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Hydrocarbon Vapor Standards for Performance Evaluation of Combustible Gas Detectors



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Paul Cascarano, Assistant Director
National Institute of Justice

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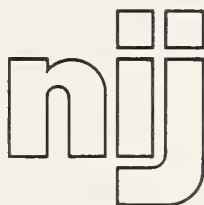
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Hydrocarbon Vapor Standards for Performance Evaluation of Combustible Gas Detectors

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This document is a law enforcement equipment report developed by LESL under the sponsorship of NIJ as part of the Technology Assessment Program, which is described on the inside front cover of this report. Additional reports as well as other documents are being issued under the LESL program in the areas of protective equipment, communications equipment, security systems, weapons, emergency equipment, investigative aids, vehicles, and clothing.

Technical comments and suggestions concerning this report are invited from all interested parties. They may be addressed to the Law Enforcement Standards Laboratory, National Bureau of Standards, Washington, DC 20234.

Lawrence K. Eliason, Chief
Law Enforcement Standards Laboratory

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HYDROCARBON VAPOR STANDARDS FOR PERFORMANCE EVALUATION OF COMBUSTIBLE GAS DETECTORS

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Calibration mixtures of hydrocarbon vapor were prepared for the calibration and performance evaluation of portable combustible gas detectors which are used to detect the presence of accelerant vapors in cases of suspected arson. The standards contained vapors of aliphatic and aromatic compounds common to petroleum distillates such as gasolines, which are frequently employed as accelerants in arson fires. These standards were prepared by a dynamic dilution technique in which the vapor evolving from diffusion tubes containing liquid hydrocarbons was diluted with air.

The concentrations of the vapors were calculated from gravimetrically determined diffusion rates of the hydrocarbons and from measured flow of air at three temperatures: 30°, 50°, and 70 °C. Gas chromatographic analyses of the vapor-air mixtures generated at 30° and 50 °C showed that the vapor concentrations are essentially constant within measurement periods of 1 to 3 d and from one period to another, and that they agreed with calculated concentrations.

Key words: accelerants; arson; combustible gas; diffusion rates; diffusion tubes; detectors; gas chromatography; hydrocarbons; vapor detectors; vapor generator; vapor standards.

1. INTRODUCTION

This is a final report on the research and evaluation of a generator of hydrocarbon vapor standards. A summary is presented of the research results obtained during the period of January 1 through October 30, 1979.

The purpose of the research was to develop standards of low concentrations of organic vapors that could be used by fire investigators to calibrate and evaluate the performance of hydrocarbon vapor detectors, also known as combustible gas detectors or "sniffers." Until this work, there existed no readily available means for calibrating the sensitivity of these detectors.

Numerous jurisdictions have initiated or are anticipating major programs to investigate suspicious fires. These programs will include the use of scientific techniques in the investigations of fire scenes when arson is suspected, and may involve the use of combustible gas detectors. The most common accelerants for initiating incendiary fires are reported to be petroleum distillates, gasoline being the most available and most frequently used [1]¹. Hence, there is need for calibration standards to evaluate the response of the detectors to low concentrations (parts per million, ppm) of hydrocarbon vapors which are common to petroleum distillates.

The sensitivity of most effective vapor detectors is variable. In addition, the sensitivity of some detectors will decrease by an unpredictable factor when the sensing element of the detector becomes contaminated, through continued use, with residues from particulate matter or with vapors of organo-metallic compounds (e.g., tetraethyl lead in leaded gasoline) which may be

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¹ Numbers in brackets indicate literature references in section 6.

encountered in the course of repetitive fire-scene investigations. Knowledge of the vapor detector performance by the investigator is essential, because the primary use of these devices is for the location of accelerant residues in incendiary fire debris, and the location of residual accelerants is a step in providing the physical evidence necessary to establish the crime of arson [2]. Thus, the detector performance must be reliable, and reliability can be assumed through frequent calibrations using readily available standards. In this report a method is described for generating predictable concentrations of hydrocarbon vapors at constant rates for long periods of time. The report presents data on gravimetrically measured rates of diffusion of hydrocarbon vapors through precision bore tubing. In all cases, the vapors evolved from liquids maintained at constant temperature. Gas chromatographic analyses of the vapors at various intervals within a diffusion period are also reported, in order to confirm the constancy of the vapor composition.

2. SELECTED HYDROCARBONS FOR VAPOR STANDARDS

For the preparation of a vapor standard, four relatively pure compounds were selected from which vapors could be generated simultaneously and at constant rates. The selection of the four compounds was based on the following considerations:

- 1) the compounds are constituents of most gasolines,
- 2) the vapor pressures are such that, at convenient temperatures, variable concentrations can be generated which approximate the concentrations that would be found at fire scenes at which accelerants had been employed, and
- 3) the compounds have vapor pressure-temperature relationships such that the vapors are produced at concentrations within the detectable limits of commercial vapor detectors.

It is recognized that gasoline formulations [3,4] contain both aliphatic and aromatic compounds, and that both of these classes of compounds should be included in the vapor standards.

Consequently, four commercially available compounds were selected. Spectrograde isooctane (2,2,4 trimethylpentane) and *n*-hexane (99 mole percent) were chosen to represent, respectively, branched and linear chain-aliphatic hydrocarbons. Scintillation-count-grade toluene and *o*-xylene were chosen to represent the aromatic hydrocarbons. The purity of these compounds was presumed to be that claimed by the manufacturer. The vapor pressures of these compounds are such that they may enable detection by sniffers, as the vapors evolve from the liquids absorbed in a variety of substrates [5]. Similarly, the vapor pressure-temperature relationships of the selected compounds are such [6] that vapor standards of various concentrations can be generated easily from the pure liquids.

3. INSTRUMENTATION

3.1 Standards Generator

A survey of the literature on commercial vapor generators showed that instruments are available which, with slight modifications, could meet the requirements for the generation of dynamic vapor standards for the evaluation of combustible gas detectors. Based on this search, a vapor generator, Analytical Instrument Development Model 350 Standards Generator², was chosen and modified. This involved replacing the diluent (air) flow meter normally provided on the instrument with one that can measure a diluent flow of up to 2 L/min. With this instrument,

² Certain commercial equipment, instruments, or materials are identified in this paper in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Bureau of Standards, nor does it imply that the equipment or material identified is necessarily the best available for the purpose.

vapors are generated from liquids contained in diffusion or permeation tubes, which are also available from the manufacturer of the generator. For this work, the diffusion tube technique was chosen as the most appropriate. A cross-sectional view of a precision bore diffusion tube and its reservoir containing a liquid sample is shown in figure 1. The liquid reservoir has a capacity of about 4 milliliters (mL); tube bores are available with diameters of 0.5, 2, or 5 mm and lengths to approximately 8 cm.

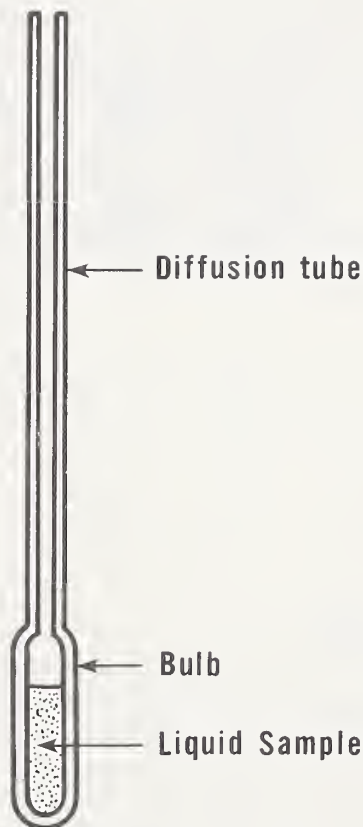
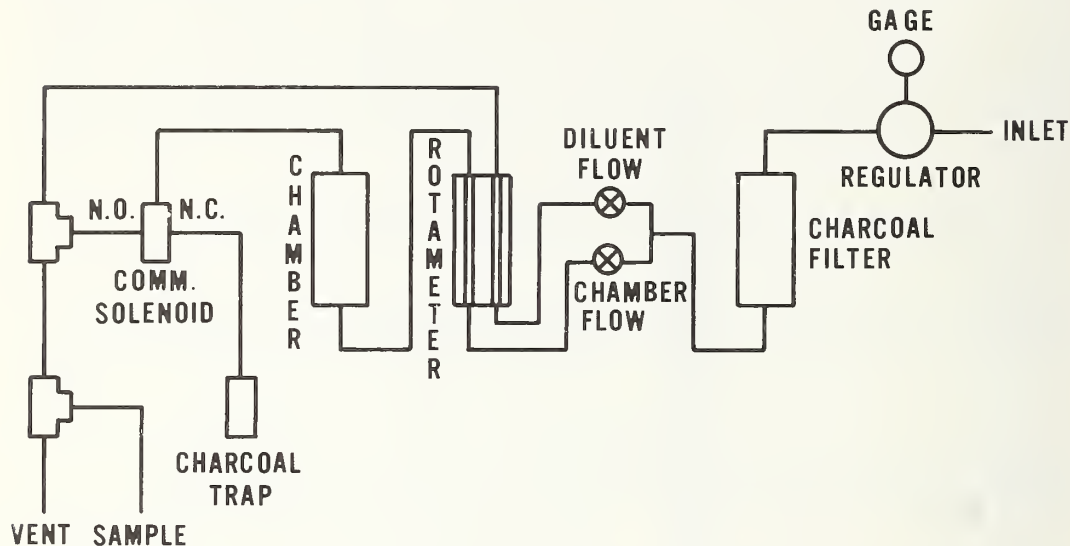


FIGURE 1. *Cross-sectional diagram of a diffusion tube.*

Vapors evolve through the diffusion tube at a constant rate when the liquid is maintained at constant temperature (isothermal) and constant pressure (isobaric) conditions. These conditions are achieved in the sample chamber of the standards generator. As many as four diffusion tubes can be accommodated simultaneously. A diluent gas, for example, air, flowing at a controlled rate is used to dilute the evolving vapor to pre-selected concentrations. A schematic diagram of the flow system is illustrated in figure 2. Air under pressure enters the instrument at the inlet and then passes through a pressure regulator and is adjusted to a lower working pressure. The air passes through a charcoal filter to remove common contaminants, to yield essentially hydrocarbon-free air. Next, the air is split into two streams, one providing diluent and the other flowing through the chamber. Both are regulated by valves and are monitored by rotameters. The chamber air is brought to thermal equilibrium with the chamber by passing it through a heat exchanger before it enters the chamber containing the diffusion tubes.

The air from the chamber passes through a three-way solenoid valve which directs the flow either to the sample and vent ports or to the vapor outlet trap. The vapor outlet trap is used when the generator is operating in a standby mode.

The flow through the chamber is continuously variable from about 50 mL/min to 150 mL/min. The diluent flow is variable from 0 to about 2 L/min. The chamber is operated at a fixed temperature of either 30°, 50°, or 70 °C.



3.2 Gas Chromatograph (GC)

The chromatograph used in this study was a commercial instrument equipped with dual flame-ionization detectors, which were operated at 250 °C. The glass GC column, about 2 m in length and 2 mm i.d., was packed with 3 percent SE30 on a solid support (supelcoport). The column was maintained at 50 °C while the carrier gas, helium, flowed at 30 mL/min. The injection ports were maintained at 150 °C. The GC was interfaced with a recording integrator (printer/plotter) from which the retention times and peak areas of the hydrocarbons were obtained.

4. GENERATION OF SELECTED HYDROCARBON VAPOR STANDARDS

The diffusion rate r , in g/s, of hydrocarbon vapors through a tube has been described [7,8] as shown in eq (1):

$$r = (DMP/RT) (A/L) \ln P/(P-p) \quad (1)$$

where D , M , P , R , and T respectively are the diffusion coefficient (cm^2/s), molecular weight (g/mol), of the compound, total pressure (atmospheres), gas constant ($\text{cm}^3\text{-atm/mol-K}$) and the absolute temperature (K). The partial vapor pressure in atmospheres of the compound at the temperature T is represented by p , and A/L (cm) is the ratio of the area to length of the diffusion tube. Hence, eq (1) may be used to predict two important factors in the preparation of dynamic vapor standards by the diffusion method: (1) the rates and length of time that the hydrocarbon vapors may be generated through diffusion tubes, and (2) the constancy of the diffusion rates. In addition, a theoretical issue remains: Does the observed data fit eq (1)?

4.1 Calibration of Diffusion Tubes

About 2 mL of the hydrocarbon were weighed into a diffusion tube and placed in the chamber of the generator for 1 to 3 d at a selected temperature. During this time, the air flowing through the chamber to remove the evolving vapor was maintained at a rate of 100 mL/min. The diffusion rate of each component was calculated from the gravimetrically measured liquid-weight loss during this period.

Table 1 summarizes the calibration of the diffusion tubes at 30 °C. For each compound, the same diffusion tube was used repetitively for its rate determinations (runs). The dimensions of the diffusion tubes are listed for each compound. Averaged rate values are shown for all measurements, although it appeared that temperature equilibrium was not obtained in runs 1 and 2 until a significant amount of the measured diffusion time had elapsed.

TABLE 1. Diffusion rates of selected hydrocarbons at 30 °C

		Diffusion rate, $\mu\text{g}/\text{min}$			
Dimensions of Tube:					
Inside diameter (mm)		2	2	5	5
Length (mm)		76	62	38	34
Run	Time (min)	<i>n</i> -Hexane	<i>Isooctane</i>	Toluene	<i>o</i> -Xylene
1	1265	14.2	5.4	33.8	8.8
2	1575	15.2	6.2	33.9	9.2
3	1350	16.1	7.1	34.4	9.5
4	4345	16.9	7.2	37.5	9.4
5	4340	16.8	7.0	37.4	10.0
Average \pm standard deviation (Runs 1-5)		15.8 \pm 1.1	6.6 \pm 0.8	35.4 \pm 1.9	9.5 \pm 0.5
Average \pm standard deviation (Runs 3-5)		16.6 \pm 0.4	7.1 \pm 0.1	36.4 \pm 1.8	9.8 \pm 0.3

TABLE 2. Diffusion rates of selected hydrocarbons at 50 °C

		Diffusion rate, $\mu\text{g}/\text{min}$			
Dimensions of tube:					
Inside diameter (mm)		2	0.5	5	5
Length (mm)		76	12	38	34
Run	Time (min)	<i>n</i> -Hexane	<i>Isooctane</i>	Toluene	<i>o</i> -Xylene
1	1360	43.0	6.4	91.4	25.8
2	1585	43.5	6.7	92.2	27.3
3	4325	43.7	6.6	91.7	28.0
Average \pm standard deviation		43.4 \pm 0.4	6.6 \pm 0.1	91.8 \pm 0.4	26.9 \pm 1.5

Results for the diffusion rates obtained at 50 °C are shown in table 2. Note that in this case, the *isooctane* diffusion tube had an i.d. of 0.5 mm and a length of 12 mm. Table 3 gives an estimate of the diffusion rates at 70 °C. Only one measurement was made at 70 °C because thermal equilibrium between the liquid in the reservoir and the chamber could not be achieved, since the boiling point of hexane is 69 °C.

The results listed in tables 1–3 show that, for the same tubes, raising the temperature 20 °C results in an expected increase in the rates at which the vapors are generated. The predictable temperature effect on the rates of the vapor generation can be understood quantitatively by calculating the ratio of the vapor diffusion rates at 70°, 50°, and 30 °C. The results of these calculations are given in table 4.

TABLE 3. Diffusion rates of selected hydrocarbons at 70 °C

Time (min)	Diffusion rates, $\mu\text{g}/\text{min}^a$			
	<i>n</i> -Hexane	<i>Isooctane</i>	Toluene	<i>o</i> -Xylene
1445	130 ^b	17.9	234	71

^a The diffusion tubes listed in table 2 were also used in these measurements.

^b Approximate value.

TABLE 4. Ratios of diffusion rates at temperature intervals of 20 °C

Temperatures (°C)	<i>n</i> -Hexane	<i>Isooctane</i>	Toluene	<i>o</i> -Xylene
50/30	2.6	^a	2.5	2.7
70/50	^a	2.7	2.6	2.6

^a Not calculated.

The magnitudes of the observed changes in the diffusion rates of *n*-hexane and toluene were compared with the expected rate changes based on eq (1), using known values of diffusion constants [7] and vapor pressures [6] at 30° and 50 °C. These comparisons were made as a means of evaluating the observed temperature dependence of the diffusion rates. For these calculations, diffusion coefficients of 0.0804 cm²/s and 0.0903 cm²/s were obtained for hexane diffusion through a 0.11-cm i.d. tube, respectively, at 30° and 50 °C. Similarly, diffusion coefficients of 0.0932 cm²/s and 0.0993 cm²/s were obtained for toluene diffusion through a 0.78-cm i.d. tube at the respective temperatures. The vapor pressures of *n*-hexane are 0.241 atm (24.4 kPa) at 30 °C and 0.525 atm (53.2 kPa) at 50 °C; and for toluene, the pressures are 0.048 and 0.121 atm (4.86 and 12.3 kPa) at the indicated temperatures. The total pressure was assumed to be one atmosphere ($\sim 10^5$ Pa) in all cases. The calculations show that the ratio of the diffusion rates of hexane at 50 °C to that at 30 °C is 2.85, and the ratio of the rates for toluene vapor is 2.63 at the same temperatures. These calculated values agree within 10 percent with the experimental values of 2.6 and 2.5, respectively. The agreement indicates that the experimentally determined diffusion rates agree with the theoretical ratios predicted by eq (1).

The useful life of a specific diffusion tube as a vapor generator may be estimated from the diffusion rate and sample volume. This period, the life expectancy in days, was calculated for 2 mL (about one-half of capacity) of each compound contained in the referenced diffusion tubes, using the diffusion rates reported in tables 1–3. The results of these calculations are listed in table 5.

Although the effect of the tube dimension on the diffusion rate was not investigated in this study, eq (1) indicates that the rate should be proportional to the square of the tube diameter and inversely proportional to the tube length. Thus, diameters of 0.5, 2, and 5 mm should change the life expectancy for a specific length in the ratio of 100:16:1 respectively.

TABLE 5. *Half-life of vapor generators (in days) at various temperatures*

Temperature (°C)	<i>n</i> -Hexane	<i>Isooctane</i> ^a	Toluene	<i>o</i> -Xylene
30	55	135	33	125
50	21	146	13	45
70	--	53	5	17

^a For 30 °C the diameter of the diffusion tube was 2 mm and its length was 62 mm; for 50° and 70 °C the tube diameter was 0.5 mm and its length was 12 mm.

4.2 Method for the Determination of the Vapor Standard Concentrations

The concentrations of the selected hydrocarbons in the vapor standards were calculated from the gravimetrically determined diffusion rates and the air flow. The air flows measured with a bubble meter were frequently monitored by the rotameters for constancy.

Although the GC analysis measured the vapor concentrations, the primary use of this analysis was to establish constancy of the vapor concentrations within a run, and from run to run for a given set of experimental conditions. The response of the chromatograph was calibrated with 0.2 percent (v/v) solutions of the selected hydrocarbons in carbon disulfide. Figure 3 demonstrates the response of the GC and the column resolution for a 2- μ L sample of the carbon disulfide solution injected from a 10- μ L liquid syringe. The GC was calibrated for each hydrocarbon. The calibrations were based on the assumption that the hydrocarbons in the carbon disulfide mixture would occupy a volume predictable from ideal gas behavior when vaporized at 25 °C. The results of this calibration are given in table 6. Figure 4 shows a chromatogram of a 0.5-mL volume of vapor-air mixture (1 mL gas-tight syringe) from the vapor generator, which was operating at 30 °C with an air flow of 100 mL/min. Table 7 shows replicate determinations of vapor concentrations in ppm and the respective standard deviation for various runs made of standards generated at 30 °C. Table 8 lists chromatographic results which demonstrate the effect of increasing the diluent flow to about 1 and 2 L/min (at 30 °C). Chromatographic results are shown in table 9 for vapors generated at 50 °C.

Summaries of the average vapor concentrations determined by GC analyses at 30° and 50 °C are compared in table 10 with concentrations computed from the averaged diffusion rates at the respective temperatures. The computed values were derived from the diffusion rates, expressed in microliters of vapor per minute, and the air flow rates, expressed in liters per minute. Generally, good agreement is found between the techniques for the determination of the vapor concentrations.

In this study on the generation and calibration of vapor standards, only vapors of compounds containing carbon and hydrogen were used. However, this method of vapor-standard preparation and concentration determination is also suitable for standards consisting of other potential accelerants such as alcohols, ethers, and ketones, as well as selected compounds contained in other multi-component accelerants such as paint thinners (solvents) and varnishes.

This method can be duplicated and conveniently used by fire investigators to calibrate and evaluate the performance of combustible gas detectors. Only commercially available equipment and materials are needed. Moreover, the manufacturer of a candidate vapor generator can provide diffusion tubes of the length and bore selected by the experimenter, for generating specific vapor standards at the desired diffusion rates and desired concentrations.

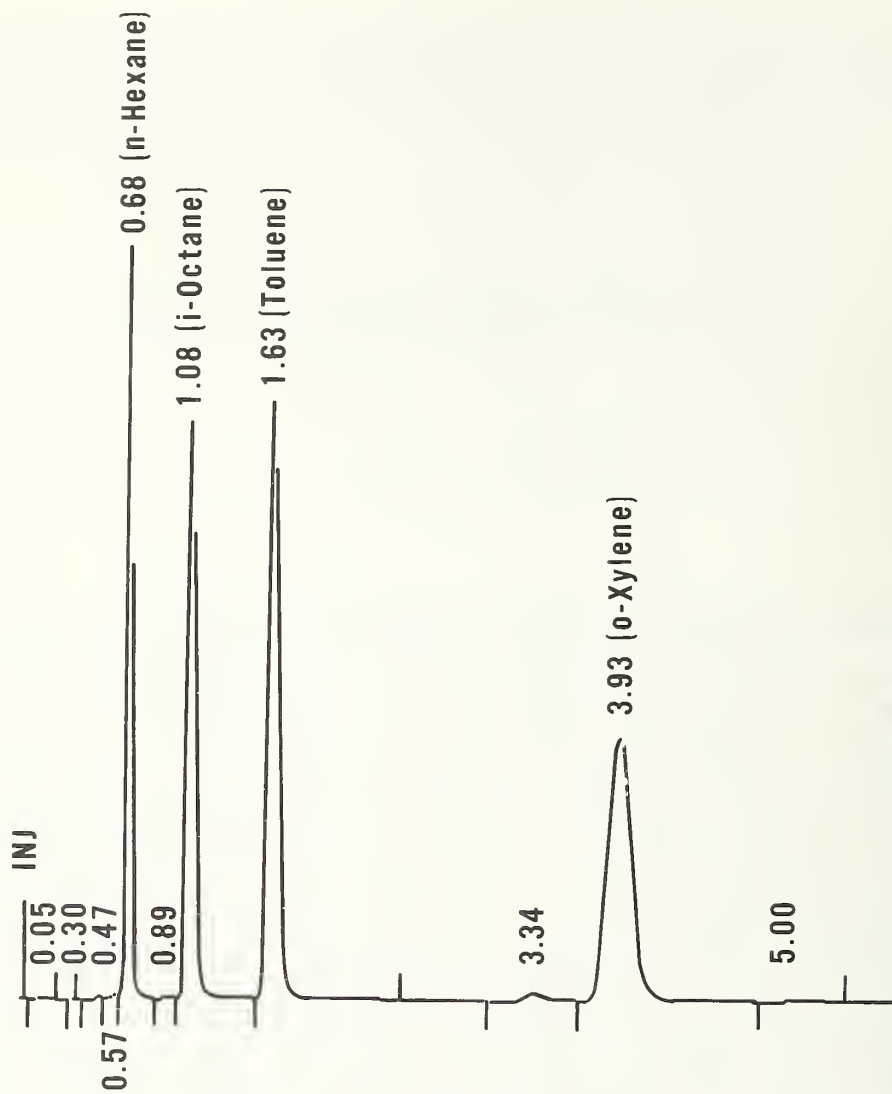


FIGURE 3. Chromatogram of selected hydrocarbon from a carbon disulfide solution.

TABLE 6. Gas chromatograph detector calibration factors obtained from carbon disulfide solutions of selected hydrocarbons

	Conversion factors [(nL vapor/peak area) × 10 ⁴]			
	<i>n</i> -Hexane	<i>Isooctane</i>	Toluene	<i>o</i> -Xylene
	9.74	7.14	8.72	7.82
	9.39	6.88	8.41	7.55
	9.56	7.01	8.57	7.64
	10.12	7.33	8.96	8.03
	10.06	7.38	9.04	8.10
	9.73	7.10	8.69	7.80
	9.77	7.15	8.76	7.83
Average ± standard deviation	9.77 ± 0.26	7.14 ± 0.17	8.74 ± 0.22	7.82 ± 0.20



FIGURE 4. Chromatogram of selected hydrocarbon vapor—air standard generated at 30 °C.

TABLE 7. Chromatographic determinations of concentrations of vapor standards generated at 30 °C with carrier/diluent gas flow at 100 mL/min

Run	Vapor concentration [ppm (v/v)]			
	<i>n</i> -Hexane	<i>Iso</i> octane	Toluene	<i>o</i> -Xylene
1	33.5	11.7	73.6	17.7
1	32.4	10.5	77.5	20.1
1	33.4	10.7	78.6	21.3
2	31.5		72.2	17.7
2	33.0	10.0	73.2	18.2
2	33.8	12.0	75.2	20.5
3	35.3	11.9	77.3	17.6
3	35.2	12.1	80.0	19.0
4	33.7	11.3	75.6	17.0
4	33.5	11.6	76.9	16.8
Average ± standard deviation	33.5±1.1	11.3±0.7	76.0±2.5	18.6±1.6

TABLE 8. *Chromatographic determination of selected hydrocarbon vapor standards generated at 30 °C with total carrier diluent flow at about 1 and 2 L/min*

Standard flow rate (L/min)	Run	Vapor concentration [ppm (v/v)]			
		<i>n</i> -Hexane	<i>Iso</i> octane	Toluene	<i>o</i> -Xylene
1.10	4	4.4	1.3	9.3	2.6
	4	3.6	1.2	8.3	1.9
	Average \pm standard deviation	4.0 \pm 0.6	1.2 \pm 0.07	8.8 \pm 0.7	2.2 \pm 0.5
2.06	4	2.3	0.6	4.2	1.0
	4	1.8	0.5	4.1	0.9
	Average \pm standard deviation	2.0 \pm 0.4	0.6 \pm 0.07	4.2 \pm 0.07	1.0 \pm 0.07

TABLE 9. *Concentration of hydrocarbon vapor standards generated at 50 °C as determined by gas chromatography*

Run	Vapor concentration [ppm (v/v)]			
	<i>n</i> -Hexane	<i>Iso</i> octane	Toluene	<i>o</i> -Xylene
3	93.9	10.6	81.8	58.8
3	92.9	10.5	82.3	62.0
3	94.5	11.1	96.0	84.0
Average \pm standard deviation	93.5 \pm 1.2	10.7 \pm 0.3	86.7 \pm 8.1	68.3 \pm 13.7

TABLE 10. *Comparison of calculated concentrations of vapor standards generated at 30° and 50 °C with concentrations determined by gas chromatographic analysis*

Hydrocarbon	Carrier gas, L/min	Vapor concentration [ppm (v/v)]					
		30 °C			50 °C		
		GC Anal	Calculated	Percent difference	GC Anal	Calculated	Percent difference
<i>n</i> -Hexane	2.06	2.0	2.3	13			
	1.10	4.0	4.3	7			
	0.10	34	47	29	94	101	7
<i>Iso</i> octane	2.06	0.6	0.7	14			
	1.10	1.2	1.4	14			
	0.10	11	15	26	11	12	11
Toluene	2.06	4.2	4.7	11			
	1.10	8.8	8.8	0			
	0.10	76	97	21	187	200	6
<i>o</i> -Xylene	2.06	1.0	1.1	9			
	1.10	2.2	2.0	10			
	0.10	19	23	18	68	64	7

5. CONCLUSIONS

It has been demonstrated that a simple vapor generator can produce concentrations of vapor on a continuous basis in the parts-per-million range. The standards may contain from one to four selected compounds. Periodic gas chromatographic analysis of the dynamic standards showed that the vapor concentrations generally are constant within 10 percent. The concentrations (ppm) of the vapor standards can be ascertained from the diffusion rate ($\mu\text{g}/\text{min}$), the vapor density (g/L), and carrier gas (air) flow (L/min).

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