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FAST GAS CHROMATOGRAPHY FOR AIR MONITORING: LIMITS OF DETECTION AND QUANTITATION*

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Gas chromatography has the potential to be a very fast method of air monitoring in the workplace and the community. The use of "fast" gas chromatographic (GC) instrumentation and methods may allow the completion of analyses in less than 10 sec when a flame ionization detector is used and in less than 30 sec when an electron capture detector is used. In this study, the fast GC system was evaluated as an air-monitoring tool for 41 different organic vapors at concentrations as low as 0.1 ppb.

Gas chromatography (GC) is often used for routine, repetitive analysis of simple mixtures. These analyses can either be laboratory based or take place by using the GC as a centrally located analyzer serving a multipoint monitoring system. Because a single chromatographic analysis of an air sample requires several minutes, the GC may be the limiting factor in such a monitoring system.

This potential for high-speed analysis was first demonstrated by Desty in 1965 when he reported the separation of as many as 15 components in less than 2 sec.(1) Since then, a number of studies have appeared that discuss both the theoretical and practical aspects of high-speed GC.(2–7) In many of these studies, extremely narrow 50- to 100-µm ID columns are used to achieve optimal analysis speed. However, adequate high-speed separation may be achieved by using columns with higher sample capacity and diameters of 0.25 mm.

Theoretical studies indicate that under optimal conditions, a 0.25-mm ID column should be capable of achieving 5000 to 7000 effective plates with retention times of 5 to 10 sec.(4,8) Although this number is low compared to most capillary systems, it is comparable to the number of plates achieved by many packed column systems with retention times of several minutes or more. Therefore, some routine GC separations that are currently performed by using packed columns or nonoptimized open tubular columns could be performed much faster with a capillary system that is optimized for speed.

Although the potential of capillary columns for high-speed analysis is well known, limitations in commercially available equipment have prevented general application of high-speed techniques. One of the limitations of such GC equipment is that the injection system produces a band of injected vapor on the GC column that is far too wide for high-speed analysis. In response to the requirement for narrow injection bands, a number of experimental inlet systems have been described in the literature.(5,9–13) Recently, two high-speed inlet systems have been described that are based on cold traps.(14,15)

Although each of these inlet system and GC system designs was shown to produce high-speed injections, many appear to be impractical, and only one was evaluated for industrial hygiene air monitoring.(15) This system, designed by the authors, was also subject to a systematic, quantitative evaluation of trapping efficiency for a variety of organics, trap heating characteristics, and chromatographic performance.(16) In this paper, the high-speed gas chromatographic system, or "fast-GC," has been evaluated for limit of detection (LOD), linearity, and for utility for the analysis of mixtures of organic vapors in air.

EXPERIMENTAL MATERIALS AND METHODS

The design of the fast GC as presented in previous publications(15,16) was followed. However, the only design feature of the trap that was retained was the trap heating circuit.(17) All other aspects of the design of the system are given in References 15 and 16. Figure 1(15) is reproduced in this paper.

Samples were introduced by using a manual, six-port valve (Valco Instrument Co., Houston, Tex.) fitted with a sampling loop that could be varied in size from 0.025 to 2.0 mL. The outlet

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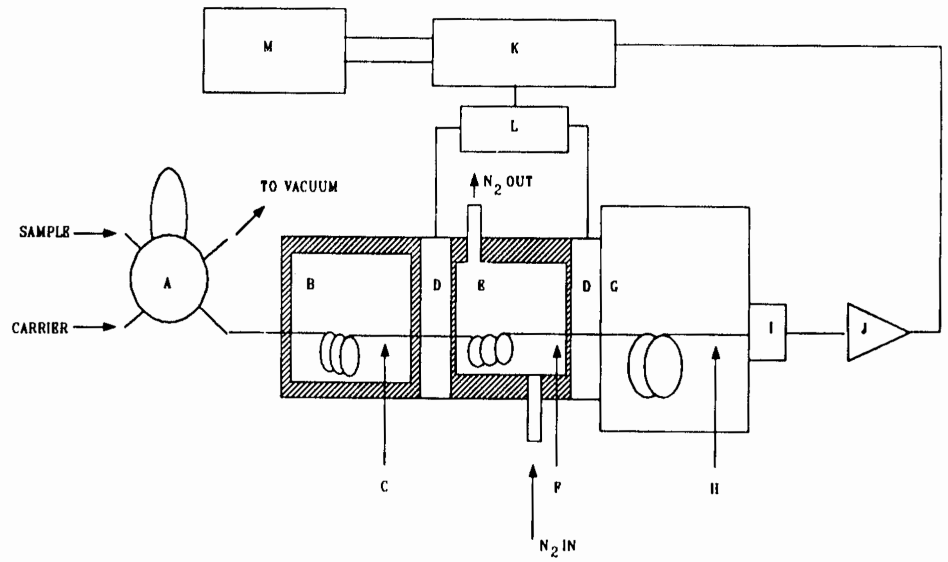

FIGURE 1. Diagram of the fast GC system.[15] *A: six-port rotary valve; B: heated buffer chamber; C: buffer column; D: heated copper electrodes; E: cold trap chamber; F: capillary cold trap; G: oven; H: capillary column; I: flame ionization detector; J: fast electrometer; K: A/D and D/A converter; L: capacitor discharge power supply; M: computer.*

of the valve was connected to a 50-cm long buffer column made from 0.25-mm ID deactivated fused silica tubing (Quadrex Corp., New Haven, Conn.). The buffer chamber was enclosed in an aluminum chamber heated to 75°C to prevent sample condensation. The downstream end was attached to a 15-cm long capillary cold trap made of 0.25-mm ID × 0.625-mm OD Monel® 400 tubing enclosed in a 9-cm long chamber made of Teflon®. The chamber was cooled to –120°C by a continuous flow of nitrogen gas, which had been cooled by running it through a copper coil submerged in liquid nitrogen.

At each end of the Teflon chamber, the trap tubing was connected to the capacitive discharge circuitry used for flash heating of the trap. During the trapping cycle, sample vapors were collected by condensation on the inner walls of the trap tubing. The frozen sample was then rapidly vaporized to form a narrow injection band by running a short pulse of current through the trap tubing. Typically, a 55-V, 55-A square wave pulse of 20 msec duration is used. The current was provided by the trap power supply, which was capable of raising the trap temperature by as much as 300°C in less than 20 msec. Details of the inlet design and performance characteristics are given elsewhere.(15–17)

The inlet system was mounted on an HNU 301 GC (HNU Systems, Newton, Mass.). All separations were performed by using isothermal analysis at temperatures between 40°C and 100°C. The column was a 5-m long, 0.25-mm ID capillary with a 0.1-μ bonded methyl silicone stationary phase (Quadrex). Hydrogen was used as a carrier gas for all separations in which a flame ionization detector (FID) was used; argon/(5%)methane was the carrier gas for all separations for which an electron capture detector (ECD) was used. The flow rate of hydrogen was 3 to 5 mL/min to produce average linear flow velocities of 102 to 170 cm/sec. This results in an unretained peak retention time of 4.9 to 2.9 sec under these conditions. For argon/methane, the flow rate was 1.0 to 1.5 mL/min to produce average linear flow velocities of 34 to 50 cm/sec.

Peaks were detected by using either an HNU Systems FID with the end of the column mounted at the flame base or an HNU Systems-Nordien ECD with a cell volume of 90 μL and the end of the column mounted inside the make-up gas annulus. Make-up gas of argon/methane was used at a flow rate of 120 mL/min.

The FID signal was directed to a fast electrometer with a response time of 7 msec. This electrometer had been specifically developed by HNU for this project. The ECD amplifier had a response time of approximately 100 msec.

Data were analyzed and the entire system was controlled by using a 12-bit analog/digital–digital/analog converter (DT2801, Data Translation, Marlboro, Mass.) mounted in a 80286-based MS-DOS personal computer. Data were collected at a frequency between 100 and 400 Hz with Labtech Notebook® software (Laboratory Technologies Corp., Wilmington, Mass.), and analysis of the data was performed by using software developed in the authors' laboratory.

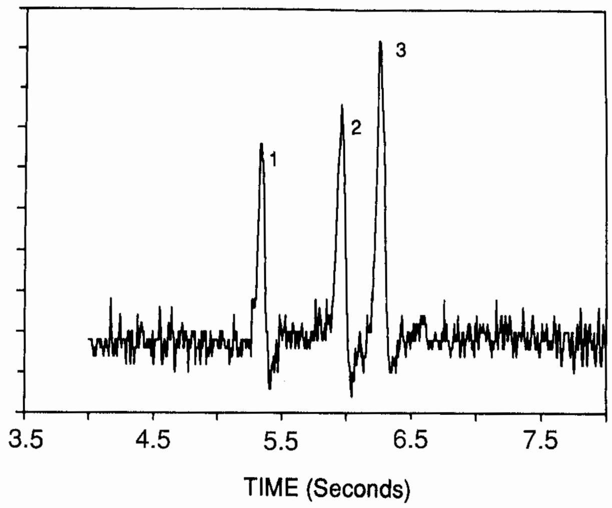

FIGURE 2. Three-component mixture of chlorocarbons in 0.5-cm^3 volume air sample at their limit of quantitation separated by fast GC in 6 sec. Peak 1: dichloromethane, 56 ppb; Peak 2: chloroform, 108 ppb; Peak 3: 1,2-dichloroethane, 51 ppb.

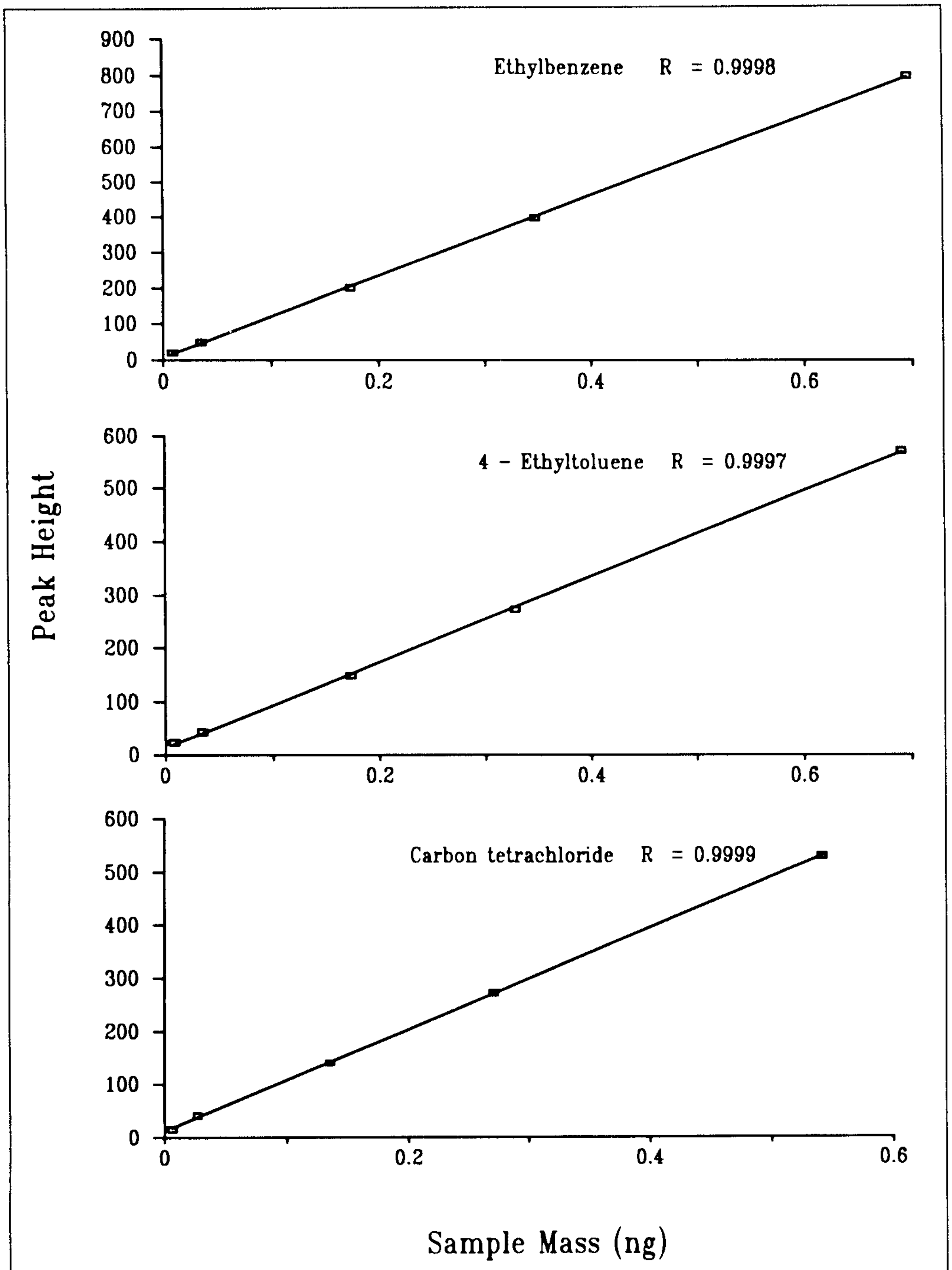

FIGURE 3. Linearity of response of fast GC to three compounds (FID used for ethylbenzene and ethyltoluene; ECD used for carbon tetrachloride)

Most of the test atmosphere samples were provided by one of the authors (Berkley); were obtained from the Environmental Protection Agency (EPA); and were delivered in gas cylinders or in Summa canisters. Other standards, and dilutions of these standards, were prepared in the authors' laboratory. "HPLC pure" solvents (Aldrich Chemical Co., Milwaukee, Wis.) were diluted in air or in nitrogen by syringe addition of the liquid into a Tedlar® bag. A comparison was made of these standards prepared by dilution of liquids in the Tedlar bags with those obtained in cylinders or canisters obtained from the EPA. No significant difference was observed.

Each LOD/limit of quantitation (LOQ) was determined from between 6 and 15 chromatographic analyses.

RESULTS AND DISCUSSION

Compounds Used for Method Evaluation

The test vapors used in this study consisted of EPA Atmospheric Research and Environmental Assessment Laboratory (AREAL, Research Triangle Park, N.C.) priority air pollutants.(18,19) These are compounds as volatile as methane and 1-carbon chlorofluoro carbons and as nonvolatile as compounds containing 6–9 carbons and up to 6 chlorine atoms. This is a list of compounds for which certain air-monitoring methods have been validated and does not constitute an official EPA regulation.

If the problems of worker and community exposure are viewed as a continuum, then monitoring of EPA-AREAL priority pollutants should be of interest to both occupational and environmental hygienists. This is especially true if consideration is not only given to concentrations of air contaminants at the worker at a specific unit process, but also at the plant fenceline, in the downwind community, and in office-type indoor air workplaces.

Thus, validation of the fast-GC method was performed over the full range of concentrations from the ppm to the low ppb level. The ppb-level data were reported in this paper. Results of the method validation in the ppm range have been previously reported, in part.(15) Other ppm-level data are not reported in this paper but are available upon request.

Limits of Detection and Quantitation

The "sensitivity" of the method was expressed as a function of the LOD and LOQ for each compound analyzed individually or in simple mixtures of less than four components in air. The LOD is defined as the sample concentration that produced a peak height equal to the average plus three times the standard deviation of the background noise. The LOQ is defined as the sample concentration that produced a peak height equal to the average plus ten times the standard deviation of the background noise.(20,21) The LOD and LOQ are calculated on the basis of analysis of an air sample of 1.0 cm^3 volume.

All chromatographic analyses were performed isothermally. Thus, peaks at longer retention times became succeedingly wider and lower. Total chromatographic time for all analyses performed with a FID was <10 sec. Because of a variety of factors explained below, the chromatographic analysis time for all analyses performed with the ECD was <30 sec. Figure 2 illustrates the chromatogram of a three-component mixture of volatile chlorocarbons at concentrations near the LOQ for each component, analyzed in under 7 sec with the FID.

Linearity of the method in the ppm concentration range has been reported in previous publications.(15,16) The same linear behavior is demonstrated for all compounds tested in this study, down to the ppb concentration range. This is illustrated for ethylbenzene, ethyl toluene, and carbon tetrachloride in Figure 3. The analyses of ethylbenzene and ethyltoluene were performed with an FID; the analyses of carbon tetrachloride were performed with an ECD. Linearity data for all compounds in this study are available upon request.

Carryover of sample on the trap between injections was also investigated. Although no significant carryover was found in routine use, the practice was to reheat the trap to 200°C between injections as prudent quality control.

Aromatic Hydrocarbons

Tables I and II give the results of the determination of the LOD and LOQ for each target analyte. Table I shows the results for 10 hydrocarbons and 6 aromatic chlorinated hydrocarbons. Note that methane, the most volatile compound on the EPA list, could not be detected by this method because the liquid nitrogen-cooled trap of the inlet system was not cold enough to trap this analyte. The LOD for each of the (nonmethane) hydrocarbons and chlorinated hydrocarbons was in the range 0.25 to 9.7 ppb, which corresponds to 0.95 to 72 pg per sample injection. A separation of an eight-component mixture of organic vapors in air taken from this list is shown in Figure 4. The separation took 8 sec.

Much of the high sensitivity, and thus the very low LOD/LOQ, associated with fast GC comes from the fact that the signal is usually associated with a peak that is only 100 msec wide (in conventional GC methods the peak may be 30 sec wide). The signal is "compressed" into a much narrower and higher peak. Thus, if all other things are equal, the longer the retention time, the wider and lower the peak and the higher the LOD/LOQ.

TABLE I. Limits of Detection and Quantitation of Aromatic Analytes[A]

	LOD[C]		LOQ[C]		Conditions	
Compound[B]	pg	ppb	pg	ppb	Time (sec)	Temp (°C)
Benzene	0.95	0.25	3.2	0.9	5.10	100
Toluene	2.6	0.7	8.5	2.3	5.40	100
Chlorobenzene	3.6	0.8	12	2.6	5.87	100
Ethylbenzene	3.9	0.9	13.1	3.0	5.80	100
p-Xylene	15.2	3.5	50.7	11.7	9.59	60
m-Xylene	19.0	4.4	63.3	14.6	9.65	60
o-Xylene	3.4	0.8	9.8	2.6	6.10	100
Styrene	21.8	5.1	72.7	17.1	5.94	100
4-Ethyltoluene	4.9	1.0	16.4	3.4	6.60	100
1,3,5-Trimethylbenzene	5.8	1.2	19.1	3.9	7.16	100
1,2,4-Trimethylbenzene	2.3	0.5	7.5	1.5	7.26	100
m-Dichlorobenzene	11.9	2.0	39.6	6.6	7.50	100
p-Dichlorobenzene	13.3	2.2	44.4	7.4	7.60	100
o-Dichlorobenzene	16.6	2.8	55.4	9.2	8.04	100
Benzylchloride	4.3	0.8	14.2	2.7	5.30	100
1,2,4-Trichlorobenzene	72.0	9.7	240	32.4	7.86	100

[A] Methane analysis cannot be performed because of volatility.

[B] Arranged in order of increasing boiling point.

[C] All analyses performed by using FID.

[D] *p*- and *m*-Xylene retention times 10 sec; *o*-xylene retention time 6 sec. Thus, LOD/LOQ for *o*-xylene is apparently lower than for *p*- and *m*-xylene. See text for explanation.

This is illustrated in Figure 5, in which the peak for trimethyl benzene becomes lower and wider as the retention time is increased from 6.3 to 7.6 to 9.4 sec. As expected, the LOD increases with increasing retention time. The LOD and LOQ at a retention time of 6.3 sec are 1.06 ppb and 3.54 ppb, respectively. At a retention time of 9.3 sec, the LOD and LOQ are 2.12 ppb and 7.08 ppb, respectively. Consequently, the LOD/LOQ values given in Tables I and II are in fact valid only for those retention times and sample compositions under which they were analyzed. The retention time of each compound is given for the LOD/LOQ values in the tables.

The effect of atmospheric moisture was tested when the FID was used. In a previous publication, there was no difference in results obtained when analyzing airborne benzene, toluene, and xylene vapors at 20% and 80% relative humidity in the ppm range.(15) In this test, the effect of relative humidity was tested for additional analytes and no effect was found.

This is illustrated in Figure 6, which shows a 1-cm^3 sample of dichloromethane (140 ppb), trichloromethane (270 ppb), tetrachloromethane (257 ppb), and 1,2-dichloroethane (175 ppb) analyzed at 10.7% and at 78.4% relative humidity. (At 78.4% relative humidity, a 1-cm^3 air sample at 22.4°C would contain 15.5 µg of water.) No differences were found with the results. This experiment was repeated with other analytes, including with benzyl chloride, a compound that is labile in the presence of water. No effects of water were observed.

The reasons for determining the effect of changes in relative humidity are as follows.

TABLE II. Limits of Detection and Quantitation of Halogenated Analytes

	LOD[B]		LOQ[B]		Conditions	
Compound[A]	pg	ppb	pg	ppb	Time (sec)	Temp (°C)
Freon® 12[C]	77.1	15.6	257	52	13.84	40
Methylchloride	11.6	5.6	38.7	18.5	4.81	40
Vinylchloride	3.4	1.3	11.3	4.4	4.71	40
Freon 114[C]	102	14.7	342	48.5	14.52	40
Methylbromide	5.2	1.3	17.3	4.5	4.94	35
Ethylchloride	5.5	2.1	18.2	6.9	4.94	40
Freon 11	636	113	2099	373	5.06	40
Freon 11[C]	0.88	0.16	2.9	0.5	15.26	40
1,1-Dichloroethene	9.5	2.4	31.7	8.0	5.30	40
Dichloromethane	15.5	4.5	51.5	14.8	5.28	40
Freon 113[C]	21.9	2.8	72.9	9.5	15.76	40
1,1-Dichloroethane	14.1	3.5	46.9	11.6	4.57	40
Cis-1,2-Dichloroethene	17.2	4.3	57.3	14.5	5.60	40
Chloroform	27.5	5.6	91.8	18.8	5.90	40
Carbon tetrachloride[C]	1.5	0.2	4.8	0.8	21.60	80
1,2-Dichloroethane	12.9	3.2	43.0	10.6	6.60	40
Methylchloroform	26.2	4.8	87.5	16.0	4.80	50
Trichloroethene	16.5	3.1	55.1	10.2	5.34	80
1,2-Dichloropropane	18.8	4.1	62.5	13.5	5.19	80
Cis-1,3-Dichloropropene	26.3	5.8	87.6	19.3	5.72	80
Trans-1,3-Dichloropropene	48.3	10.6	161	35.5	6.04	80
1,1,2-Trichloroethane	10.1	1.8	33.5	6.1	5.80	80
Tetrachloroethene[C]	4.5	0.7	14.9	2.2	19.08	80
1,2-Dibromethane	70.3	9.2	234	30.5	5.50	100
1,1,2,2-Tetrachloroethane[C]	31	4.5	103	15	18.98	100
Hexachlorobutadiene[C]	9.6	0.9	32	3.0	30.84	120

[A] Arranged in order of increasing boiling point.

[B] All analyses performed by FID, except as indicated.

[C] Analysis performed by ECD.

1. It is good practice to do so for any method developed for ambient air monitoring, even if no literature references are available that indicate that such an effect has been previously observed.
2. Because the cold trap may be kept at temperatures as low as −120°C, ice may form in the trap, which might contribute to a reduced trapping efficiency, reduced reinjection efficiency, or the hydrolysis of water-reactive compounds.
3. Incomplete trapping of water vapor or any other atmospheric gases might lead to unforeseen changes in instrument response. Indeed, this was seen when the ECD was used (see text below).

Nonaromatic Compounds, Including Freons

Table II shows the LOD/LOQ values for the nonaromatic compounds on the EPA-AREAL

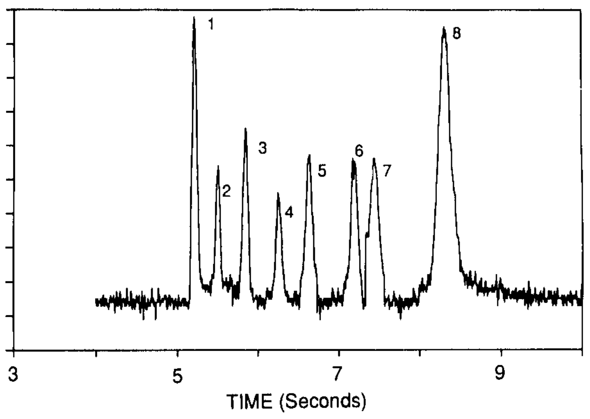

FIGURE 4. Eight-component mixture of aromatic compounds in 1.0-cm^3 volume air sample separated by fast GC in 8 sec. Peak 1: benzene, 5.0 ppb; Peak 2: toluene, 5.0 ppb; Peak 3: ethylbenzene, 8.0 ppb; Peak 4: o*-xylene, 5.5 ppb; Peak 5: 4-ethyltoluene, 7.0 ppb; Peak 6: 1,3,5-trimethylbenzene, 7.0 ppb; Peak 7:* m*-dichlorobenzene, 4.4 ppb; Peak 8:* o*-dichlorobenzene, 9.0 ppb.*

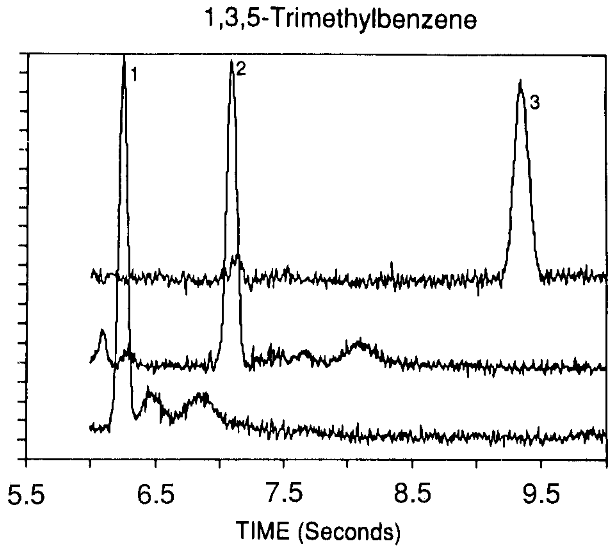

FIGURE 5. Chromatograms of 1,3,5-trimethylbenzene at concentration near limit of quantitation. Column temperature shown in Tracing 1: 120°C; Tracing 2: 100°C; Tracing 3: 80°C.

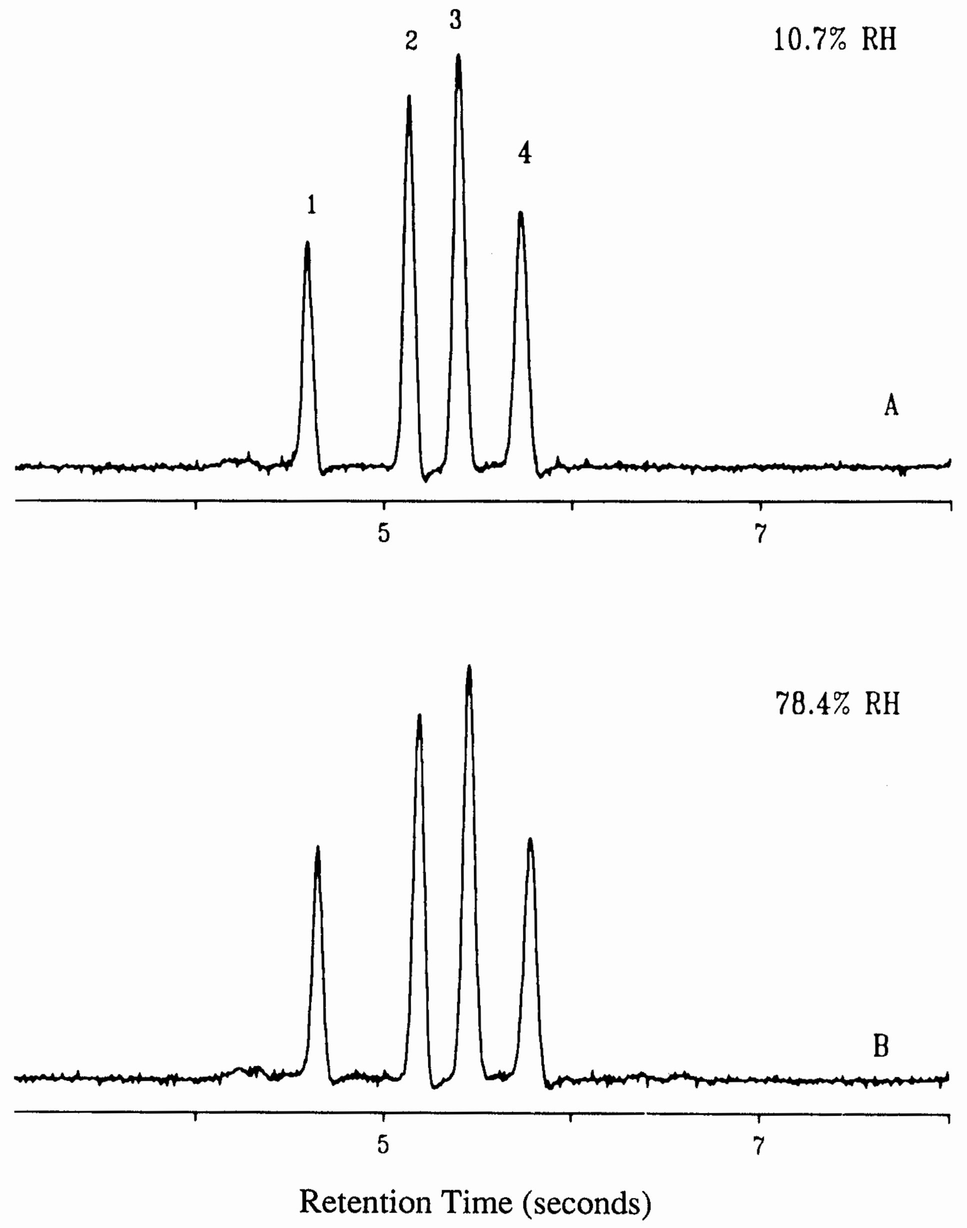

FIGURE 6. Four-component mixture of chlorocarbons in 1.0-cm^3 volume air sample separated by fast GC in 6 sec. Top tracing: 10.7% relative humidity; Bottom tracing: 78.4% relative humidity. Peak 1: dichloromethane; Peak 2: trichloromethane; Peak 3: tetrachloromethane; Peak 4: 1,2-dichloroethane.

priority list. These compounds include Freons® (4 compounds), one-carbon halocarbons (6 compounds), two-carbon halocarbons (11 compounds), cis and trans 1,3-dichloropropene, and hexachlorobutadiene. Some halocarbons have been chromatographed with the FID, some with the ECD, and some with both conditions. These are indicated on the table.

As can be seen from the example of Freon 11 (fluorotrichloromethane), the LOD when using an FID is 113 ppb. Although this is more than adequate for monitoring of workplace air, it is inadequate for monitoring of community ambient air and possibly inadequate for indoor air. This inadequate LOD is the case for perhalocarbons because the FID will not show good sensitivity to compounds lacking carbons plus hydrogens. However, the LOD for Freon 11 when using an ECD is 0.16 ppb, which is almost three orders of magnitude lower than that obtained when using an FID. The ECD is sensitive to halocarbons, especially those with three or more chlorine atoms.(22)

An improvement in LOD for halogenated compounds when switching from an FID to an ECD can be as much as five orders of magnitude for certain compounds. However, the full improvement is not obtained for ECD when used with fast GC because of a number of factors, primarily involving peak broadening.

The factors that contribute to peak broadening, when compared to the FID, include (1) the volume of the ECD cell (90 µL) versus that of the FID (essentially "0" volume); (2) the slower response of the ECD electronics (rise time of approximately 100 msec) versus that of the FID (approximately 7 msec); and (3) the use of Ar/methane carrier gas for the ECD, versus hydrogen for the FID. This last difference, necessary because the ECD required Ar/methane carrier gas for sensitivity and stability, increased the plate height and thereby reduced column efficiency when compared to that obtainable when using hydrogen as a carrier gas.(22) This longer retention time and broader peaks are illustrated in Figure 7. Significant errors in quantification based on peak heights or areas might be expected for such a chromatogram.

The use of hydrogen carrier gas with argon/methane make-up gas to the ECD detector cell should solve the problem of loss of column efficiency. Indeed, in preliminary tests, this improvement was noted. When these experiments are completed, this will be the subject of a separate paper in which the use of hydrogen carrier gas and an ECD with high-speed electronics will be reported. Many chlorinated compounds respond well to the FID, especially two-carbon compounds with at least one hydrogen and no more than two chlorine atoms. Most of these compounds have LODs below 5 ppb when using the FID. A comparison of the separation of components

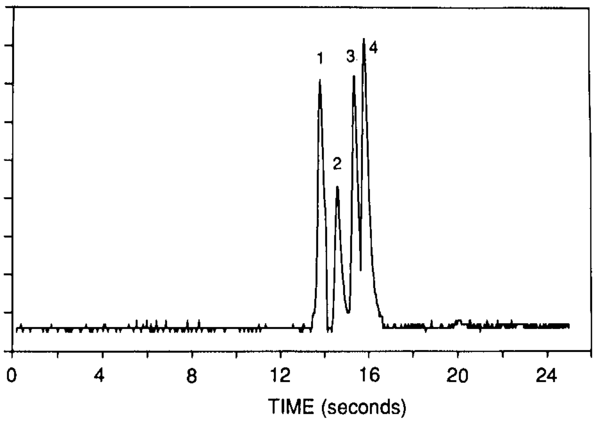

FIGURE 7. Four-component mixture of Freon® in 1.0-cm^3 volume air sample separated by fast GC in 16 sec with electron capture detector. Peak 1: Freon 12, 0.81 ppm; Peak 2: Freon 114, 0.83 ppm; Peak 3: Freon 11, 0.011 ppm; Peak 4: Freon 113, 0.17 ppm.

of the vapor mixtures obtained in Figures 6 and 7 illustrates the advantage in using the FID whenever possible.

The effect of atmospheric moisture, as well as atmospheric oxygen, was also tested with the ECD. In this test, target analyte vapors were prepared in nitrogen, in air, and in humidified air. The moisture showed no effect, as was the case with the FID. However, the difference between the chromatograms obtained by using the ECD when the sample was prepared in nitrogen or in air was that in air, a broad (about 20-sec wide) flat baseline shift took place immediately after injection of the sample. This shift was caused by the fact that oxygen is too volatile (boiling point = –183°C) to be trapped in the cold trap injector but does cause a shift in the ECD signal.(22) However, no difference in sensitivity was observed for analyte peaks that coeluted with this baseline shift.

Limitations of Fast GC

As with any monitoring or analysis method, there are limitations that must be considered.

1. Not all compounds can be chromatographed. With all gas chromatographic methods, there are fundamental limitations based on boiling point, polarity, and thermal lability.
2. Not all compounds have been evaluated for analysis by fast GC. Although the results with 41 compounds were impressive, this represents a very small fraction of potential analytes.
3. The system requires approximately 1 L of liquid nitrogen per hour of use. Although a new system is being designed to radically reduce the nitrogen use, it has not yet been tested.
4. Isothermal separation of mixtures is perhaps the biggest limitation. By using traditional GC methods, complex mixtures can be separated far more efficiently with temperature programming, which cannot be used with fast GC.
5. The whole concept of fast GC is to optimize a system to separate critical groups of compounds in a very short time. In doing this, separating power (resolution) is compromised.
6. By using traditional GC methods, coupling a GC to a mass spectrometer (MS) or to a Fourier transform infrared (FTIR) spectrophotometer is a common, and very powerful, practice that is used to identify unknown components of the mixture. Neither GC-MS nor GC-FTIR can be routinely used at present with fast GC because the MS and FTIR are too slow.
7. Fast GC instruments are not yet commercially available.

CONCLUSIONS

High-speed gas chromatography, or fast GC, has been evaluated for 41 compounds. The flame ionization and electron capture detectors were evaluated for use. The FID was found to have the advantages of detection speed and of high sensitivity to hydrocarbons and to certain halocarbons. The ECD had the advantage of superior performance for use during chromatographic analysis of perhalogenated compounds.

With the reservations listed above, the fast GC method should now be considered as a viable option for application to industrial and environmental health air-monitoring problems. However, additional investigations must be performed, such as improvement of resolution when using the ECD and investigations of optimal conditions for separation of more complex mixtures of air contaminants.

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