.6	5.38	812,916.0	48,926	16.615	3.088
68.6	5.72	12,831.5	789	16.263	2.843
77.1	6.43	130,806.4	6,565	19.925	3.099
81.2	6.77	602,239.0	32,013	18.812	2.779
83.7	6.98	114,216.2	5,417	21.085	3.021
102.0	8.50	14,531.2	625	23.250	2.735
105.8	8.82	4,708.5	200	23.543	2.669
115.8	9.65	41,145.8	1,722	23.894	2.476
120.8	10.07	46,886.3	1,894	24.755	2.458
126.2	10.52	61,691.3	2,471	24.966	2.373

Average loss = 2.808 pg/bbl-yr

$$\frac{2.808}{2} \times 6.6097 = 9.3 \text{ lb/bbl-yr}$$

III. EVAPORATION LOSSES (Total loss minus soakage)

Bourbon whisky-- Soakage is a one-time loss that occurs during the first 6 to 18 months of aging. To estimate evaporation loss, the estimated soakage (calculated in Section I) of 5.175 pg/bbl is subtracted from the total proof gallon loss per barrel. This new loss per barrel should be representative of the evaporation loss.

Aging						
Months	Years	Total loss, pg	Total barrels	Total loss, pg/bbl	Total loss minus soakage, pg/bbl	Loss, pg/bbl-yr
56.5	4.71	2,807.2	193	14.545	9.370	1.989
61.8	5.15	538,799.4	34,179	15.764	10.589	2.056
64.6	5.38	812,916.0	48,926	16.615	11.440	2.126
68.6	5.72	12,831.5	789	16.263	11.088	1.938
77.1	6.43	130,806.4	6,565	19.925	14.750	2.294
81.2	6.77	602,239.0	32,013	18.812	13.637	2.014
83.7	6.98	114,216.2	5,417	21.085	15.910	2.279
102.0	8.50	14,531.2	625	23.250	18.075	2.126
105.8	8.82	4,708.5	200	23.543	18.368	2.083
115.8	9.65	41,145.8	1,722	23.894	18.719	1.940
120.8	10.07	46,886.3	1,894	24.755	19.580	1.944
126.2	10.52	61,691.3	2,471	24.966	19.791	1.881

Average loss = 2.056 pg/bbl-yr

$$\frac{2.056}{2} \times 6.6097 = 6.8 \text{ lb/bbl-yr}$$