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A Method for Determination of Methyl *tert*-Butyl Ether in Gasoline Vapors and Liquid Gasoline Samples

John Palassis,[A,B] Richard W. Hartle,[C] and John L. Holtz[A]
Divisions of [A]Physical Sciences and Engineering, [B]Training and Manpower Development, [C]Surveillance Hazard Evaluations & Field Studies, National Institute for Occupational Safety and Health, Centers for Disease Control and Prevention, Public Health Service, Department of Health and Human Services, 4676 Columbia Parkway, Cincinnati, Ohio 45226-1998

This article describes the development of a method for determining the presence of methyl tert-butyl ether (MTBE), in a matrix of gasoline vapors and in liquid gasoline samples, with the simultaneous analysis of benzene, toluene, xylenes, and total hydrocarbons. An assessment of the method's performance based on field use is also included. The method recommends collection of air samples for MTBE on 400- and 200-mg coconut-shell charcoal tubes in series, desorption in carbon disulfide, split-vent injection, analysis by gas chromatography (GC) using a capillary column, and detection by flame ionization detector. The analysis of liquid gasoline samples is accomplished by direct GC injection. The estimated limit of detection for air samples is 20 μg MTBE per sample, and 0.002 percent MTBE for liquid gasoline samples. The estimated analytical precision of MTBE standards in the range of 9 to 426 μg/mL is 4.5 percent. Samples stored at ambient temperature are stable for at least 5 days. MTBE-spiked samples stored at −7°C for 27 days indicated 90 percent recovery. Air sample volumes of 8 L are recommended when MTBE concentrations are near 10 ppm. Larger volumes should be collected if lower MTBE concentrations are expected. The method was field tested at different service stations in Los Angeles, California, Phoenix, Arizona, and Cincinnati, Ohio, where 121 personal (breathing zone) air samples were collected on charcoal tubes, and 75 liquid gasoline samples were collected and analyzed for MTBE and other gasoline hydrocarbons. The method worked well for all of these samples. The majority of personal exposures to MTBE were near the detection limit. MTBE concentrations in gasoline bulk samples varied from nondetectable to 12.4 percent. **Palassis, J.; Hartle, R.W.; Holtz, J.L.: A Method for Determination of Methyl *tert*-Butyl Ether in Gasoline Vapors and Liquid Gasoline Samples. Appl. Occup. Environ. Hyg. 8(11):964-969; 1993.**

Introduction

As lead is phased out of motor fuels, methyl tert-butyl ether (MTBE) is gaining acceptance as an octane booster and a blending agent(1) in gasolines, having itself an average octane value of 110.(2) Currently, the Environmental Protection Agency (EPA) has permitted up to 15 percent blending of MTBE in gasolines. Certain cities, where there is an air pollution problem, have been given permission by the EPA to blend up to 20 percent MTBE as an oxygenate in gasolines to reduce automobile exhaust pollution.

Long-term toxicological studies have not been performed for MTBE. There is neither a recommended exposure limit for MTBE by the National Institute for Occupational Safety and Health (NIOSH), nor a permissible exposure limit established for MTBE by the Occupational Safety and Health Administration.

NIOSH researchers in coordination with the American Petroleum Institute developed a project to determine exposures to MTBE, benzene, toluene, xylenes, and total hydrocarbons among service station attendants in different cities. The NIOSH *Manual of Analytical Methods*(3) did not contain a method for MTBE in air, nor was there an analytical method available for MTBE from other governmental agencies or industrial hygiene associations. A literature search revealed no direct analytical method for personal air sampling and measurement of MTBE. Several research papers, mainly from the petroleum industry, described analysis and measurement of various components of gasoline. However, the gas chromatographic instrumentation used in these analyses was somewhat complicated, incorporating multidimensional gas chromatographs such as dual-column, dual-oven, dual-detector configurations, backflushable columns, and gas chromatographic heart-cutting techniques that are not typically available in many environmental and industrial hygiene laboratories. Therefore, an attempt to develop a method for MTBE that would be simple and usable by most laboratories was undertaken.

Lockwood and Caddock,(4) from Shell Research Ltd. in Great Britain, described the analysis of premium gasolines and light-catalytically cracked gasolines that were spiked with alcohols and ethers, one ether being MTBE. They used a 50-m capillary column with a nonpolar stationary phase and an initial oven temperature of 8°C, achieved with a subambient cooling accessory. Their gas chromatographic conditions appeared feasible for separating MTBE from other gasoline components and was used as a basis, with modifications, to develop the analytical method reported herein.

Experimental Materials and Methods

Reagents

Reagent grade MTBE, carbon disulfide, hexane, benzene, toluene, *m*-xylene, *p*-xylene, methanol, and ethanol were obtained from J.T. Baker Co. (Phillipsburg, New Jersey) and Burdick and Jackson (Muskegon, Michigan). Carbon dioxide in cylinders equipped with eductor tubes was used for subambient cooling of the gas chromatographic oven. Liquid nitrogen has been used and found to be equally satisfactory for subambient cooling.(5) High purity helium was used as carrier and makeup gas. In-house compressed air, purified for moisture and hydrocarbons, and hydrogen, electrolytically generated and dried, were used for the flame ionization detector (FID). Coconut-shell charcoal tubes (separate 400- and 200-mg tubes, lot 120, catalog # 226-37) were obtained from SKC. Unleaded gasolines, premium and regular grades, with octane values ranging between 87 to 92, were obtained from local service stations.

Apparatus

The initial analytical research was conducted with a Hewlett-Packard (HP) model 5880-A gas chromatograph equipped with temperature programming, an HP model 7673A autosampler, and a carbon dioxide subambient cooling accessory. The analytical column was a 50-m × 0.32-mm inner diameter (i.d.) fused silica capillary column coated with 0.5-μm film of cross-linked dimethyl silicone gum stationary phase; the gas chromatograph was equipped with a split-vent flow injector and FID. An HP model 3354 Lab Automation Computer System was used for integration. Personal air sampling pumps (Dupont model P200) were used for drawing air through the charcoal tubes for the room temperature storage study and for the breakthrough volume study.

Qualitative Experiments

In preliminary qualitative experiments three different polarity capillary columns were used to check the elution order and separation of MTBE with regard to other components from a national brand premium gasoline sample. One-microliter injections of nine individual compounds were made on each column to determine the elution order and retention times of the nine compounds. These compounds were methanol, ethanol, carbon disulfide, MTBE, benzene, hexane, toluene, *m*-xylene, and *p*-xylene. All of these compounds, except for carbon disulfide, are components of gasoline. A mixture of these compounds in carbon disulfide was prepared and analyzed periodically to check the stability of retention times and column resolution.

The premium gasoline sample spiked with MTBE was analyzed with an initial temperature of 30°C followed by temperature programming to improve the MTBE peak resolution. Column flow rates, injection split-vent flow rates and temperature programming were modified for each of four analytical columns, until no further improvement in peak resolution was achieved.

The first column evaluated was a medium polarity DB-210 fused silica capillary column (30-m × 0.236-mm i.d. with a 0.25-μm film coating). Separation of the MTBE peak was not achieved due to interference from coeluting gasoline component peaks. The second column was a nonpolar DB-1 fused silica capillary column (60-m × 0.236-mm i.d. with a 0.25-μm film coating). There was a substantial improvement in the MTBE peak resolution, but baseline separation from other gasoline components could not be achieved. The third column was a polar DB-WAX column (30-m × 0.5-mm i.d. with a 0.5-μm film coating). Again, coeluting gasoline component peaks severely interfered with the MTBE peak.

Finally, an HP dimethyl silicone gum capillary column (50-m × 0.32-mm i.d. with a 0.5-μm film coating) was evaluated also at an initial 30°C temperature because the subambient cooling accessory had not yet arrived. The HP column was similar in polarity to the DB-1 column utilized earlier; this column was 50 m instead of 60 m long and contained about twice the film thickness of the DB-1 (i.e., 0.54 instead of 0.25-μm film of dimethyl silicone gum as a stationary phase). The separation using the HP column was better than with the DB-1 column; however, at 30°C the MTBE peak was not completely resolved from an interfering peak. The interfering peak was identified as 2,3-dimethylbutane by gas chromatography/mass spectrometry (GC/MS).

After the subambient cooling accessory was installed, the analytical conditions reported by Lockwood and Caddock(4) were employed. They recommended a temperature program with an initial temperature of 8°C for the analysis of liquid gasoline. Implementing that temperature program, the MTBE peak was still not baseline-resolved from the 2,3-dimethylbutane peak. The analytical conditions were modified and optimized for the best MTBE peak separation; these conditions are presented in Table I. A typical chromatogram of a premium gasoline sample analyzed under the conditions listed in Table I is depicted in Figure 1; the individual peaks were identified by GC/MS. The analytical conditions listed in Table I were used for the remaining experimental work. In one extended run of 45 minutes, over 260 chromatographic peaks were observed from the premium gasoline sample, indicative of very good capillary column efficiency. *Better resolution for MTBE can be obtained by lowering the initial temperature to −15°C.*(5)

Six gasoline samples purchased from different service stations in the Cincinnati area were first analyzed as received to check peak resolution of MTBE and benzene peaks; the gasolines were then spiked with MTBE to make

TABLE I. Gas Chromatographic Conditions

Initial temperature	0°C
Initial hold time	7 minutes
Temperature step program 1	5°C/min
Final step temperature	25°C
Final hold time	0 minutes
Temperature step program 2	25°C/min
Final step temperature	250°C
Final hold time	5 minutes
Recycling time to 0°C	4 minutes
Analysis time	26 minutes
Injection volume	1 μl
Split-vent flow rate	100 ml/min
Split-vent ratio	60:1
Helium carrier flow rate	1.7 ml/min

2 percent (vol/vol) concentrations, and reanalyzed. Utilizing the same chromatographic conditions listed in Table I, methanol, ethanol, MTBE, hexane, benzene, toluene, and the xylenes peaks were resolved. Based on these satisfactory qualitative results, the second phase of work was initiated.

Quantitative Experiments

The first task of the quantitative work was to determine the precision and linearity of MTBE standards over an extended concentration range that covers the expected 0.1 to 15 percent of MTBE in gasoline. Calibration standards were prepared at seven concentration levels, three standards per level, over an analytical range of 0.74 to 111.1 mg/ml MTBE in carbon disulfide. MTBE calibration standards, which cover the expected MTBE air concentrations, were prepared in the range of half the calculated limit of detection (LOD) (i.e., 9.3 μg/ml, up to 426 μg/ml).

Charcoal Tubes

Commercially available charcoal tubes containing 400 mg of coconut-shell charcoal (SKC Lot 120) in a single section were used along with separate back-up tubes containing 200 mg of charcoal. The reason for choosing the tandem 400/200 mg charcoal tubes over the smaller size tubes (i.e., 100/50 mg) was to have an extra capacity factor to avoid overload of the tubes with gasoline vapors that could cause breakthrough. For calculating the spiking amounts of MTBE an assumption was made that a 2-mg loading of gasoline vapors would be collected per tube; this was based on experience obtained from other surveys.(6) MTBE concentrations in gasoline in the range of 1 to 20 percent (vol/vol) would result in 0.020 to 0.400 mg MTBE per tube. Amounts prepared in this range were spiked on the 400-mg charcoal tubes for the recovery and storage studies.

MTBE/Gasoline Spiking Mixtures

Three spiking solutions were prepared in the range of 0.1 to 4 percent MTBE in a gasoline that was analyzed by GC and determined to contain no MTBE. The spiking volumes

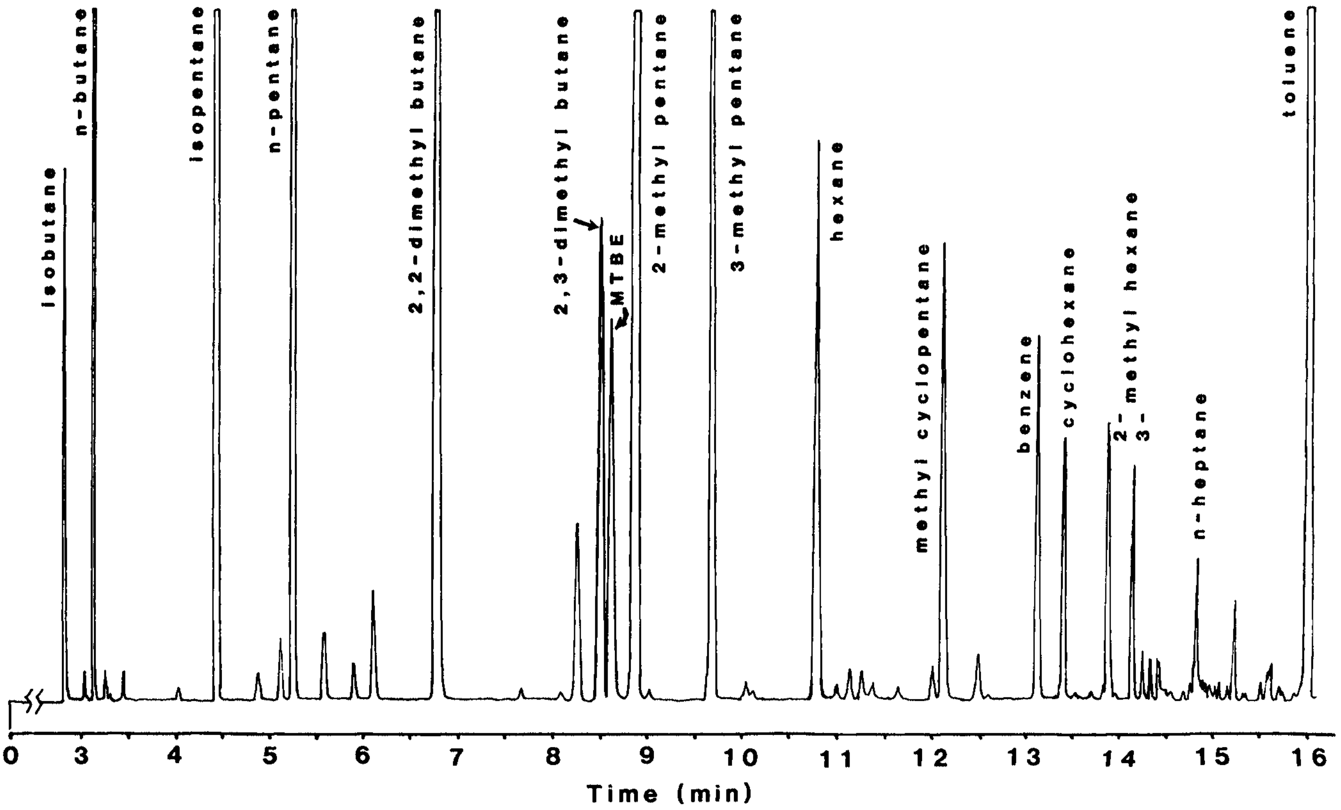

FIGURE 1. Chromatogram of a premium gasoline sample (national brand). Fifty meter-dimethyl silicone gum nonpolar capillary column.

varied from 7 to 24 μL. This corresponded to levels of 17.8 to 415 μg MTBE per charcoal tube.

Spiking Charcoal Tube Procedure

To prepare fortified charcoal tube samples, the ends of charcoal tubes were broken and connected to a vacuum pump that drew air through a critical orifice at 100 cc/min. Then, a specified volume of the MTBE/gasoline mixture was drawn out of a vial with a microsyringe and was spiked inside the charcoal tube near the glass wool plug. After several minutes of drawing air through the charcoal tube, the tube was disconnected and end-caps were immediately placed on both ends. The charcoal tube was marked for identification and placed in a freezer at −7°C.

MTBE Extraction Procedure

The contents of each spiked charcoal tube were emptied into a 10-ml vial containing 2 ml carbon disulfide, and the vial was immediately capped and crimped. The vial was left at room temperature for 30 minutes and was swirled every 10 minutes for a 10-second duration. At the end of the 30-minute period, a 1-ml aliquot was taken from each 10-ml vial with a syringe and was transferred to an autosampler vial for analysis.

MTBE Recovery/Storage Stability Study

Using the spiking procedure, 24 charcoal tubes were spiked at 6 levels of MTBE, 4 per level, in the range of 17.8 to 415 μg/tube. These levels cover the 2-mg gasoline vapor loading expected to be collected per charcoal tube, which may contain 1 to 20 percent MTBE vapor. Immediately after spiking they were placed in the freezer at −7°C. For the recovery study, one tube from each level (N = 6) was taken out of storage, extracted, and analyzed with a new set of standards at intervals of 1, 17, 23, and 31 days.

Room Temperature/Freezer Comparative Storage Study

A 4 percent spiking stock mixture of MTBE in gasoline was prepared; 14 μl of this stock were spiked onto 12 separate tubes resulting in a loading of 415 μg MTBE per tube; after spiking they were separated into three sets. The first set consisted of four charcoal tubes that were capped and left at room temperature (25°C) in the dark for 5 days. For the second set, lab air was drawn through the four charcoal tubes at 100 cc/min for 6 hours using four calibrated Dupont P200 personal sampling pumps. The tubes were then placed in the dark in a freezer at −7°C for 5 days. The remaining four charcoal tubes were left at room temperature for 6 hours with no air drawn through them, then placed in the dark in a freezer at −7°C for 5 days. At the end of the storage time all tubes were extracted and analyzed with a new set of calibration standards.

Breakthrough-Volume Study

MTBE breakthrough-volume experiments were conducted with the 400-mg charcoal tube connected in series with a short piece of tygon tubing to a back-up charcoal tube containing 200 mg of charcoal. Air was drawn through the tubes at 100 cc/min using a Dupont P200 personal sampling pump. This setup was used also in the field to collect air samples.

Using the 20 percent MTBE/gasoline mixture, a 135-μl aliquot containing 20 mg MTBE was spiked onto the 400-mg charcoal tube. A 5 percent breakthrough of MTBE would be equivalent to 1 mg MTBE. After spiking, air was drawn through the tube at 2-minute intervals for the first 20 minutes, then at 10-minute intervals; at the end of each interval the air flow was stopped and the back-up tube was changed, capped, and marked. Eleven back-up tubes were collected in 30 minutes. The second experiment repeated the first collection procedure for a total time of 60 minutes. A 10-mg aliquot of MTBE in gasoline was spiked on a new charcoal tube, and air was sampled at a flow rate of 100 cc/min. Fourteen back-up tubes were collected. The third experiment was conducted using a 10-mg aliquot of MTBE in gasoline spike on a new charcoal tube. The air flow rate was twice the previous flow rate, thus 200 cc/min, for a continuous period of 6.5 hours. The back-up tubes were changed at 30-minute intervals. Thirteen back-up tubes were collected. A 5 percent breakthrough would equal 0.5 mg of MTBE on the back-up tube. Out of a total of 38 back-up tubes collected, 21 tubes were selected and analyzed for breakthrough determination.

Field Application

Personal breathing zone samples were collected among service station attendants in Cincinnati, Ohio, Phoenix, Arizona, and Los Angeles, California. It was expected that different MTBE exposure potentials would result because of the MTBE blend variations in gasolines. A total of 75 liquid samples of regular, premium, and super grades of gasoline were analyzed by direct-injection GC analysis using this method. In addition, 121 personal (breathing zone) charcoal tube samples were collected and analyzed by the analytical method described above. The first personal air samples analyzed indicated low concentrations at or near the LOD of MTBE. Therefore, to ascertain that appreciable amounts of MTBE, benzene, and other hydrocarbons would be collected on charcoal tubes, the NIOSH industrial hygienists used sampling flow rates up to 500 cc/min. Samples with air volumes of 24 to 120 L in 4-hour shifts were collected.

Results and Discussion

Qualitative Analysis

The HP 50-m capillary column accomplished the best resolution of all the gasoline components up to C_7 hydrocarbons that include the compounds of interest to industrial hygienists, (i.e., methanol, ethanol, MTBE, benzene, hexane, toluene, *m*-xylene, and *p*-xylene). Personal exposure to total hydrocarbons can also be determined from the

TABLE II. MTBE Calibration Standards*

Concentration (μg/ml)	Number of Standards	Peak Area (X ± s)	% RSD
9.3	4	1.8 ± 0.2	12.1
18.5	5	4.2 ± 0.2	5.1
55.5	5	17 ± 0.5	2.7
148.0	5	47 ± 1.8	3.8
222.0	5	75 ± 1.7	2.2
315.0	5	105 ± 3.7	3.5
426.0	5	140 ± 3.1	2.2

*Coefficient of variation ($\overline{CV}$) = 4.5 percent; slope (s) = 0.335; y-intercept = −1.5; standard curve error = 2.15; linear correlation coefficient = 0.998; limit of detection = 20 μg/ml; limit of quantitation = 70 μg/ml.

same analytical run by totaling the chromatographic peak areas of a personal air sample and comparing against hexane standards. Results for total hydrocarbon exposure would be reported as equivalent to hexane concentrations.

Quantitative Analysis

The extended calibration curve that can be used to determine the concentration of MTBE in liquid gasoline covered the range of 0.74 to 111.1 mg/ml MTBE and indicated a pooled coefficient of variation of 3.3 percent. Linear regression analysis indicated a linear correlation coefficient (r) of 0.9998.

The calibration curves used for determining air concentrations of MTBE covered the analytical range of 9.3 to 426 μg/ml MTBE. An example of these calibration data presented as a composite is displayed in Table II. The LOD defined by the American Chemical Society (ACS) was calculated as three times the standard error of the curve divided by the slope and reported rounded off to one significant figure. The limit of quantitation, defined by ACS as 3.3 times the LOD, was calculated and rounded off to one significant figure.

Storage Stability Study

The first-day storage recovery results for MTBE on charcoal indicated a mean recovery of 102.2 ± 8.7%, which included the recovery of the spike made near the LOD. No trend was observed in the recovery results. The 17.8, 51.8, 141, 207, 326, and 415 μg MTBE per tube levels indicated 118, 94.8, 104.3, 101.9, 94.5, and 99.5 percent recoveries, respectively. The summary results of the 31-day recovery/storage stability study are presented in graphical form in Figure 2. Note that 95 percent of MTBE is recovered by the 11th day of storage, and 90 percent is recovered by the 28th day of storage.

Room Temperature/Freezer Comparative Storage Study

The results of the room temperature storage study indicated that the MTBE recovery was essentially 100 percent. No loss of MTBE at the 415-μg level was observed within the 5 days of room temperature storage. Also no breakthrough at the 415-μg MTBE level occurred at 100 cc/min flow rate for 6 hours (36 L air).

Breakthrough-Volume Study

The results of the breakthrough-volume study concluded that at 100 cc/min the loadings of 10 and 20 mg MTBE in gasoline matrix did not break through after 30 minutes. The 10 mg MTBE in gasoline spike showed no breakthrough even at 200 cc/min for 6.5 hours (77.7 L air sampled). In both experiments only the very light hydrocarbons (i.e., isobutane, *n*-butane, and isopentane), usually not retained by charcoal, indicated breakthrough. Therefore, it was determined that the use of the 400-mg charcoal tube for air sampling MTBE/gasoline vapors would be acceptable for collecting 2-mg gasoline vapor samples without MTBE breakthrough.

Field Application

Results of the NIOSH field surveys conducted using this method are listed in Table III. Details of these industrial hygiene surveys were presented at a symposium.(7) The 27 Cincinnati gasoline samples indicated MTBE (vol/vol) concentrations from nondetectable to 0.18 percent, and a mean MTBE personal exposure of less than 0.16 ppm (N = 32 charcoal tubes). The 15 Phoenix gasoline samples indicated 12.4 to 13.2 percent (vol/vol) MTBE, and a mean MTBE personal exposure of 0.32 ppm (N = 41 charcoal tubes). The 33 Los Angeles gasoline samples indicated 0.03 to 2.1 percent (vol/vol) MTBE, and a mean MTBE personal exposure of 0.14 ppm (N = 48 charcoal tubes). No breakthrough was indicated from analyses of the back-up charcoal tubes except for light hydrocarbons. The highest MTBE exposure was 3.88 ppm or 1.2 mg/tube in a 100-L air sample. (Benzene and total hydrocarbon exposures were determined in the three-city surveys; those data are reported elsewhere.)

Conclusions

In summary, MTBE can be collected from air containing gasoline vapors using tandem 400/200-mg charcoal tubes

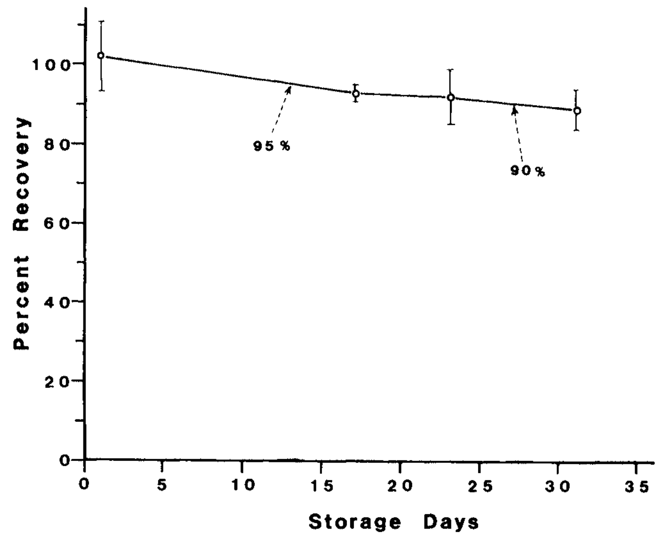

FIGURE 2. Thirty-one-day MTBE charcoal tube storage stability study (−7°C).

TABLE III. Field Study Results*

Location	Gasolines (N)[A]	MTBE in Gasolines (%)	Personal Samples (N)	Personal Exposures (ppm MTBE)
Cincinnati	27	ND[B]-0.18	32	ND[C]-0.16
Phoenix	15	12.4-13.2	41	ND[C]-3.88
Los Angeles	33	0.03-2.10	48	ND[C]-0.73

*Benzene personal exposures in three cities were below 0.07 ppm.[7]

[A] N = number of samples collected.

[B] ND = <0.002 percent MTBE.

[C] ND = <0.03 ppm MTBE.

operating at a flow rate of 100 to 500 cc/min. MTBE collected on charcoal tube samples is stable at ambient temperature for 5 days. The 400/200-mg charcoal tube capacity is adequate for sampling MTBE. When samples are stored in the freezer at −7 °C, 95 percent recovery is achieved after 12 days, and 90 percent recovery after 27 days. Gas chromatographic analysis of carbon disulfide-desorbed samples takes less than 30 minutes per sample. Complete separation of MTBE, along with methanol, ethanol, butanes, pentanes, hexane, benzene, toluene, and xylenes is accomplished using the developed chromatographic conditions. Total hydrocarbon personal exposure can be determined from the same personal sample. The mean precision of the calibration standards in the 9- to 426-μg/ml range is less than 5 percent. The precision for synthetic samples is less than 9 percent. The method was published in the NIOSH *Manual of Analytical Methods* as Method 1615.(8,9) Field applications of the method indicate that it is valid for the analysis of MTBE in a matrix of either gasoline vapor or liquid gasoline.

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Disclaimer

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