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GASEOUS EMISSIONS FROM WHISKEY FERMENTATION UNITS

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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.

* Civil Engineer.
† Professor of Civil Engineering.
‡ 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.

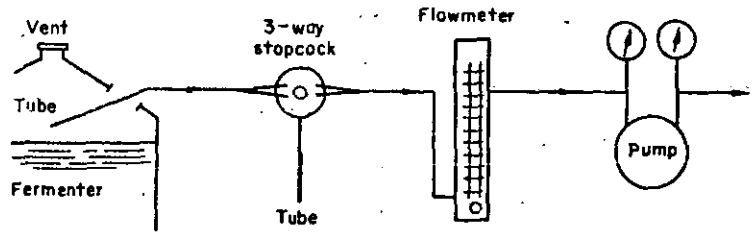

Fig. 1. Sampling apparatus.

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

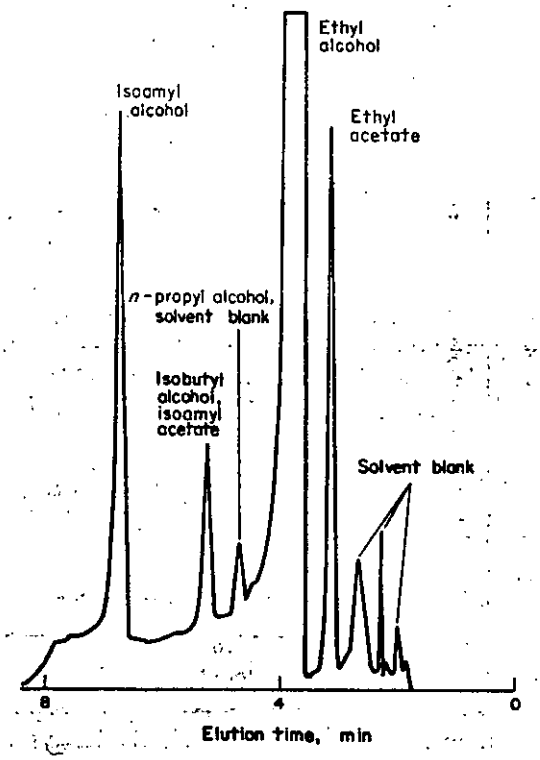

Fig. 2. Chromatogram, charcoal No. 1.

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.

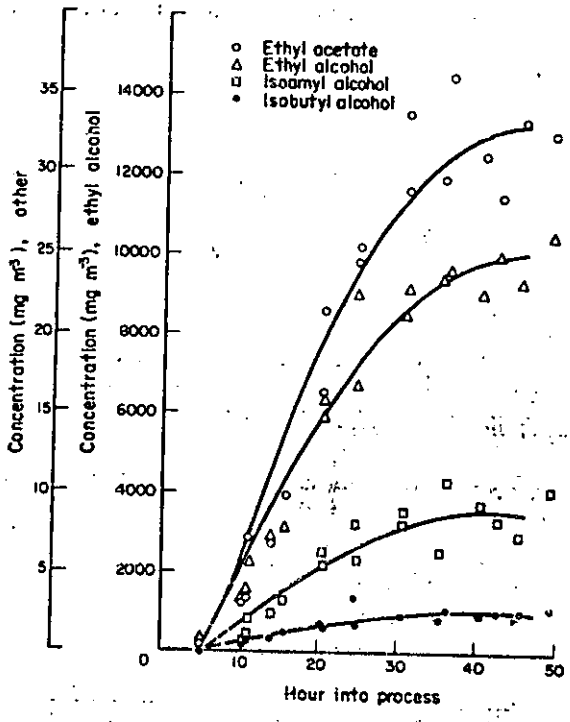

Fig. 3. Organic concentration.

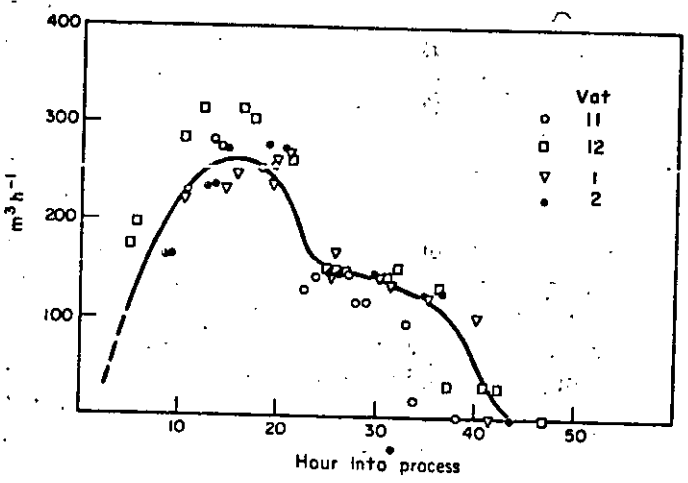

Fig. 4. Measured exit gas (composite).

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

Vat number	11	12	1	2
Volume (m^3)	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*

Compound	Vat number				
	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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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~~

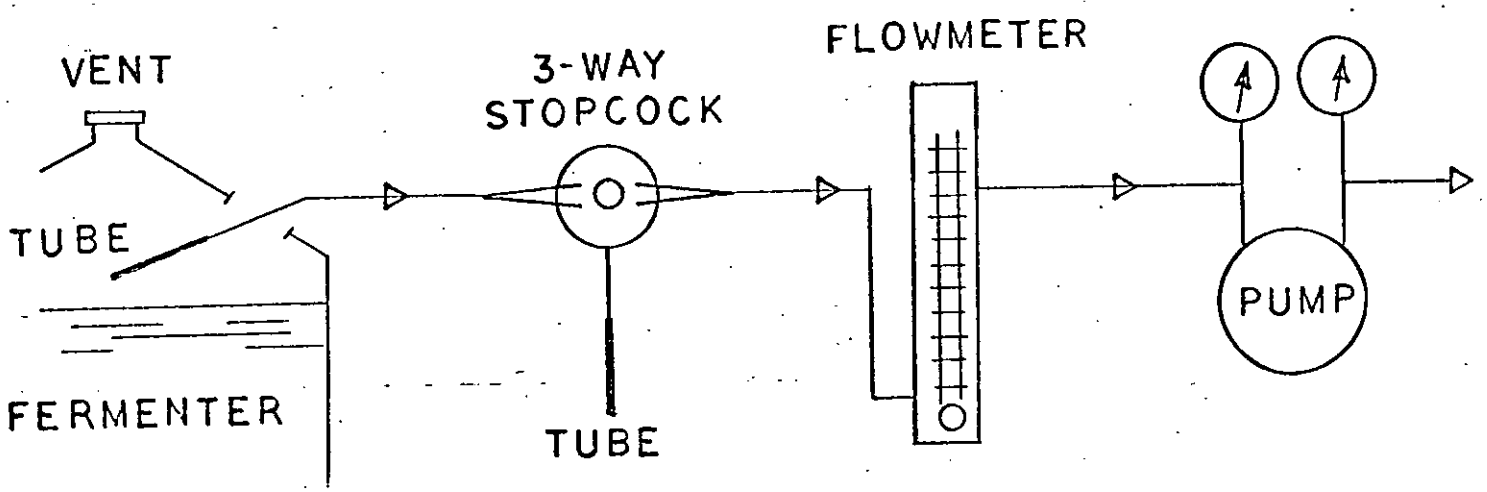

FIGURE 1. SAMPLING APPARATUS

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.

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

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

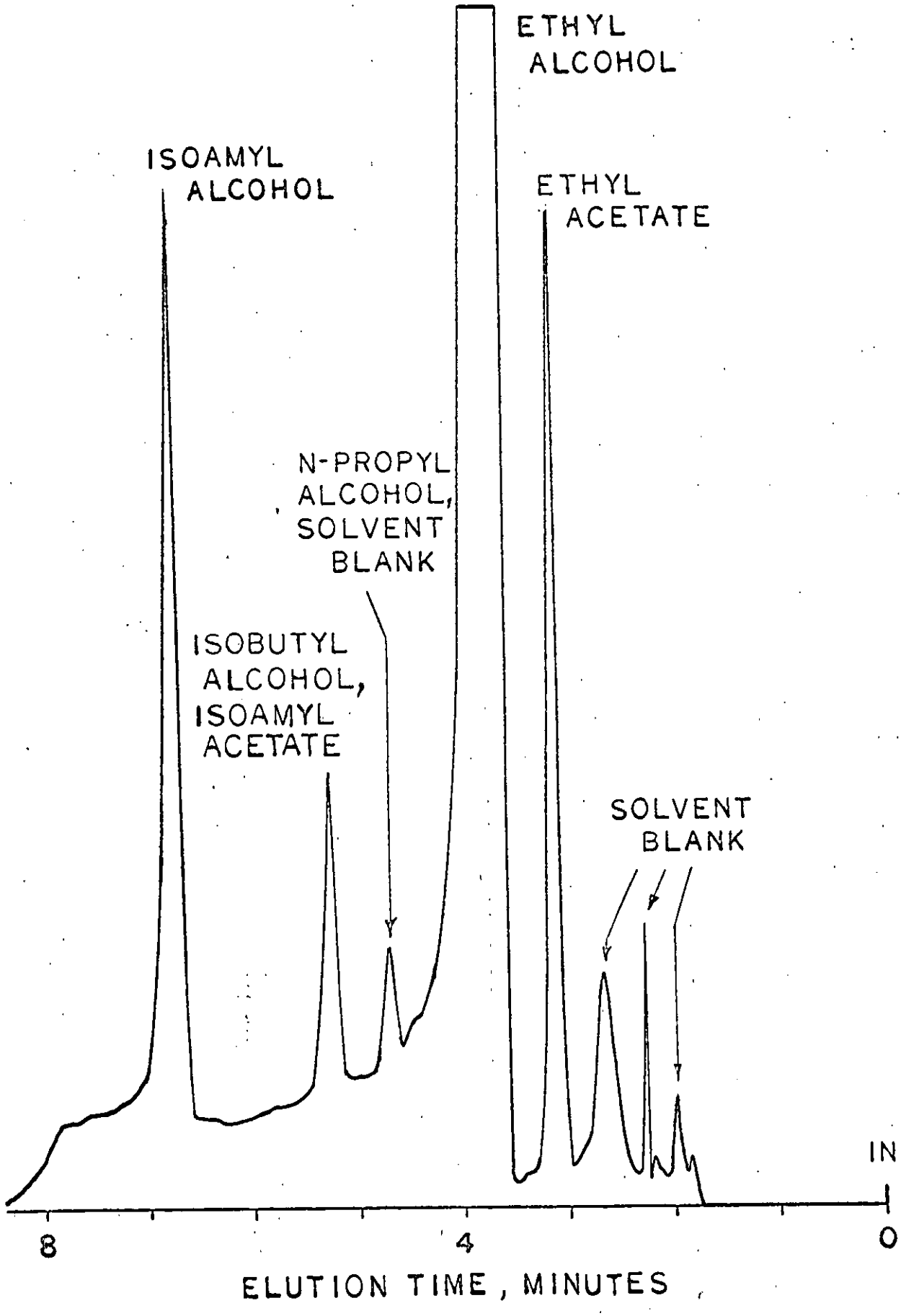

FIGURE 2. CHROMATOGRAM, CHARCOAL SECTION #1

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 *

Compound	Vat Number 11	12	1	2	ave.	
Ethyl Acetate	17.58	20.94	22.11	23.06	20.91	20.92
Ethyl Alcohol	5523.	6396.	6729.	7029.	6419.	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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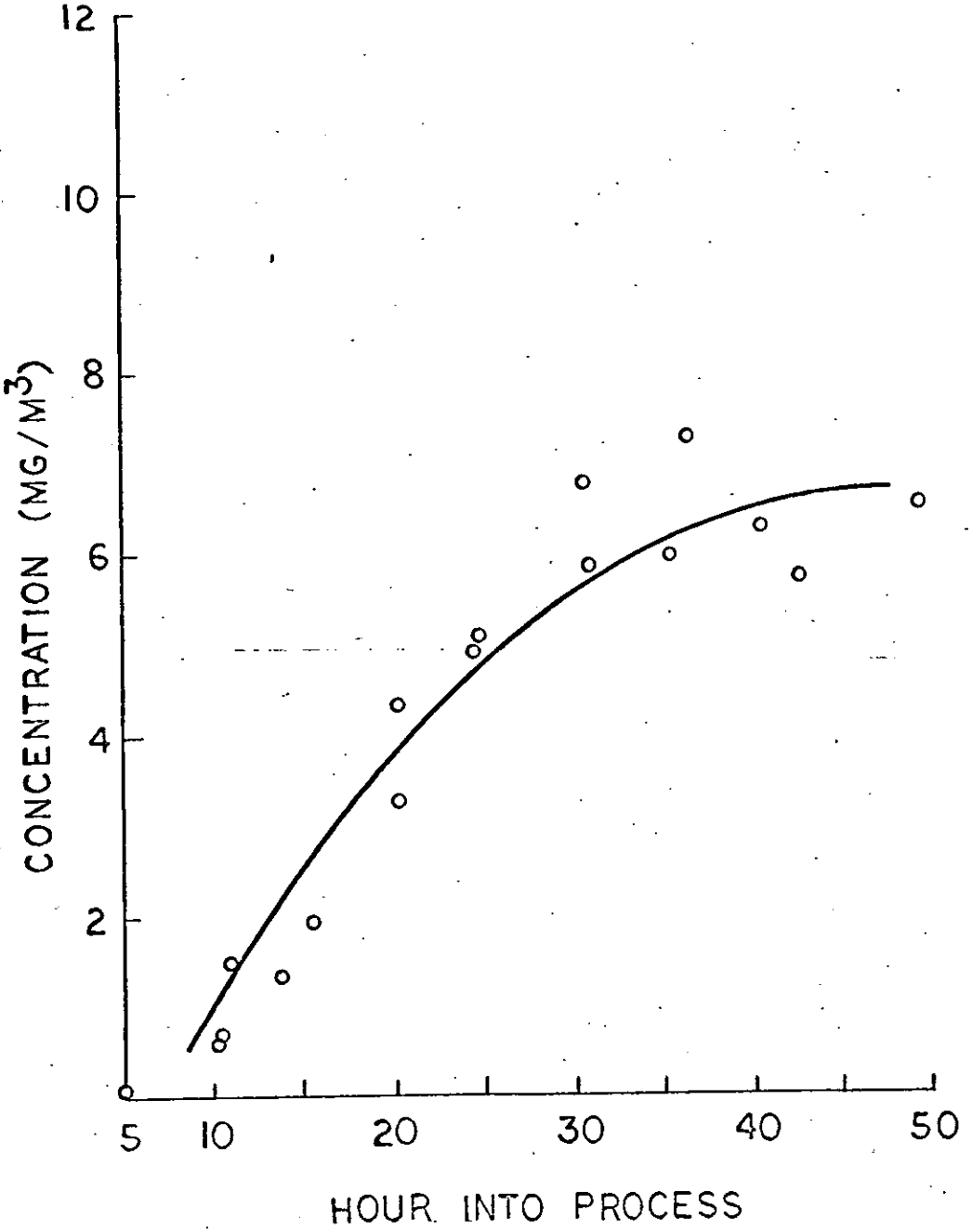

FIGURE 3. ETHYL ACETATE

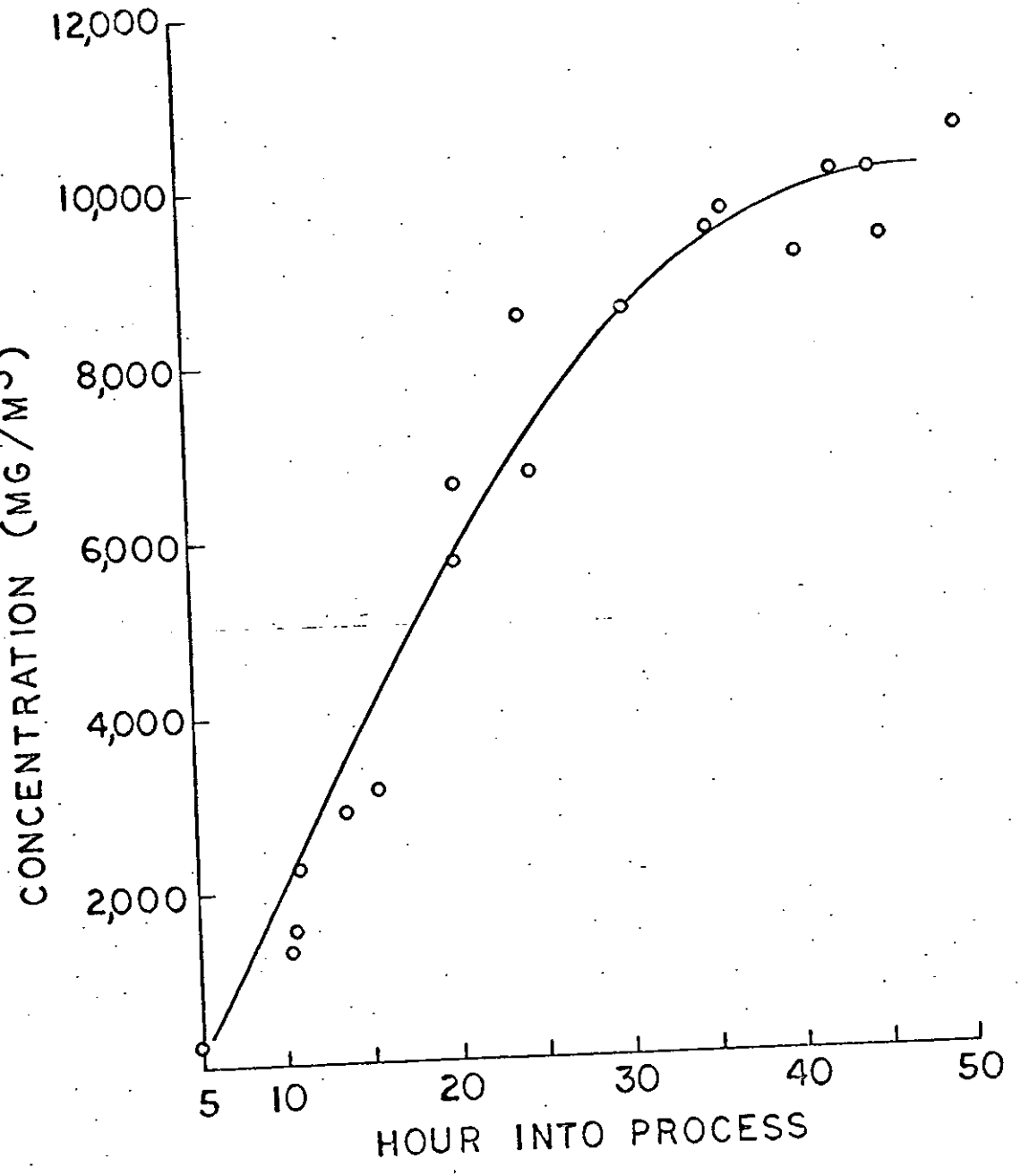

FIGURE 4. ETHYL ALCOHOL

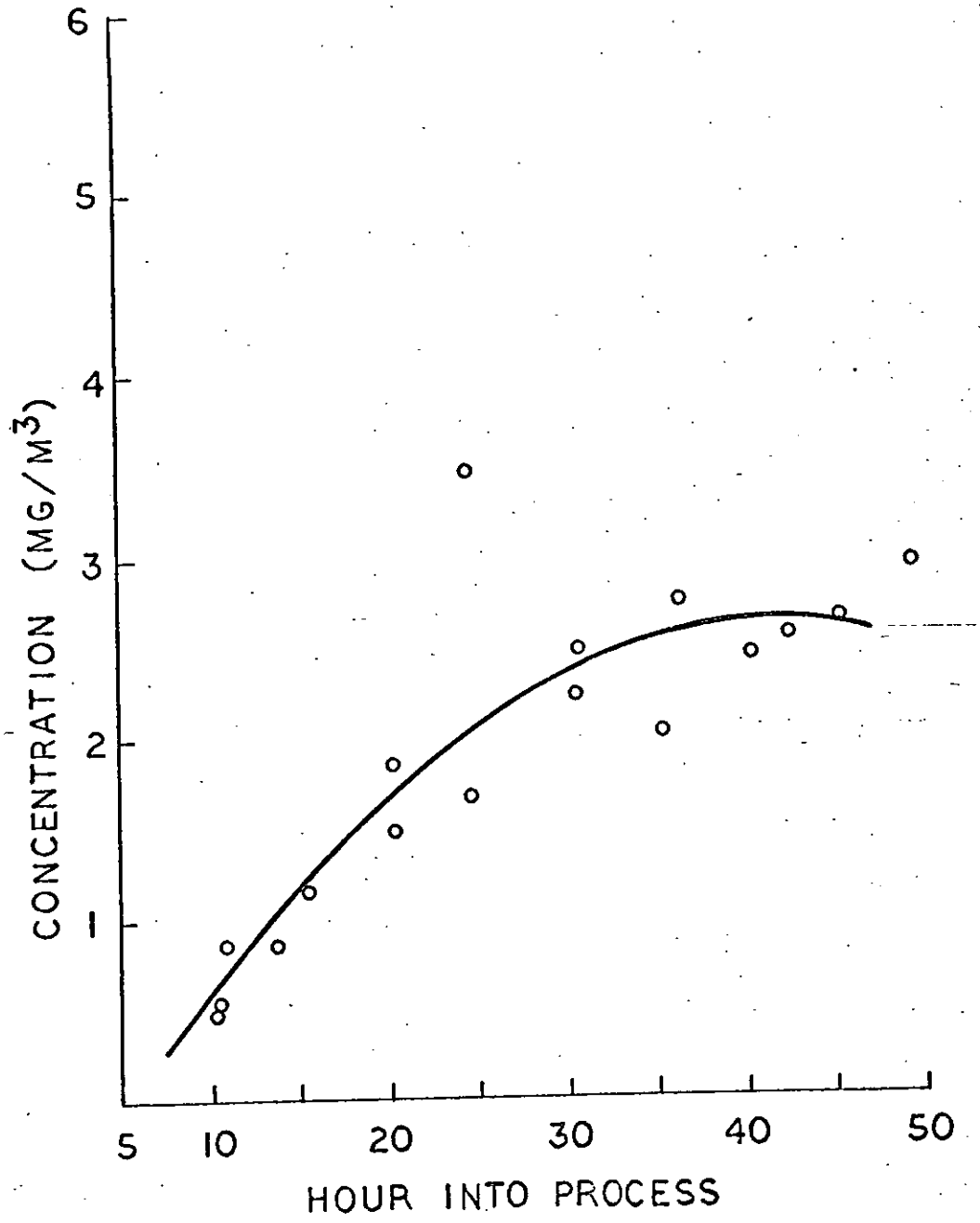

FIGURE 5. ISOBUTYL ALCOHOL

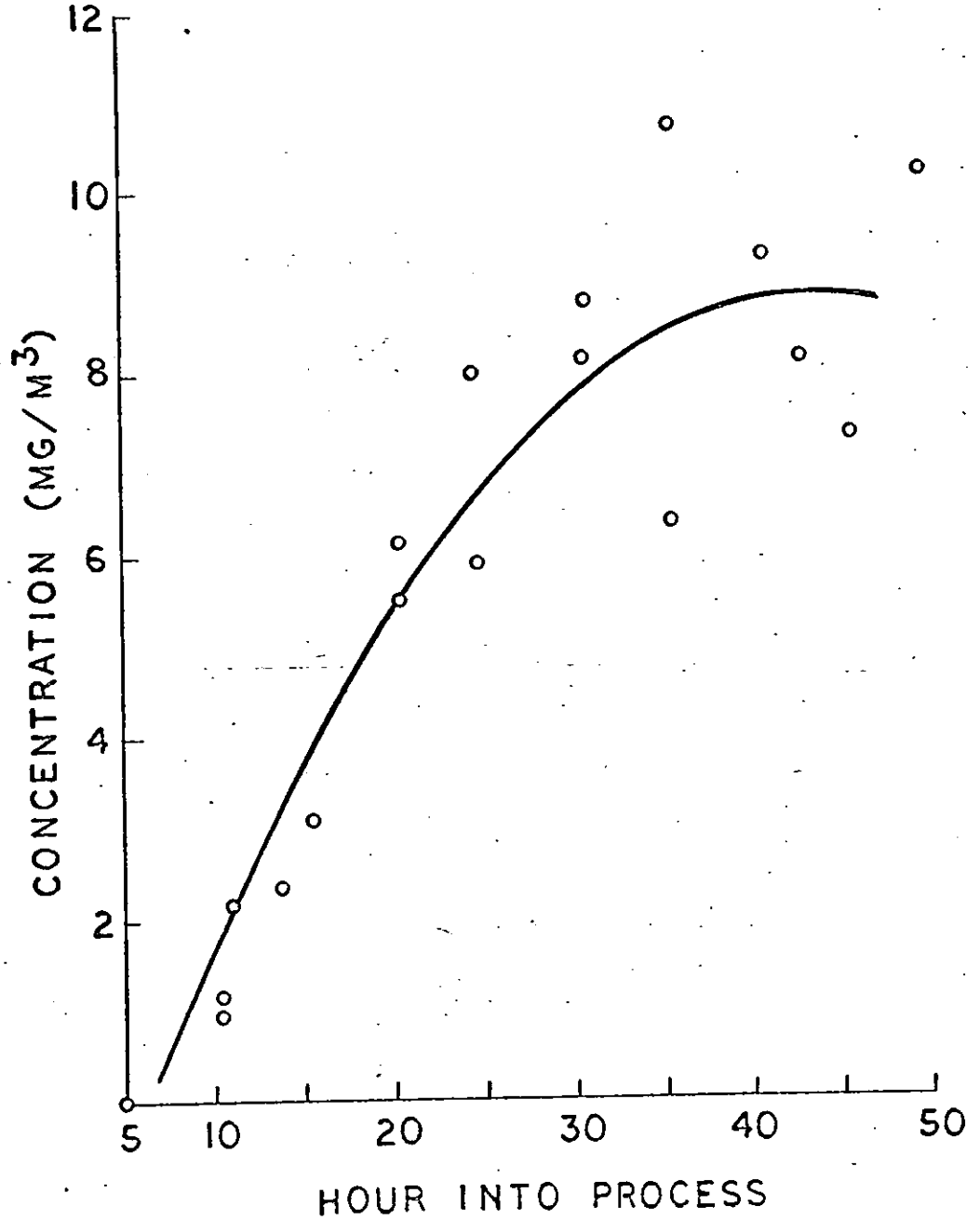

FIGURE 6. ISOAMYL ALCOHOL

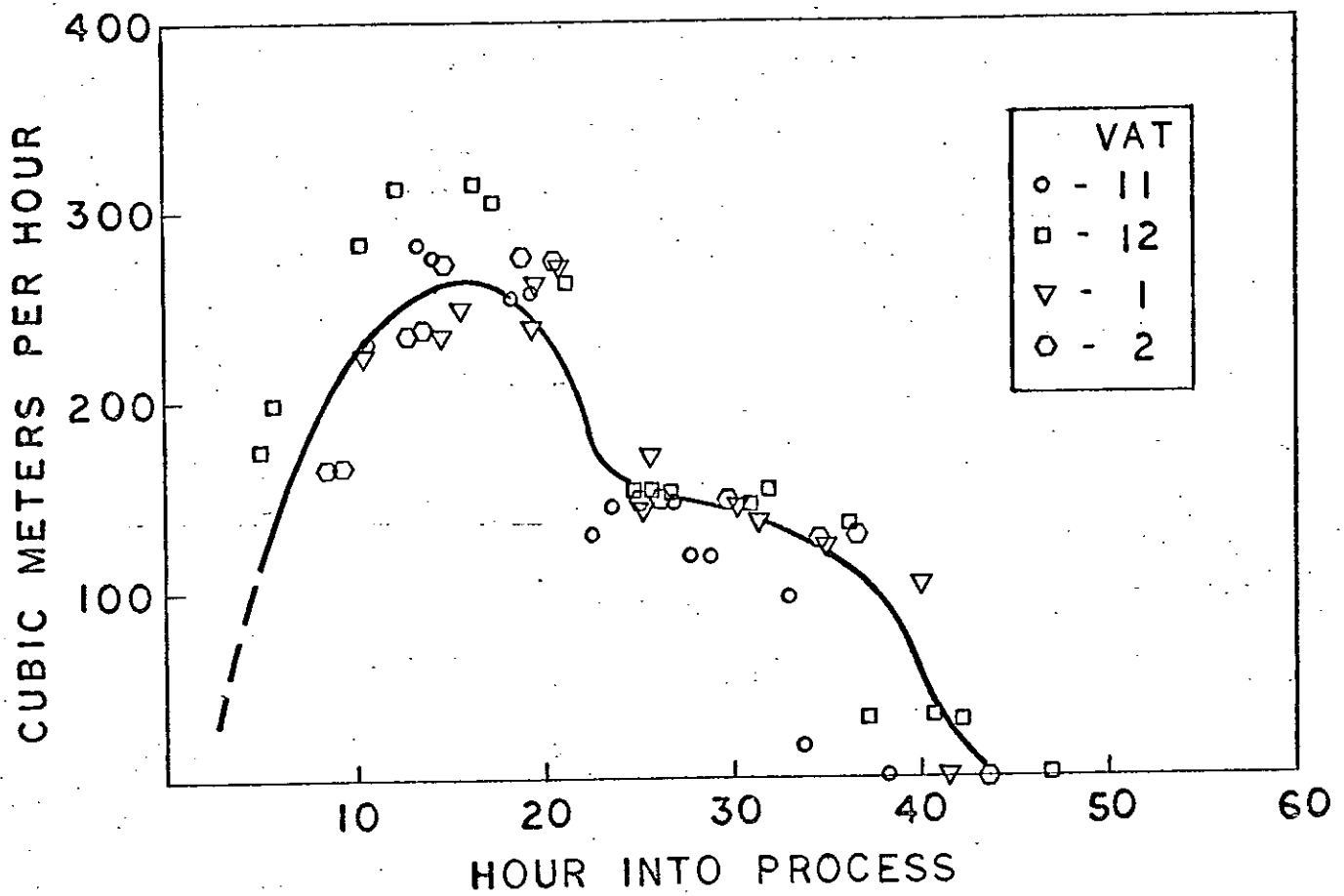

FIGURE 7. MEASURED EXIT GAS (COMPOSITE)