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AN EVALUATION OF FOUR FIELD SCREENING TECHNIQUES FOR MEASUREMENT OF BTEX

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The objective of this research effort was a laboratory investigation of field-portable technology to demonstrate that available analytical methods for volatile aromatic hydrocarbons can produce data of known quality in a timely, cost-effective manner. Based on the results of a literature search, which was made to identify current field analytical techniques for the detection of benzene, toluene, ethyl benzene, and xylene (BTEX) in environmental samples, four promising technologies were selected for laboratory evaluation. No single field methodology was found to be superior to others; each method has advantages and disadvantages. Guidelines for choosing proper analytical methods for specific problems was provided. Also presented was method performance along with advantages and limitations for the procedures investigated.

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EXECUTIVE SUMMARY

A laboratory investigation of field-portable technology demonstrated that available analytical methods for volatile aromatic hydrocarbons can produce data of known quality in a timely, cost effective manner. Based on the results of a literature search, which was made to identify current field analytical techniques for the detection of benzene, toluene, ethyl benzene, and xylene (BTEX) in environmental samples, four promising technologies were selected for laboratory evaluation. No single field methodology was found to be superior to others; each method has advantages and disadvantages.

The Antox immunoassay test is simple to perform and can be used as a quick indicator of BTEX contamination in water. This test provides a reliable, qualitative indicator for BTEX at levels above 75 parts per billion (ppb). Although the manufacturer claims sensitivity to 25 ppb, 75 ppb appears to be a more practical method detection limit.

Detector tubes are simple to use and can provide a semi-quantitative determination for BTEX in water. The lower detection level is 500 ppb.

The Lab In A Bag sample extraction system provides a reliable means to prepare water and soil samples for volatile hydrocarbon analysis. Low detection limits (10 ppb in water and 40 ppb in soil) are achievable when used with a portable gas chromatograph.

The Microsensor Systems, Inc. (MSI) gas chromatograph provides accurate and precise quantitation for BTEX. This instrument offers the advantage of providing quantitation for individual target analytes.

All the methods investigated can be used with few or no modifications. This document provides guidelines for choosing proper analytical methods for specific problems. Method performance is presented along with advantages and limitations for the procedures investigated.

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PREFACE

The research described in this report was carried out by Lockheed Environmental Systems & Technologies Company, Las Vegas, Nevada, under Contract Number 68-CO-0049 with the U.S. Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Las Vegas, Nevada.

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This report has been reviewed by the Public Affairs Office and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.

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LIST OF ABBREVIATIONS

AC	Alternating Current
BTEX	Benzene, Toluene, Ethylbenzene, Xylene
BTX	Benzene, Toluene, Xylene
CF	Calibration Factor
cc	Cubic Centimeter
cm	Centimeter
cm^3/min	Cubic Centimeters Per Minute
°C	Degrees Celsius
DC	Direct Current
EMSL-LV	Environmental Monitoring Systems Laboratory-Las Vegas, Nevada
EPA	United States Environmental Protection Agency
FID	Flame Ionization Detector
GC	Gas Chromatograph
µ	Micron
MDL	Minimum Detection Limit
MSI	Microsensor Systems, Inc.
mg/L	Milligram Per Liter
mL	Milliliter
nm	Nanometer
%RSD	Percent Relative Standard Deviation
PID	Photoionization Detector
ppb	Parts Per Billion
ppm	Parts Per Million
QA	Quality Assurance
S/R Ratio	Sample/Reference Ratio
SAW	Surface Acoustical Wave
SITE	EPA Superfund Innovative Technology Evaluation Program
TOVD	Total Organic Vapor Detector
TRIS	Tricyanoethoxypropane
VOA	Volatile Organic Analysis
VOC	Volatile Organic Compound

SECTION I

INTRODUCTION

A. OBJECTIVE

The purpose of this study was to select, then evaluate, field methods for detecting and measuring benzene, toluene, ethylbenzene, and xylene (BTEX) in soil, water, and gas samples.

For each field method selected for this study, accuracy and precision were compared to standard laboratory methods. Methods were also evaluated based on ease of use, cost per sample and/or cost of equipment, and minimum detection limits (MDLs). This report includes detailed descriptions of each method (Section II), step-by-step descriptions of method procedures (Section III), a discussion of method performance (Section IV), conclusions (Section V), recommendations (Section VI), and a reference list (Section VII).

B. BACKGROUND

Aromatic hydrocarbons such as BTEX are common contaminants at military installations due to spillage of hydrocarbon fuels and leakage of storage tanks. Cleanup of these contaminated areas requires numerous chemical analyses for site characterization and remediation monitoring. The most commonly used traditional techniques for measuring volatile compounds involve the collection of field samples for shipment to an analytical laboratory. This process is time consuming and costly. In addition, sample handling and transport increase the potential for error, especially for volatile organic compounds (VOCs), which are easily lost during each manipulation of the sample. Field screening and field analytical methods are faster and potentially more precise than traditional techniques for gathering data to evaluate remediation efforts. Using state-of-the-science field analytical techniques, remediation efforts can be monitored for a fraction of the cost of traditional laboratory-based analytical techniques.

Many field analytical instruments are commercially available. However, reliable performance data are lacking for many of these instruments. Available performance data are usually provided by the manufacturer and support the manufacturer's claims about the device. These claims are sometimes unrealistic. Many of these instruments and the data they produce have not been thoroughly examined by the scientific community.

Total Organic Vapor Detectors (TOVDs) are commonly used for field analysis. A number of hand-held portable detectors are available for the detection of BTEX vapors. Most of these units use either a photoionization detector (PID) or a flame ionization detector (FID). The PID is quite sensitive to aromatic hydrocarbons, but does not respond to light hydrocarbons such as methane. The FID, although not as sensitive as the PID, is useful for applications in which measurement of a broader range of analytes is desired. Gas Chromatography (GC) has also been extensively used for field analysis of BTEX, and a number of field-portable GC units

are available from various manufacturers. Chromatography offers the advantage of separating and quantitating individual compounds. The TOVD and GC can be used to analyze water or soil samples if a preparation procedure, such as a headspace technique, is used to transfer the volatile components to the vapor phase.

Other methods are available for field BTEX detection. Many of these techniques are established analytical methods recently adapted for field BTEX detection. For example, immunoassay techniques, commonly used by medical technologists to monitor drug levels in clinical patients, have recently been commercially introduced for analysis of environmental samples (1). Detector tubes have been used for years to monitor ambient air for industrial hygiene. A simple extraction apparatus allows detector tubes to be used for analysis of water samples (2,3,4). These methods represent practical alternatives to more expensive laboratory methods for analysis of environmental samples.

C. SCOPE/APPROACH

The first phase of the study involved identifying field analytical methods currently used and available for investigators. Only methods applicable to BTEX were considered. A computer literature search was conducted to gather information on the full scope of techniques now used for BTEX analysis. Methods in the research phase and not ready for routine monitoring activities, such as fiber optic chemical sensors, were not considered for this evaluation. Commercial vendors were identified by scanning advertisements in trade journals and by contacting field investigators. Product literature and specifications were obtained from vendors and reviewed for matrix applicability and MDLs. A summary of currently available commercial methods, along with manufacturer's specifications, is presented in Table 1.

Data generated by use of field screening methods were compared to laboratory data obtained by using GC with a PID according to the U.S. Environmental Protection Agency (EPA) Method 8020 (5). All analytical techniques were conducted within standard quality assurance (QA) guidelines.

A common problem in evaluating field detection methods is the natural variation in contaminated sites. A single location can yield a wide variety of soil types and characteristics that create a masking variation in samples used in the evaluation of a method. To better determine and minimize matrix variability, some test procedures were performed on reconstituted soil columns and in water matrices that were prepared in the laboratory. By using these relatively homogenous test media, the investigator can usually determine subtle characteristics of a method without the confounding influence of field variation.

TABLE 1. FIELD ANALYTICAL METHODS REVIEWED FOR DETECTING AND QUANTITATING BTEX

Method	Matrix	Minimum Detection Limits (ppb)
Total Organic Vapor Detector	Vapor	1,000
	Water*	1,000
	Soil*	5,000
Field Gas Chromatography	Vapor	5-500
	Soil*	50
	Water*	10
Immunoassay	Water	25
Detector Tubes	Vapor	5,000
	Water*	1,000
Handby Procedure	Water	50
	Soil	500
Thin Layer Chromatography	Water	2,000
	Soil	5,000
Photoacoustic Spectroscopy	Vapor	40
Underground Storage Tank Sensors	Vapor	10,000
UV-Visible Spectroscopy	Water	500

Abbreviations: ppb = parts per billion.

* Matrix can be analyzed if sample preparation techniques are employed such as: static headspace, dynamic headspace, thermal desorption, purge and trap, and solvent extraction.

D. METHOD SELECTION

Methods were selected for evaluation if (1) the required equipment and supplies were commercially available, (2) the procedure was cost effective, and (3) published performance evaluations of the methodology were limited. In addition, an attempt was made to select a variety of methods, ranging from simple, where ease of use is a more important criterion than precision and accuracy, to more complex methods requiring some skill in instrumentation.

Methods that can be used by a wide range of field investigators have greater utility than techniques requiring personnel with advanced, specialized knowledge of chemistry or electronics. For the purposes of this study, the ideal field method has a simple, easy to understand procedure, has few mechanical or electronic components that can malfunction, is easy to calibrate, is specific and sensitive to BTEX, is easy to operate, and is cost effective. Based on these criteria, four methods were selected for evaluation: the Antox BTX water screen kit, detector tubes, the Lab In A Bag, and the MSI-301A Organic Vapor Monitor. These methods are described in Sections II and III.

SECTION II

METHOD DESCRIPTIONS

The field methods selected for this study are simpler, faster, and more economical than conventional laboratory methods. Field screening methods can provide reliable analytical results for a wide range of applications, including site characterization, remediation monitoring, and leak detection. However, no single field method is best for all applications. The strengths and limitations of each technique are discussed in this section. The costs of the selected methods varied considerably and can be presented in a variety of ways. Information is presented which will allow evaluation of all methods on a cost per sample basis if analyst and standards costs are factored in. The major cost for Antox and detector tubes is the kits, while the major cost for the MSI organic vapor monitor and the Lab In A Bag is the equipment. The method descriptions presented below are in order of increasing complexity and cost.

A. ANTOX

The Antox BTX Water Screen is an immunoassay for the detection of benzene, toluene, and xylene (BTX) in water samples. The immunoassay kit contains reagents in dropper bottles with special cuvettes (Figure 1).

1. Theory of Operation

The Antox BTX Water screen kit uses a test tube (cuvette) coated with rabbit polyclonal antibodies that bind to and hold BTX compounds or other closely related hydrocarbons. BTX or similar compounds in the sample will compete with the enzyme-activated analog of the analyte for binding to the immobilized antibody: the more BTX in a sample, the less enzyme.

Substrate and chromogen are added to the cuvette and turn color in the presence of the enzyme. This color, a pale yellow, can be measured on a photometer. The amount of BTX in the sample is inversely proportional to the color development. By comparing each sample to a reference sample of pure water, the relative concentration of BTX or similar compounds in the sample can be determined.

2. Characteristics and Costs

The Antox procedure is useful for performing quick screening tests on water samples for the determination of the presence or absence of BTX. The main advantage of the test is its ease of use. Inexperienced operators should be able to learn the procedure with some training from an experienced operator. The colorimetric nature of the test assures the operator that the procedure is working properly, with only a minimum of quality control checks. As

Figure 1. Antox Immunoassay BTX Kit with Special Cuvettes and Reagent Bottles.

many as four water samples can be analyzed during the 30 minutes required to complete the analysis. A single operator can comfortably process 64 samples in an 8-hour day. At present, the test can reliably determine if BTX is present above 75 ppb. Future immunoassay kits may achieve a lower detection limit. The cost of the equipment for this test is approximately $600.00. The cost per sample for the test kits depends on the level of quality control, and can be as low as $9.15 if four samples are analyzed with each control tube.

3. Limitations

The primary limitation of the test is a lack of quantitative information; the test can only indicate if a contaminant is present, not precisely how much or exactly what compounds are present. The test responds to aromatic hydrocarbons and other compounds containing carbon-carbon double bonds, such as trichloroethylene and tetrachloroethylene. As a result, a positive test does not necessarily prove the presence of BTX; rather, it indicates that some type of contamination is present. For this reason, the test is most appropriate for screening or monitoring of BTX at sites known to be contaminated with BTX.

4. Conclusions

Despite its limitations, the Antox procedure should be useful for many applications. The presence of a contamination problem can be quickly determined at a field site. Screening many samples at the field site can provide a cost-effective guideline for the selection of samples to be sent for complete laboratory analysis. In addition, quick screening results can be used to assist a laboratory in determining an appropriate dilution for a sample.

B. DETECTOR TUBES

Detector tubes are used to measure the concentration of a specific compound or group of compounds in a gas sample. In the presence of the target compounds, the material in the tubes gradually changes color or stains along the length of the tube. The longer the stain, the higher the concentration of contaminant. Detector tubes have been used for years by industrial hygienists for the analysis of ambient air. Using simple apparatus (Figure 2), these tubes can also be used for analyzing water samples. The detector tubes used for this study were manufactured by Drägerwerk AG Lübeck (Germany) and obtained from SKC WEST (parts # 800-28561 and # 800-23001).

1. Theory of Operation

Contaminant-bearing air is produced by bubbling scrubbed ambient air through a water sample. Ambient air is pumped through a charcoal-filled scrubbing tube into the water sample in a gas-washing column. The air then passes through a detector tube attached to the outflow of the gas-washing column (See Figure 2). Chemicals in the detector tube react with

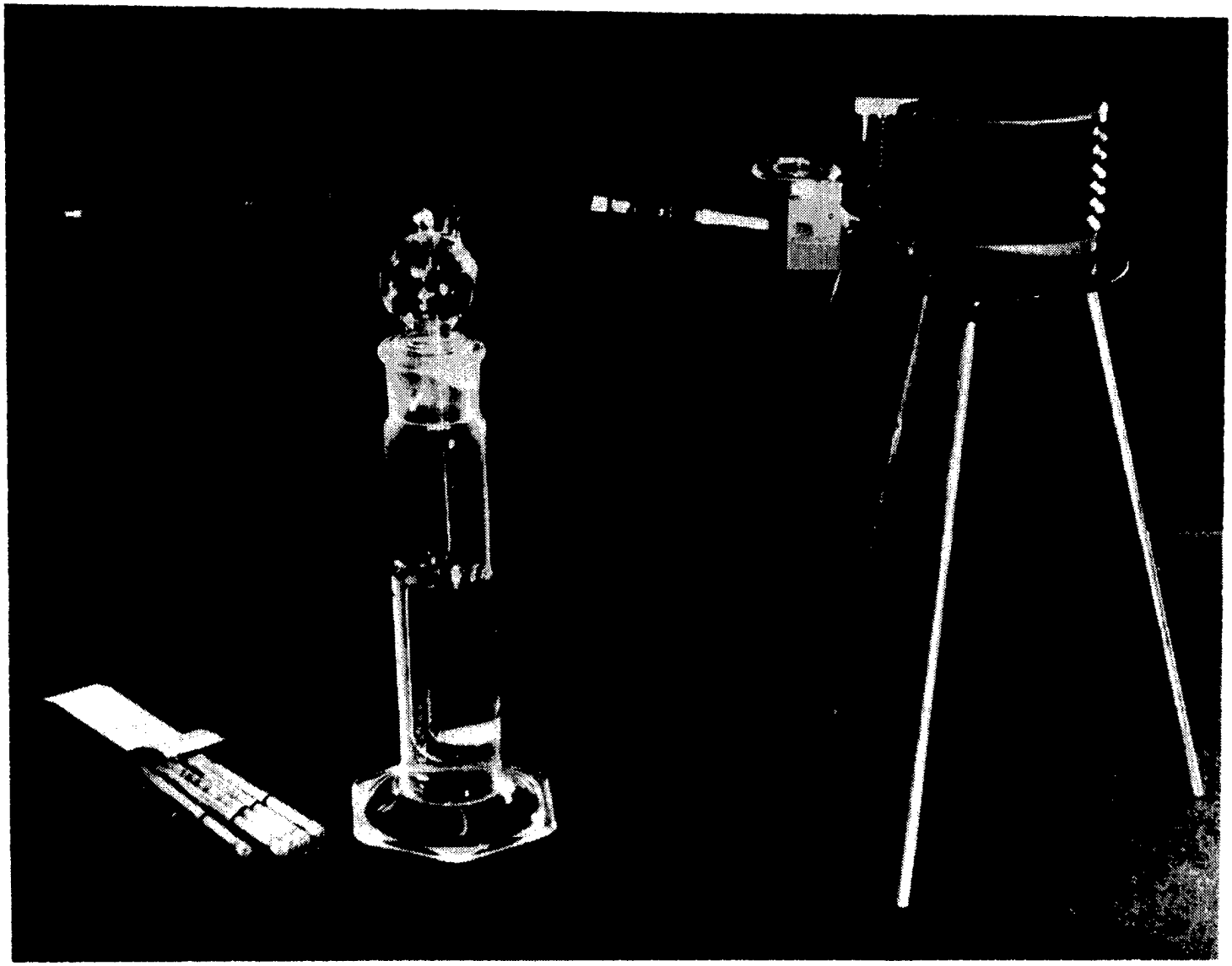


Figure 2. Detector Tubes with Charcoal Scrubbing Tube, Gas Washing Column, Detection Tube, and Air Pump.

a specific compound or group of compounds. This reaction causes the chemicals to change color gradually from one end of the tube to the other. The length of the stain is compared to the calibration markings on the tube to determine the approximate concentration of BTEX in the sample.

2. Characteristics and Costs

Detector tubes provide a quick and simple screening procedure for detecting volatile organic compounds (VOCs) in water samples. The method can detect BTEX down to 0.5 mg/L (parts per million; ppm) with a relative standard deviation of ± 20 to 30 percent. The procedure is easy to learn and uses simple glassware apparatus. Since the tubes are precalibrated for air analysis, a calibration curve must be prepared for comparing the detector tube reading with the concentration of analyte in the water. A single operator can perform each test in approximately 5 minutes; therefore, an operator can be expected to analyze approximately 100 samples in a standard working day. The equipment required to run the test costs $890.00. The cost per sample is approximately $20.

3. Limitations

The primary limitations of this method are potential interferences and temperature effects. Compounds chemically similar to the component being tested can produce a color change in the tube. The main interferences are other aromatic hydrocarbons. The tubes are calibrated for one specific target analyte, although other aromatic hydrocarbons will also give a positive indication. For example, a tube calibrated for toluene will respond to other volatile aromatic hydrocarbons, such as xylene. Therefore, this method will provide an overall indication of aromatic hydrocarbon contamination instead of levels for one specific analyte. This is a not a problem for screening contaminated fuel sites, when the objective is to characterize the site for fuel contamination. In addition, the temperature of the water sample can have a significant effect on the test results.

4. Conclusions

Detector tubes achieve a higher degree of quantification than the Antox method. The operator must calibrate the method at the same temperature that the test will be run for optimum results. Although this colorimetric method is not as sensitive or specific as instrumental analytical methods, the simplicity of operation is a real advantage for field screening projects. The supplies can be easily carried onto a site, with no electrical power required. The procedure is easy to understand and learn for field technicians.

C. LAB IN A BAG

The Lab In A Bag is a sample preparation system for analyzing VOCs in the air (headspace) above soil or water samples (Figure 3). This method provides controlled conditions for field analysis without the need for additional equipment or supplies. The battery-powered unit and required

supplies are included in a carrying case that allows easy transportation to a field site. The Lab In A Bag is a commercial analytical version of a Polyethylene Bag Sampling System (5,6).

1. Theory of Operation

The instrument inflates a 1-quart polyethylene bag, containing the sample, to a specific volume with scrubbed air; the instrument then agitates the sample for a preset period (between one to eleven minutes) to allow for the release of VOCs from the sample into the headspace in the bag. A valve attached to the bag allows the headspace to be analyzed by a portable TOVD or by a field-portable GC. The concentration of VOCs measured in the headspace is proportional to the concentration of VOCs in the sample. The field-portable GC used in these evaluations was the MSI-301A (see Subsection D).

2. Characteristics and Costs

The instrumentation provides a precise procedure for headspace analysis in the field. The kit is lightweight, providing easy mobility to a field site. Suitable precision and accuracy can be achieved for measuring BTEX in water samples when good operating techniques are used. The performance for soil samples is not as good as for water due to soil matrix effects such as irreversible binding, but the achievable data is suitable for many applications. The method is simple to perform and includes an easy to understand operating manual. The total cost of the equipment for Lab In A Bag is approximately $2,000.

3. Limitations

This method requires an instrument for measuring the concentration of analyte in the headspace of the bag; therefore, it requires two calibration steps: one for the detection instrument and one to correlate the headspace concentration to the concentration of analyte in the matrix. Although a calibration curve can be prepared in a laboratory prior to analyzing samples in the field, it is best to perform calibration in the field because field conditions can affect the response of the instrument. The Lab In A Bag has no temperature control, so the method should be calibrated at the same temperature as field measurements will be taken.

4. Conclusions

This device provides a simple, portable sample preparation capability for headspace analysis. Headspace techniques are commonly performed in field screening studies, but usually with little quality control. Typically, an organic vapor meter is waved over a sample in whatever container (if any) is available. Lab In A Bag provides controlled conditions for headspace analysis without the cost of laboratory-grade instrumentation. When used in conjunction with an appropriate detector, water and soil samples can be analyzed at the low ppb levels.

D. MSI-301A ORGANIC VAPOR MONITOR

The MSI-301A Organic Vapor Monitor (Figure 3) is a field-portable, commercially available GC for the analysis of specific VOCs. This model is designed to detect BTEX. The instrument provides controlled conditions for field analysis of soil or water sample headspace or soil gas without requiring any additional equipment or supplies. The unit can be operated on alternating current (AC) or battery (DC) power and uses scrubbed ambient air as carrier gas.

1. Theory of Operation

A gas sample is injected into the GC. The sample passes through a heated column, separating volatile compounds according to molecular weight: the lightest VOCs move the fastest and reach the detector first. The vapors are detected at the output of the column using a solid-state surface acoustical wave (SAW) detector. Each vapor is identified by its retention time (length of time required to travel through the column) compared to the retention times of known standards of the same compounds. The quantity of vapor is proportional to the signal produced by the detector.

2. Characteristics and Cost

This GC can provide reliable results for the analysis of BTEX in a field situation. The GC uses ambient air for carrier gas, providing an advantage over field-portable GCs that require compressed gas cylinders for operation. The MSI-GC operates on a 12-volt source (either a car battery or a rechargeable battery pack), as well as on a 120-volt source. The laboratory-grade instrument is easy to operate. The operating conditions, such as carrier flow rate and column temperature, are pre-set and optimized for the analysis of BTEX. The operation is menu driven, directing the operator in a step-wise fashion toward either instrument calibration or sample analysis. An operator with little or no GC experience can analyze samples without extensive training. When operated in conjunction with Lab In A Bag, a single operator can analyze 6-8 samples per hour or 48-64 samples in a standard working day. The instrument costs approximately $9,000.

3. Limitations

Because the system is specifically designed to detect BTEX, it lacks the versatility of other GCs, which can be set up to analyze other compounds. Only vapor samples can be injected into the instrument; water or soil samples require a sample preparation method, such as Lab In A Bag.

4. Conclusions

This instrument offers the advantage of GC separations for BTEX using ambient air as a carrier gas. The primary advantage of this instrument is its portability and ease of use.

This instrument, when used in conjunction with a sample preparation technique such as Lab In A Bag, provides precise, quantitative capabilities for measuring BTEX in the field. Compounds chemically similar to BTEX may interfere with the analysis; therefore, the instrument is best used at sites where BTEX is the known contaminant.

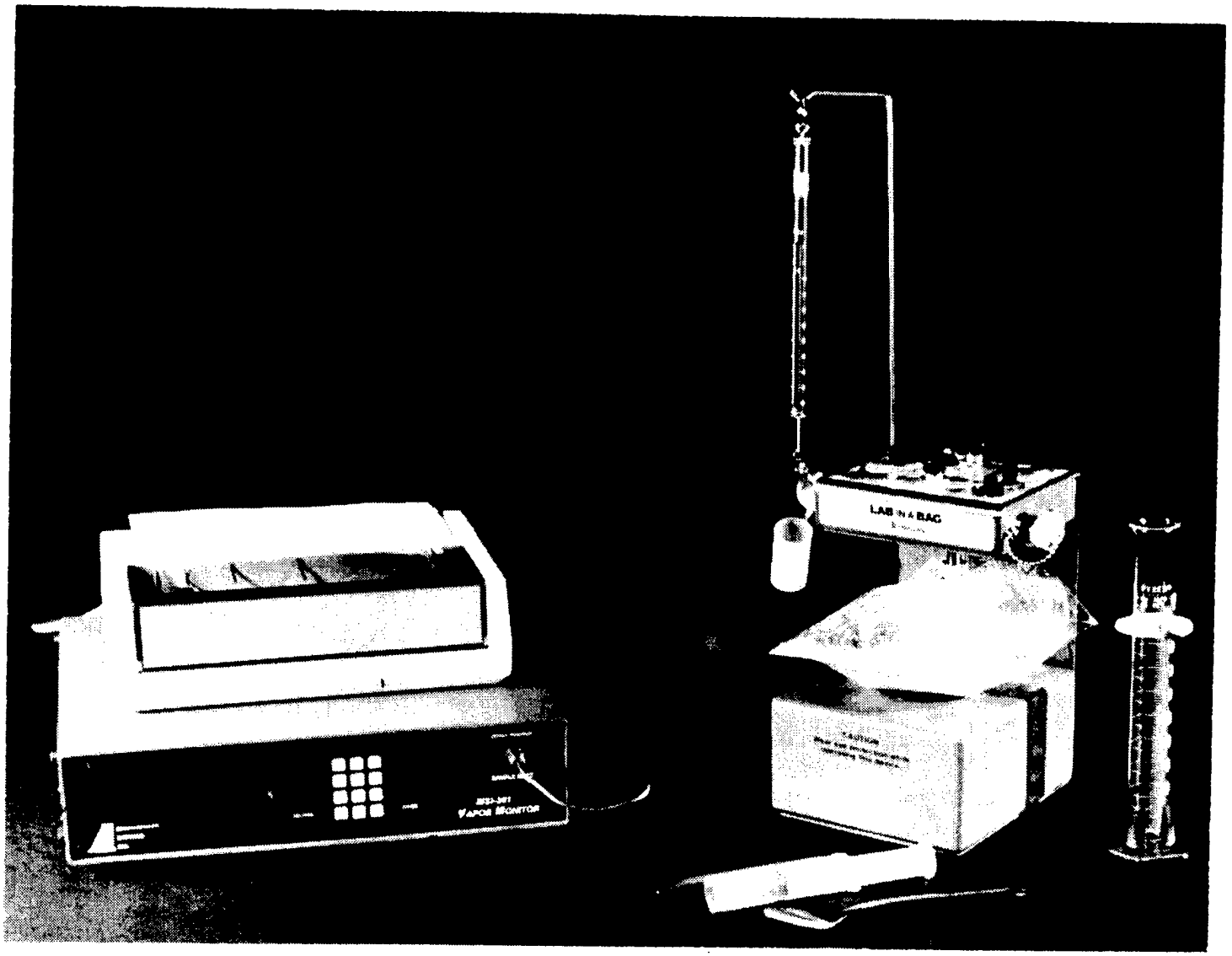


Figure 3. Lab In A Bag and MSI-301A Portable Gas Chromatograph Systems.

SECTION III

METHOD PROCEDURES

This section provides a description of each method procedure. A guideline to the materials required for each procedure is presented, along with step-by-step procedures.

A. ANTOX IMMUNOASSAY

1. Materials

The Antox immunoassay kit includes reagents and cuvette test tubes required to perform the analysis. Five different reagents are included with the kit, each with color coded caps. A spectrophotometer is required to measure absorbance. The spectrophotometer must have a cell path of 1 cm, and must be capable of reading absorbance at 450 nm. Several models of field-portable, battery-powered units are available, and one may be purchased from Antox. Other materials required to perform this procedure, but not included in the kit include:

- Volatile organic analysis (VOA) sampling vials
- Distilled water
- 200-mL liquid dispenser
- Quality control samples
- Minute timer
- 4-mL transfer pipettes
- Ice chest
- Cuvette holder

Test kit reagents should be stored at 2° to 8°C when not in use, except for the color developer #2 (blue cap), which should be stored at room temperature. Before performing the test, the color developer #1 (black cap) and the bagged cuvettes should be allowed to come to ambient temperatures. The other reagents, including distilled water, are kept refrigerated or placed in an ice chest cooled to near 4°C.

2. Procedure

For each set of sample analyses, a reference standard (blank distilled water) is analyzed with the samples. As many as four samples can be analyzed, along with one reference standard. Each reagent should be added to the reference cuvette, then the sample cuvette(s). The elapsed time between adding reagents to each cuvette in a batch should be kept at a minimum to avoid a variation in color development due to incubation times.

The antibody-coated cuvettes are labeled "R" for the reference and an appropriate code (such as sample #1, sample #2, etc.) for the samples, and are set into a cuvette holder. A

disposable syringe or transfer pipette is used to measure and dispense 4.0 mL of distilled water into the "R" cuvette. The same technique is used to measure 4.0 mL of the sample into the appropriately labeled cuvette. A clean pipette should be used for each sample to avoid cross-contamination.

Add 4 drops of the buffer solution (gray cap) to all cuvettes. Immediately add 4 drops of the enzyme solution (red cap) to all cuvettes. Cap and invert cuvettes four times to evenly distribute and mix samples. Incubate the tubes for 10 minutes in an upright position. Using distilled water, wash and decant the cuvettes four times, discarding the contents of the cuvette after each wash. Add 4 drops of color developer 1 (black cap) to each cuvette. Add 4 drops color developer 2 (blue cap) to each cuvette. Tap cuvettes gently to assure all solution is in the bottom of the cuvettes. Allow tubes to sit for 5 minutes. Add 4 drops of the terminating solution (purple cap) to each cuvette. Mix by swirling gently. The absorbance of each sample cuvette and the reference cuvette is measured by the spectrophotometer at a wavelength setting of 450 nm.

The absorbance of the sample is divided by the absorbance of the reference. If the result is less than 0.85, the test is positive. For example, if the absorbance reading of the reference is 1.0 and the absorbance reading of the sample is 0.5, the ratio is 0.5: the test is positive.

3. Calibration

The spectrophotometer must be properly calibrated to avoid false positive or false negative results. The exact calibration procedure depends on the specific model of instrument; however, this is a simple procedure for most field-portable units. A two-point calibration is required. A zero point is set when no light is transmitted, and a 100% transmittance point is set using pure water.

B. DETECTOR TUBES FOR ANALYSIS OF WATER SAMPLES

1. Materials

Detector tubes are clear glass tubes filled with chemical reagents that react with a specific compound or class of compounds. The tubes contain a scaled indicating section that changes colors in the presence of the target compound. The length of the color change is proportional to the concentration of the analyte.

The equipment required to perform this test is supplied in a kit available from National Draeger, Inc. The tubes must be purchased separately. The equipment can also be purchased separately, with the items specified as follow:

- 250-mL gas washing bottle with a frit porosity of 70-100μ.
- Hand-operated bellows pump. The pump should be the model specified by the manufacturer of the detector tubes.

- Thin-walled glass tube, containing activated charcoal to purify the inlet air.

- Thermometer to measure the ambient air and water sample temperatures.

A number of detector tubes for BTEX are commercially available. The detector tubes used for this study were manufactured by Drägerwerk AG Lübeck (Germany) and were obtained from SKC WEST (parts # 800-28561 and # 800-23001). Pure distilled water is required for method blanks and calibration samples.

2. Procedure

The basic principle of this method is the volatilization of the target analyte from its aqueous solution by means of air purged through the sample, and simultaneous analysis of the air/analyte mixture by a suitable detector tube.

First, a tube with activated charcoal is attached to the inlet port of the gas washing bottle. The detector tubes are labeled by the manufacturer with an arrow indicating the direction of the air flow. The end of the detector tube in the opposite direction of the arrow is opened and attached to the outlet port of the gas washing bottle. The aqueous sample is then slowly poured into the gas washing bottle to the 200-mL mark. The bottle is closed immediately after adding the sample to avoid undue loss of volatile analyte. The end of the detector tube in the direction of the arrow is opened and attached to the bellows pump. The pump is firmly squeezed the required number of strokes (as specified by the manufacturer) for the particular detector tube. The concentration is read as the length of color change on the detector tube scale.

3. Calibration

A calibration curve is required to determine the concentration of the target analyte in water because the "ppm" scale on the detector tube measures concentration of the compound in air, not water. A calibration curve is prepared for each individual gas washing bottle and for each analyte with calibration standards at a minimum of three concentration levels. The calibration standards are made up of water spiked with known amounts of the analyte. One of these external standards should be near, but above, the method detection limit for the particular tube/analyte combination. The concentration of the other standards should be prepared to correspond to the expected range of concentrations of the analyte found in the samples.

For each calibration standard, the ratio of concentration of analyte in the water to the reading from the detector tubes is defined as the calibration factor (CF).

$$\text{Calibration Factor} = \frac{\text{Spike Concentration in Water } (\mu g/mL)}{\text{Detector Tube Reading}}$$

A daily calibration check is performed by analyzing a standard at a mid-range concentration prior to the first and following the last sample of the day, whenever operating conditions change, or whenever a change in detector tube performance is suspected.

C. LAB IN A BAG

The Lab In A Bag sample preparation procedure requires a detector to determine the concentration of the compounds of interest. When used with a portable GC, such as the MSI-301A, analytical results approach the precision and accuracy of laboratory methods and instruments. This section covers the procedure for the Lab In A Bag, not the detection system.

1. Materials

The Lab In A Bag comes as a kit that includes many of the supplies required to perform the sample preparation procedure. The kit includes a spring scale for weighing soil samples, a graduated cylinder for measuring water samples, a micro-dispenser and glassware for preparing standards, and miscellaneous items for collecting and analyzing the samples. The instrument and the kit are well packaged in a rugged, air-tight carrying case suitable for shipping to remote sites. The instrument operates on rechargeable batteries.

Other equipment and materials required to perform this procedure, but not included in the kit, are listed below:

- An organic vapor detector such as a portable GC, an FID, or a PID. This detector must be connected to the Lab In A Bag with a connector tube.
- One-quart heavy-duty polyethylene freezer bags with zipper-type closures.
- Paper towels
- Distilled water

2. Procedure

Use a 1-quart polyethylene freezer bag to analyze both soil and water samples. Attach the bag to the instrument through a hole cut in the bag using the template and hole cutter

provided with the instrument. Slip the bag onto a brass fitting on the instrument. A knurled nut with a gasket on the brass fitting is tightened to provide the bag with an air-tight seal.

First inflate the bag while empty to pressure test the bag to ensure it does not leak, then flush the bag and instrument of any residual contaminants. Inflate the bag as follows:

a. Zip the bag closed.

b. Set the instrument controls as follows:
   (1) Turn the toggle switch to ON.
   (2) Turn the upper valve to VENT TO ATMOS.
   (3) Turn the lower valve to PURGE OR FILL.

c. Push the FILL button and release. A red pilot lamp will light while the bag is filling. This red light will turn off when the bag is sufficiently filled. If the bag will not inflate within 1 minute, there is probably a leak in the bag. Reseat the bag by removing it from the brass fitting and reattach it. Open the bag and zip it closed carefully. If the bag still will not inflate, it should be discarded.

If the bag properly seals, the instrument is ready to analyze a sample. Reopen the bag and quickly place the sample and a magnetic stir bar into the bag. For a water sample, use 100 ml. For a soil sample, place 25 grams into the bag along with 100 ml of deionized water. Zip the bag to close immediately after adding the sample and stir bar. Fill the bag with air by pushing the STIR button. Care must be taken to assure the stir bar is properly centered on the magnetic stir plate, or the bar will not spin properly. Some manual manipulation for proper placement may be necessary. The stir time and speed can be optimized for a particular sample type by using the optimization procedure in the instruction manual. For most applications, a stir time of 5 minutes is sufficient.

At the end of the stir cycle, a beeper sounds and a green light flashes. Turn the toggle switch to OFF. Immediately turn the upper valve to SAMPLE to allow the headspace air to be sampled and analyzed by the GC or TOVD.

3. Calibration

Calibrate the detector before calibrating the Lab In A Bag. The procedure for calibrating the MSI (which was used in this analysis) is described in Subsection D-3.

The Lab In A Bag procedure is calibrated with external standards. A series of standards at several concentrations are prepared in a matrix similar to that of the samples and analyzed using the routine procedure for sample treatment. A calibration curve is prepared by comparing the response of the detector with the concentration of the spiked standards.

D. MSI-301A ORGANIC VAPOR MONITOR

The MSI-301A Organic Vapor Monitor is a field-portable, commercially available GC designed for the analysis of specific VOCs. This model is set up for the detection of BTEX. The unit can be operated on AC power or battery power. Scrubbed ambient air is used as the carrier gas.

1. Materials

The MSI-301A is a field-portable GC using a solid-state proprietary surface acoustical wave (SAW) type detector. The carrier gas is ambient air passed through a charcoal scrubber. The column is 1/8 of an inch by 43 inch 10 percent tricyanoethoxypropane ("TRIS") on 80/100 mesh Supelcoport. The column is kept at an isothermal 65°C. The carrier flow rate is about 30 cm^3/minute. The gas sample is first concentrated by the instrument using a Tenax trap. After absorbing onto the Tenax at ambient temperature, the trap is heated to 140°C, desorbing the analytes onto the column. These parameters cannot be changed by the operator. A charcoal filter that attaches to the inlet port is supplied with the instrument for analyzing system blanks. The GC is calibrated using gas standards, which can be commercially obtained. Analytical results are stored on an internal data logger, which can be downloaded onto a serial printer or a computer.

2. Warm-Up and Blank Measurement

The GC must be allowed to warm-up for 30 minutes before operating to permit time for the column oven to achieve the proper operating temperature. Before analyzing any samples, an instrument blank and calibration standard are analyzed to insure that the instrument is operating correctly. The instrument blank is analyzed by attaching the charcoal scrubber to the inlet of the instrument and pressing "1=RUN" on the instrument keyboard. A system menu will guide the operator with specific options, such as report type. Reports can be simple, including only the name of the compound with the concentration, or more detailed with retention times and peak areas included. The analysis takes about 6 minutes for a complete run. The results are displayed on the front instrument display or on a printer, if attached. The blank sample should be zero or less than the MDLs. Carryover from previous samples may prevent zero readings for the blank. For very low detection limits (< 10 ppb), two blank runs are generally required to flush the system of any trace amounts of BTEX normally present in the atmosphere.

3. Calibration

After a blank has been successfully analyzed, a calibration standard is run by pressing "2=CALIB" on the keyboard. The menu will prompt the user to enter the concentration of the standard calibration gas and to connect the gas standard to the instrument. After the calibration gas has been analyzed, the operator is prompted with new calibration factors and instructed whether or not to enter the new factors into the instrument. If the new factors vary significantly from the previous factors (i.e., a difference of greater than 25 percent), this may be an indication of a problem.

In this case, the calibration standard should be reanalyzed. When duplicate calibration standards produce response factors within 10 percent of each other, the system is ready to analyze samples.

4. Operation

Once the instrument has been calibrated, it can be used to measure vapor samples. The samples can be introduced into the MSI directly from a headspace generator such as Lab In A Bag. A direct injection can be made using a 10-mL syringe or smaller, or an internal pump can be used to automatically pull in a sample from a port on the front of the panel. The system queries the operator for the desired selection. Calibration must be performed using the same technique as the injection mode. For example, the same size syringe must be used for calibration as for sample injection.

SECTION IV

METHOD PERFORMANCE

This section provides information on the performance of each method evaluated in the laboratory. In addition, limited field testing was done with the Antox test. Methods were evaluated for detection range, accuracy, precision, ruggedness, training requirements, and costs. Criteria used for evaluation of all methods are shown in Table 2.

TABLE 2. EVALUATION CRITERIA FOR BTEX FIELD METHODS

Performance Parameter	How Determined
Detection Range	Analysis of spiked samples over a large concentration range.
Accuracy	Comparison of the field method to accepted laboratory procedure.
Precision	Replicate analysis of the same sample.
Ruggedness	Operator observations.
Training Required	Evaluation of instruction manual; operator observations.
Cost	Speed of analysis, cost of supplies and equipment.

A. ANTOX IMMUNOASSAY

The Antox immunoassay is designed for the rapid analysis of water samples for determining the presence of BTX and related compounds. (See Reference 1 for a review of immunoassay techniques.) The test is a simple to learn, qualitative procedure, allowing personnel lacking an extensive knowledge of analytical chemistry to monitor sites for the presence of BTX.

The Antox test results for a sample are compared to a blank water reference sample. The absorbance of the sample is divided by the absorbance of the reference. This is referred to as the sample/reference ratio (S/R ratio). If the S/R ratio is less than 0.85, the test is positive for the presence of BTX. The S/R ratio shows an inverse relationship to the concentration of BTX in the sample (i.e., the more BTX in the sample, the less color develops in the tube). Figure 4 shows this relationship in water samples spiked with increasing amounts of toluene. The inverse relationship depicted in Figure 4 indicates the potential for the test to provide semiquantitative information.

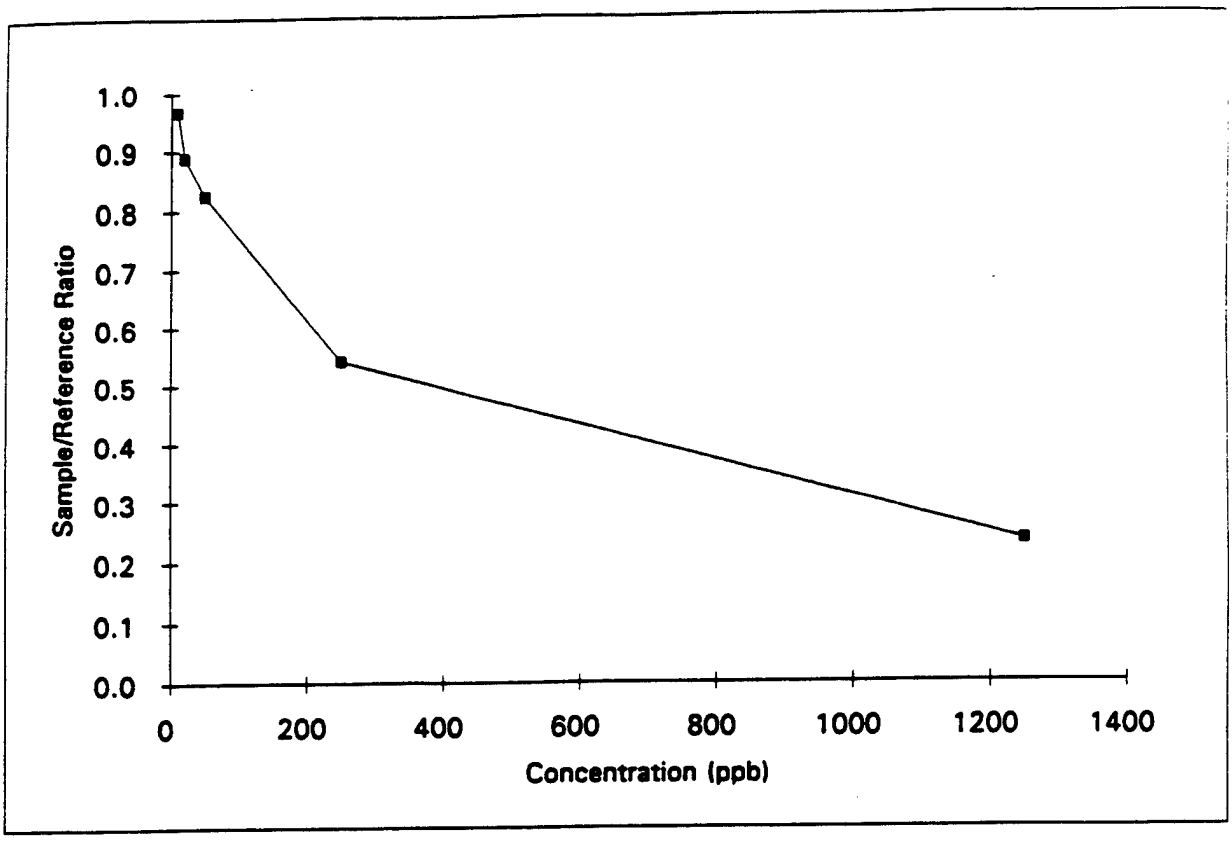


Figure 4. Response of the Antox S/R Ratio at Increasing Concentrations. An S/R Ratio of <0.85 Indicates a Positive Response.

1. Method Detection Limit

The manufacturer's claim for detection limits was 25 ppb. Preliminary analysis (Figure 4) showed that 50 ppb was very close to an S/R ratio of 0.85. Replicate tests were run at 50 and 75 ppb, respectively, to evaluate the reliability of the test near the detection limits (Table 3). In addition, during field testing, QA standards for toluene at 25 ppb and 100 ppb were run between January 22 and February 28, 1992 (Table 4).

The test was found to be reliable down to 75 ppb in water (Table 3). Although the test was expected to be positive, only 4 of the 12 tests were positive at 50 ppb, while 11 of 12 were positive at 75 ppb. In field QA checks, only 2 out of the 15 tests run at 25 ppb were positive, as compared to 14 positive results out of 15 at 100 ppb. This is a higher detection limit

TABLE 3. ANTOX LABORATORY ANALYSES OF WATER SPIKED WITH 50 AND 75 ppb TOLUENE

SAMPLE #	S/R RATIO 50 ppb	FLAG	S/R RATIO 75 ppb	FLAG
1	0.886	-	0.752	+
2	0.850	+	0.725	+
3	0.929	-	0.691	+
4	0.905	-	0.786	+
5	0.923	-	0.777	+
6	0.855	-	0.811	+
7	0.790	+	0.761	+
8	0.786	+	0.873	-
9	0.910	-	0.696	+
10	0.863	-	0.754	+
11	0.916	-	0.658	+
12	0.819	+	0.692	+

Abbreviations: ppb = parts per billion; S/R ratio = Sample/Reference ratio; - = S/R ratio of >0.85 a negative test; + = S/R ratio of <0.85 a positive test.

for toluene than the manufacturer's claim of 25 ppb. The achievable detection limit probably depends on the individual operator, due to the timing and hand-operated measurements required of this procedure. A person with good physical dexterity, patience, and composure will excel in performing this procedure. In addition, the model of spectrophotometer used and environmental conditions at the site will affect detection limits. The MDL should be verified by analyzing a spiked standard at the desired detection limit prior to analyzing samples.

2. Accuracy

The accuracy of the method was determined by analyzing field samples, contaminated with hydrocarbon fuels, by both the Antox test and by a laboratory GC method (6), then comparing the results. The majority of the field samples and results were obtained from an EPA Superfund Innovative Technology Evaluation Program (SITE) study conducted through the EPA Environmental Monitoring Systems Laboratory at Las Vegas (EMSL-LV) Immunochemistry Program (8). The SITE study of the Antox kit was conducted at the same time as the evaluations for this study. The SITE demonstration was designed to investigate the ability of the immunoassay to perform as a portable, on-site screening method for BTX-contaminated groundwater samples. The Las Vegas

Valley of Nevada provided a range of concentration levels for gasoline-contaminated groundwater. Sample splits were analyzed on-site using the BTX immunoassay, and in the laboratory by analysis using GC. Additional findings with respect to the BTX immunoassay evaluation may be found in a recent EPA internal report (9).

The concentration of BTX in the environmental samples, as determined by the laboratory GC method, is compared to the Antox results in Figure 5. Samples were analyzed in duplicate by the Antox test, and both points were plotted separately. Sample points below the horizontal line on Figure 5 are positive results according to the Antox tests (an S/R ratio less than 0.85). Most of these points were measured at greater than 25 ppb with the GC method (the vertical line on Figure 5). Likewise, most of the samples with negative Antox results show less than 25 ppb with the GC method. A few samples show GC concentrations above 25 ppb that were not detected by the Antox method, agreeing with the detection limit data in Tables 3 and 4.

This again indicates that the lower detection limit of the Antox test should be greater than the 25 ppb level specified by the manufacturer. The GC concentration is a sum of the BTEX concentrations in the water sample expressed in parts per billion. The Antox results for field samples were rather scattered. Samples with a GC concentration near 1,000 ppb were all detected above the lower detection limit with the Antox test with an S/R ratio varying between 0.1 to 0.7. These results show that the method is useful as a field screening qualitative test to identify which water samples are contaminated with BTX, but accurate quantitation is not possible.

TABLE 4. ANTOX ANALYSES OF FIELD QA WATER SAMPLES SPIKED WITH 25 AND 100 ppb TOLUENE.

SAMPLING DATE	S/R RATIO 25 PPB	FLAG	S/R RATIO 100 PPB	FLAG
1/22/92	0.79	+	0.50	+
2/24/92	1.02	-	0.83	+
2/25/92	1.40	-	0.95	-
2/25/92	1.02	-	0.84	+
1/22/92	0.98	-	0.72	+
2/24/92	0.69	+	0.51	+
1/23/92	0.95	-	0.66	+
1/23/92	0.92	-	0.81	+
2/26/92	0.93	-	0.72	+
2/27/92	0.87	-	0.65	+
2/28/92	1.06	-	0.85	+
2/25/92	0.93	-	0.80	+
2/26/92	1.00	-	0.72	+
2/27/92	0.91	-	0.68	+
2/28/92	0.87	-	0.68	+

Abbreviations: ppb = parts per billion; S/R ratio = Sample/Reference ratio; + = S/R ratio of <0.85 a positive test; - = S/R ratio of >0.85 a negative test.

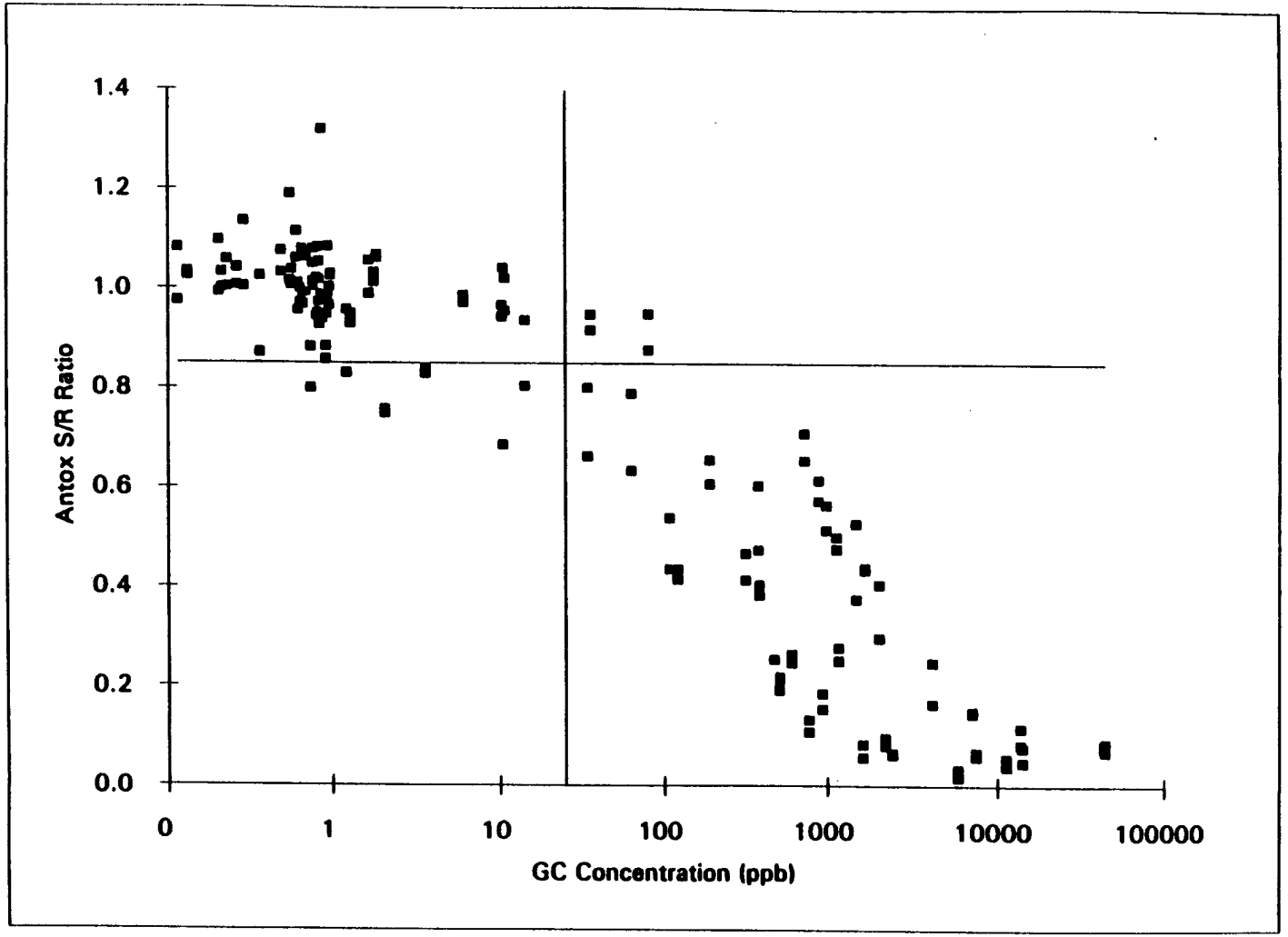


Figure 5. Antox S/R Ratio Compared to Total BTEX Concentration Determined from Field Samples.

3. Precision

Method precision was measured at 50 and 75 ppb in the laboratory and 25 and 100 ppb in the field (Tables 3 and 4). The "+" flag indicates an S/R ratio of <0.85, or a positive test, while the "-" flag indicates a negative test. The range of S/R ratios from laboratory tests at 50 ppb was 0.929 to 0.786 (0.143 range). At 75 ppb, the range was 0.873 to 0.658 (0.215 range). Field QA tests at 25 ppb had an S/R range of 0.69-1.40 (0.71 range); field QA tests at 100 ppb had an S/R range of 0.50-0.95 (0.45 range). This indicates a fairly large range of response in S/R ratios to a given concentration of toluene under field conditions. Analysis of field samples agreed with these data.

4. Ruggedness

For each test, the sample tube was compared to a reference tube containing distilled water. A few times the reference tube failed: the reading on the spectrophotometer was too low (<1.0). The exact cause of this failure was not determined; however, it could have resulted from either operator error or a bad tube. The reference tube should have an absorbance reading of between 1.0 to 1.9 absorbance units. If not, the test should be re-run. For the best reliability, the test should be run twice for each sample, and each test should be run with a separate reference tube.

The reading of the spectrophotometer may be affected by ambient light. In other words, direct sunlight on the instrument may produce erroneous results. A cover should be placed over the cuvette to prevent stray light from entering the light path of the instrument. Some portable photometers have such a cover, but others do not. A tight cover should be ensured to reduce the potential for error.

Temperature may influence the results of the test. The manufacturer recommends that for ambient temperatures of greater than 24°C, several steps should be performed in an ice water bath. The effect of temperature on the results of this test has not been fully investigated in this evaluation. All laboratory tests were run between 21°C and 24°C. Field tests were performed between 18°C and 32°C. To guarantee quality results, water spiked with toluene (or the site-specific analyte) at the desired detection limit should be analyzed at the field site.

5. Training Required

The test is simple to learn, and easy to perform. The manufacturer includes a five-page instruction manual with each shipment of test kits. These instructions are brief but contain sufficient detail to properly instruct the user. Only a few hours of training should be required. However, the operator should be aware that certain steps are critical to the successful completion of the test. Four drops, **no more or less**, of each reagent are required at specific times. If more drops are added by accident, the test should be discarded and repeated with new tubes. The

timing of the steps is also important. As with any procedure, the test should be practiced in a laboratory setting prior to being used at a field site.

6. Cost

The cost of performing the Antox tests depends on how the test is performed. If each test tube is measured against a separate reference tube, the test is most reliable, but also the most costly. The manufacturer recommends no more than four test tubes be run in conjunction with a single reference tube. During our tests, four tubes were used with each reference tube, resulting in a per sample materials cost of approximately $9.00 per sample. If a single test had been run with each reference, the cost would have increased to approximately $15.00 per test. The cost for an analyst will vary with experience, but can be factored into a cost estimation by adding the analyst's daily wages to the cost of producing 64 tests (a typical daily number of runs). The cost of the spectrophotometer required for the test is approximately $600.00.

B. DETECTOR TUBES FOR ANALYSIS OF WATER SAMPLES

Detector tubes have been used for years for the analysis of ambient air in industrial hygiene applications. Using simple apparatus, these tubes can also be used for analyzing water samples (2,3,4). Using a hand-operated pump, ambient air is purged through a water sample into a detector tube, which changes color in proportion to the concentration of the specific analyte in the water. This method has been reported to work for soil samples (3); however, soil was not tested for this study, as a protocol has not yet been developed for soil samples. The concentrations of spiked water samples for all analyses performed below were verified using an HP5890 GC and EPA Method 8020 for analysis (5).

1. Method Detection Limits

Two different tubes were evaluated in this study: Draeger tube #800-23001 calibrated for detection of toluene, and Draeger tube #800-28561 calibrated for detection of benzene. The tube evaluated for toluene is the one recommended by Draeger for water analysis. The manufacturer claims this tube can detect toluene in water from 1 to 10 mg/L (ppm) and can detect ethylbenzene and xylene in water at approximately the same range. However, benzene is not detected with this sensitivity. Instead, Draeger now recommends tube #800-01231 for detecting benzene in water, with a range from 0.5 to 5 mg/L (ppm). At the time this study was conducted, this particular tube was not being marketed by Draeger specifically for water analysis, and was not evaluated. Tube #800-28561 was selected for use in this study because it has a lower reported detection limit for air analyses, and was evaluated to determine if these lower detection limits could be achieved in water samples. If successful, this tube would provide a more sensitive screening test than the tube currently recommended by the manufacturer.

The toluene tube was found to meet the manufacturer's specifications (Table 5). Toluene was detected from 0.625 ppm to 10 ppm in spiked water samples. Ethylbenzene was

detected down to 0.625 ppm and o-xylene was detected down to 0.5 ppm using this tube. Ethylbenzene, o-xylene, and benzene were only tested near the manufacturer's reported detection limits and not above 1.25 ppm. Further testing of this tube with benzene was not considered useful because the manufacturer's claims of reduced sensitivity were substantiated. Ethylbenzene and xylene were not evaluated for linearity of response (see Section B-3) because their response at low levels was very similar to toluene, and the manufacturer indicates that these three chemicals will react very similarly with this tube.

TABLE 5. DETECTOR TUBE #800-23001 RESPONSE TO INCREASING SPIKED CONCENTRATIONS OF BTEX IN WATER.

	DETECTION TUBE READINGS		
Spiked Conc. (ppm)	Toluene	Ethylbenzene	o-Xylene
0.5	----	---	40
0.625	32.5	25	---
1.25	47.5	60	65
2.5	77.5	---	---
5.0	150.0	---	---
10.0	345.0	---	---

Abbreviations: ppm = parts per million; --- = not determined.

The benzene tube evaluated in this study showed good sensitivity to benzene in spiked water, ranging from 0.025 to 0.5 ppm. Although this tube has better sensitivity than the tube recommended by Draeger, other observations suggest this tube may not be appropriate for field use (as shown in Subsections 3 and 4, following).

2. Precision

The precision of the method was good, with the %RSD for replicate analyses below 15 percent (Table 6). Replicate analyses were performed with water spiked at 1.25 ppm of toluene for tube #800-23001 and 0.075 ppm of benzene for tube #800-28561. The precision of reading for the tubes is limited by the sharpness of color response within the incremental scale on the tube. The reading on the tube should be recorded as the nearest scale line. It is possible, but difficult to estimate, the reading between scale markings. The color stain sometimes does not have a sharp boundary. In addition, various operators may record the measurement differently, depending on one's interpretation of where the limit of the color stain is, further decreasing the precision of the method.

TABLE 6. REPLICATE DETERMINATIONS OF WATER SPIKED WITH 0.075 ppm BENZENE AND 1.25 ppm TOLUENE.

Run #	Benzene Readings Tube #800-23001 Tube Scale Reading	Toluene Readings Tube #800-28561 Tube Scale Reading
1	4	60
2	4	50
3	3	40
4	3.5	50
5	3.5	45
6	4	50
MEAN	3.7	49.1
STANDARD DEVIATION	0.41	6.6
%RSD	11.1	13.5

Abbreviations: ppm = parts per million; %RSD = percent relative standard deviation.

3. Accuracy

Accuracy was evaluated as a proportional response to a known spiked concentration in a sample (e.g., see Table 5). All readings were performed in duplicate, and the linear response plotted against concentration. If the response is linear, then the concentration in the sample can be reliably predicted by the detector tube reading. The toluene tube was found to have a linear response to increasing analyte concentration spiked in water (Figure 6). The points on this plot fall very near a straight line. A simple linear regression could be effectively used to convert the tube readings into concentration in ppm.

The benzene tube did not show good linearity, as shown in Figure 7. This lack of linearity under controlled laboratory conditions suggests quantitative data could not be obtained under field conditions. While the shape of the curve in Figure 7 could be fit to a nonlinear regression model, this increases the complexity of analysis and would not lend itself to typical field screening procedures.

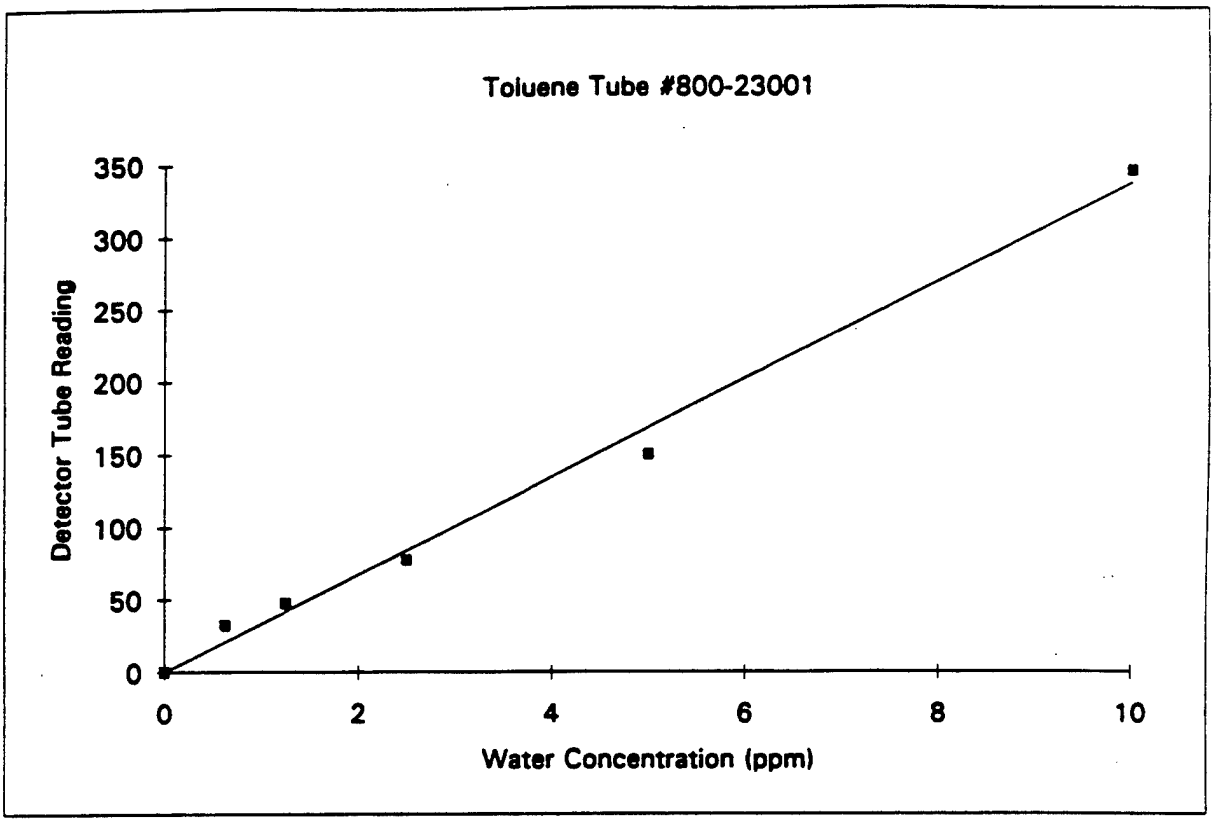


Figure 6. Response of Detection Tube #800-23001 to Increasing Concentrations of Toluene in Water.

4. Ruggedness

The simplicity of the method is an advantage, because minimal equipment is required. All the supplies for the method can be carried in a carrying case. The glassware is somewhat fragile, and care must be exercised to avoid breakage in the field.

The results of the method can be affected by the temperature of the water sample. Method calibration should be performed at the same temperature as the water samples.

Not all types or brands of tubes are suitable for field screening tests. For example, tubes in which high moisture content can adversely affect the color indicator are not suitable. One of the tubes evaluated in this study (Draeger Benzene tube #800-28561) did not have a linear response to increasing concentrations in water. The color change was hard to detect, probably due to the effect of the high moisture content of the purged headspace. This effect could easily be worsened in the field under variable lighting and temperature regimes. The other tube evaluated (Draeger Toluene tube #800-23001) performed much better, although not with the sensitivity of the Benzene tube. It is important, therefore, to use tubes specified by the kit manufacturer, or to test tubes for linearity in a laboratory before using them in the field.

5. Training Required

The procedure is quick and simple to learn; only a few hours of training should be required, depending upon the attitude and motivation of the trainee. Although a chemistry background is not required to learn this procedure, some knowledge of chemicals and familiarity with measuring processes (such as following recipes) would be helpful.

6. Cost

The cost of Draeger tubes is approximately $20.00 per test. As with the Antox test, the cost of an analyst's wages should be added to the number of samples that can be run in a typical day. In our tests, approximately 100 samples per day could be completed. The approximate cost of the equipment required to the run the test is $890.

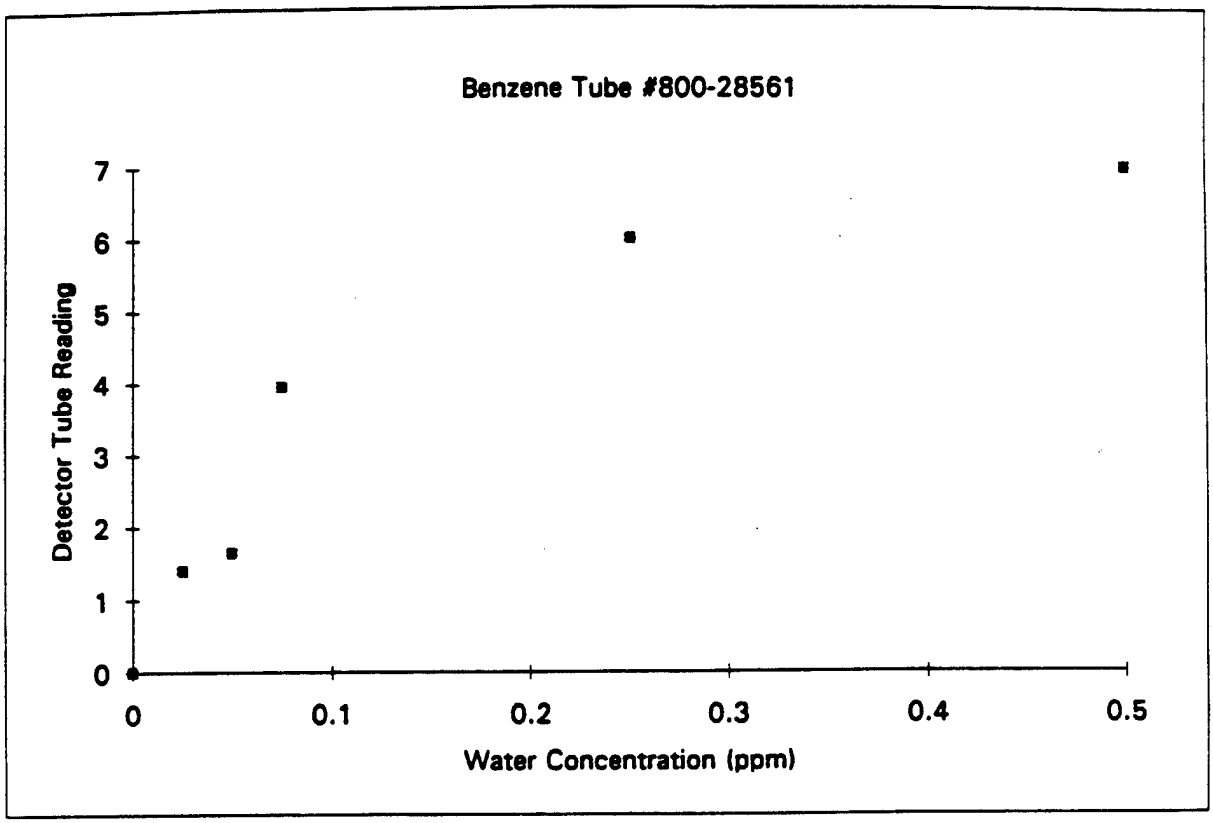


Figure 7. Response of Detection Tube #800-28561 to Increasing Concentrations of Benzene in Water.

C. LAB IN A BAG

Lab In A Bag is a sample preparation method for analyzing water or soil samples for VOCs. The Lab In A Bag is a low-cost instrument designed to use the polyethylene bag sampling system as a headspace method (6,7). The concentration of all samples reported in this section were verified using an HP5890 GC and EPA Method 8020 for analysis (5).

1. Method Detection Limit

This method provides relatively high sensitivity for a field method. The method was found to provide gas headspace that allowed detection of 10 ppb BTEX in water samples (Table 7) and 40 ppb BTEX in soil samples (Table 8). This detection limit was achieved when using the Lab In A Bag with the MSI-301A GC (see Subsection D). The detection limit should be lower for the more volatile analytes. The more volatile the compound, the more it will move into the bag headspace. Some of the analytes could not be detected reliably at the low ppb levels; notably, xylene was not detected until 50 ppb in water and 200 ppb in soil. At these low levels, the analytes are probably irreversibly absorbed into the polyethylene bag or complexed within the soil matrix. The results for ethylbenzene are somewhat erratic near the detection limit. This may be due to contamination present in the bags or in the water used for the experiment.

TABLE 7. MEASUREMENTS OF LAB IN A BAG HEADSPACE SAMPLES AT INCREASING CONCENTRATIONS IN WATER USING THE MSI PORTABLE GC.

	MSI Headspace Concentration (ppm)			
Spike Concentration in Water (ppm)	Benzene	Toluene	Ethylbenzene	o-Xylene
0.0	0.012	0.003	0.071	0.000
0.005	0.078	0.010	0.055	0.001
0.0075	0.125	0.021	0.000	0.001
0.010	0.195	0.044	0.146	0.007
0.050	1.054	0.464	0.811	0.211
0.200	4.48	2.36	1.99	1.53
0.500	10.52	5.46	4.16	3.68

Abbreviations: ppm = parts per million.

TABLE 8. MEASUREMENTS OF LAB IN A BAG HEADSPACE SAMPLES AT INCREASING CONCENTRATIONS IN SOIL USING THE MSI PORTABLE GC.

	Headspace Concentration (ppm)			
Spike Concentration in Soil (ppm)	Benzene	Toluene	Ethylbenzene	o-Xylene
0.000	0.008	0.001	0.090	0.000
0.040	0.041	0.014	0.125	0.003
0.200	1.153	0.492	0.722	0.233
0.800	4.847	3.082	4.865	3.704
2.000	10.882	6.191	5.961	4.596
3.000	14.622	8.316	7.431	6.153
4.000	18.249	10.163	9.311	7.854

Abbreviations: ppm = parts per million.

As a comparison to the Lab In A Bag procedure, the laboratory method using purge-and-trap technology can achieve detection limits of 0.2 ppb in soil and water (5). The achievable detection limits for the Lab In A Bag method will vary with the matrix type, especially for soil. The organic content, soil type, and sampling methods can affect the sensitivity of the method. Before analyzing samples, the detection limit should be verified by spiking a contaminant-free matrix at a level near the desired detection limit, then analyzing the spiked samples.

2. Precision

The method can provide good precision for water samples, as shown in Table 9, which lists the results of replicate water samples spiked at 5 ppb, 10 ppb, and 500 ppb. At the 500 ppb spike level, the precision was less than or very close to 5 percent for all analytes. The precision is best for the more volatile analytes, with benzene showing less than a one %RSD at 500 ppb. Precision was also good at the 10 ppb level for water (near detection limit) with the exception of xylene (the least volatile component), which showed a 49 %RSD. Benzene, the most volatile component, shows a %RSD of less than 10 percent, which was similar to ethylbenzene (11.5 percent). Toluene was somewhat intermediate in precision with a %RSD of 27.

The precision of the method for soil samples was not as good as for water samples (Table 10). The precision ranged between 4.7 percent and 38 percent, but was not dramatically better at high concentrations (500 ppb), as compared to near detection limit concentrations. Ethylbenzene appears to have a somewhat higher detection limit in our soil matrix than the other compounds (Table 10). Results for this compound were also somewhat erratic in water samples near the detection limit (see Tables 7 and 9).

3. Accuracy

In a similar manner as detector tubes, the accuracy of the Lab In A Bag procedure depends on the concentration of the volatile compounds in the bag headspace (gaseous phase) being proportional to the original concentration in the sample. If the sample and headspace volatile concentration is directly proportional, then the method accuracy should be high.

The linearity of the Lab In A Bag method was evaluated in the laboratory by analyzing water and soil spiked with increasing levels of BTEX. Water was spiked at four different levels; the results are illustrated in Figure 8. Soil was spiked at six different levels, and the results are shown in Figure 9. The water samples show good linearity of matrix concentration with Lab In A Bag headspace concentration. This linearity is similar to that of the laboratory purge-and-trap method (5). The linearity using soil samples was not as good as for water samples. This was probably a function of soil matrix effects, which will reduce the accuracy of headspace sample analyses.

TABLE 9. REPLICATE MEASUREMENTS OF LAB IN A BAG HEADSPACE SAMPLES AT WATER SPIKE LEVELS OF 5, 10, AND 500 ppb USING THE MSI PORTABLE GC.

	MSI HEADSPACE CONCENTRATION (ppm)			
Spike Conc. (ppb)	Benzene	Toluene	Ethylbenzene	o-Xylene
5	0.067	0.009	0.000	0.000
5	0.097	0.013	0.164	0.002
5	0.070	0.009	0.000	0.001
MEAN	0.078	0.010	0.055	0.001
%RSD	17.3	18.2	141.4	81.6
10	0.206	0.071	0.185	0.014
10	0.161	0.045	0.150	0.006
10	0.188	0.031	0.138	0.005
10	0.188	0.038	0.135	0.006
10	0.197	0.040	0.138	0.007
10	0.221	0.044	0.133	0.005
10	0.202	0.038	0.142	0.003
MEAN	0.195	0.044	0.146	0.007
%RSD	8.9	27.1	11.5	49.4
500	10.580	5.538	4.331	3.945
500	10.601	5.280	3.89	3.346
500	10.458	5.330	4.074	3.553
500	10.431	5.606	4.364	3.884
500	10.532	5.570	4.151	3.679
MEAN	10.52	5.46	4.16	3.68
%RSD	0.6	2.4	4.2	5.9

Abbreviations: ppb = parts per billion; ppm = parts per million; %RSD = percent relative standard deviation.

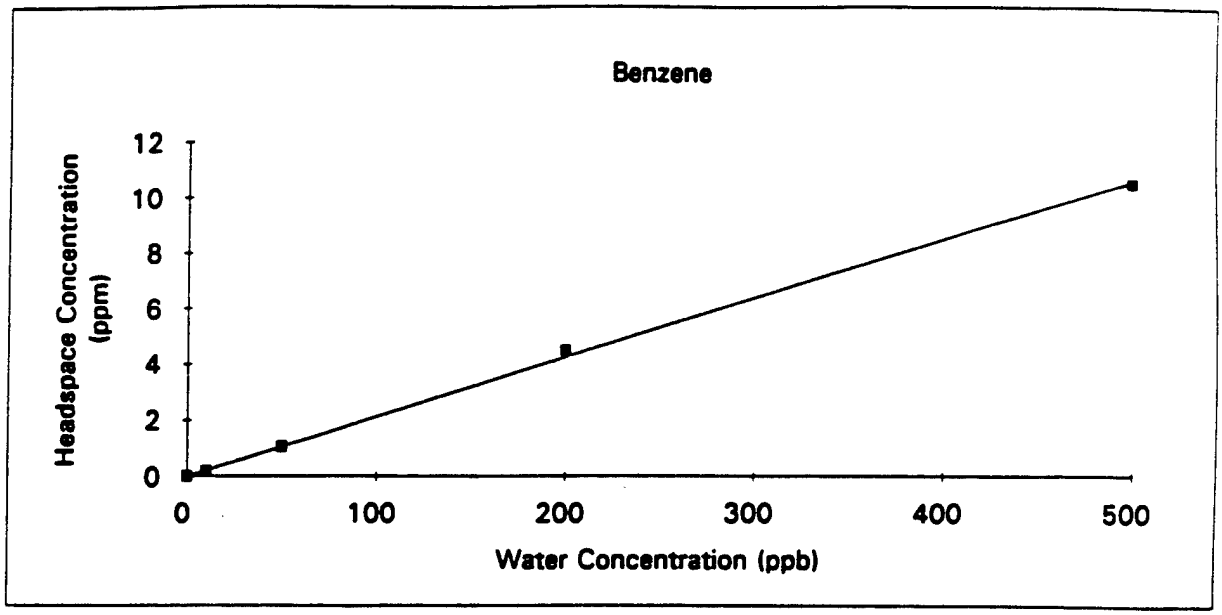
TABLE 10. REPLICATE MEASUREMENTS OF LAB IN A BAG HEADSPACE SAMPLES AT SOIL SPIKE LEVELS OF 50 AND 500 ppb USING THE MSI PORTABLE GC.

	MSI HEADSPACE CONCENTRATION (ppm)			
Soil Conc. (ppb)	Benzene	Toluene	Ethylbenzene	o-Xylene
50	0.241	0.071	0.000	0.020
50	0.162	0.057	0.000	0.015
50	0.257	0.063	0.000	0.027
50	0.226	0.052	0.000	0.021
50	0.312	0.078	0.000	0.030
MEAN	0.240	0.064	0.000	0.023
%RSD	20.3	14.3	0.0	23.2
500	1.921	0.408	0.415	0.124
500	1.449	0.262	-----	0.098
500	1.331	0.164	-----	0.070
500	3.193	0.359	0.466	0.208
500	2.572	0.355	0.444	0.184
MEAN	2.093	0.310	0.442	0.137
%RSD	34.6	28.0	4.7	38.1

Abbreviations: ppb = parts per billion; ppm = parts per million; %RSD = percent relative standard deviation.

4. Ruggedness

The operator should be confident that the analytes in the bag have reached equilibrium between the liquid and vapor phase before analyzing. The manual suggests that the operator determine the proper stirring time for a particular standard substance or site contaminant by running a series of identical samples through the Lab In A Bag, using different stirring times for each sample. Figure 10 is a graph of instrument response versus stirring time for a water sample and for a soil sample spiked with benzene. Other analytes (toluene, ethylbenzene, and o-xylene) showed similar curves. These results show that a stir time of 5 minutes resulted in a stable gas-phase sample for our experimental conditions.



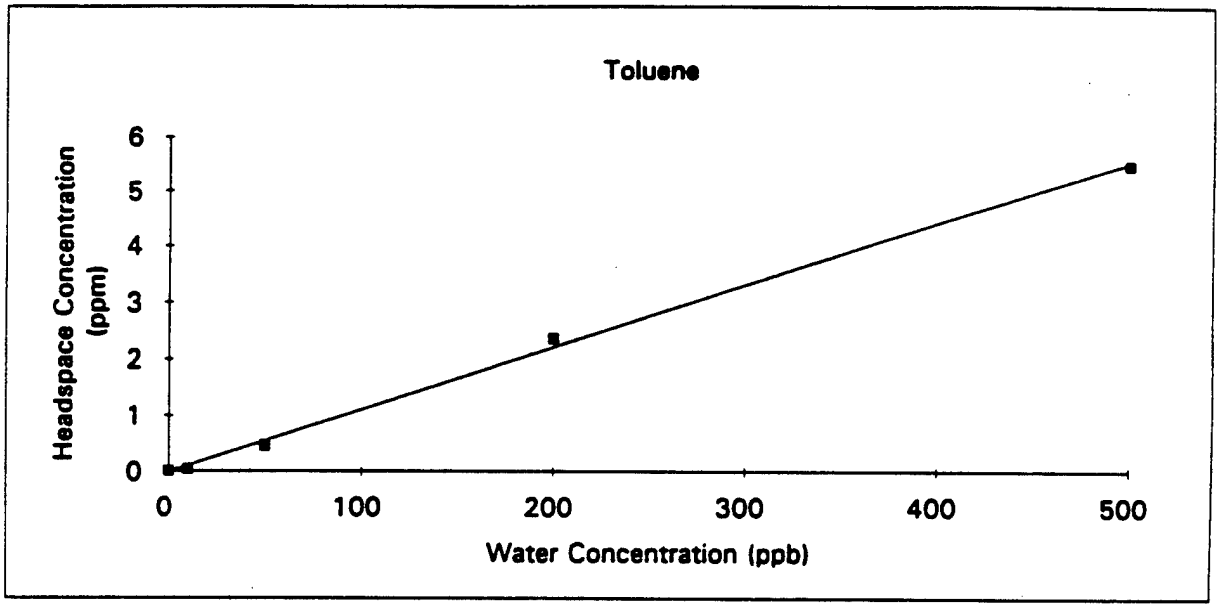
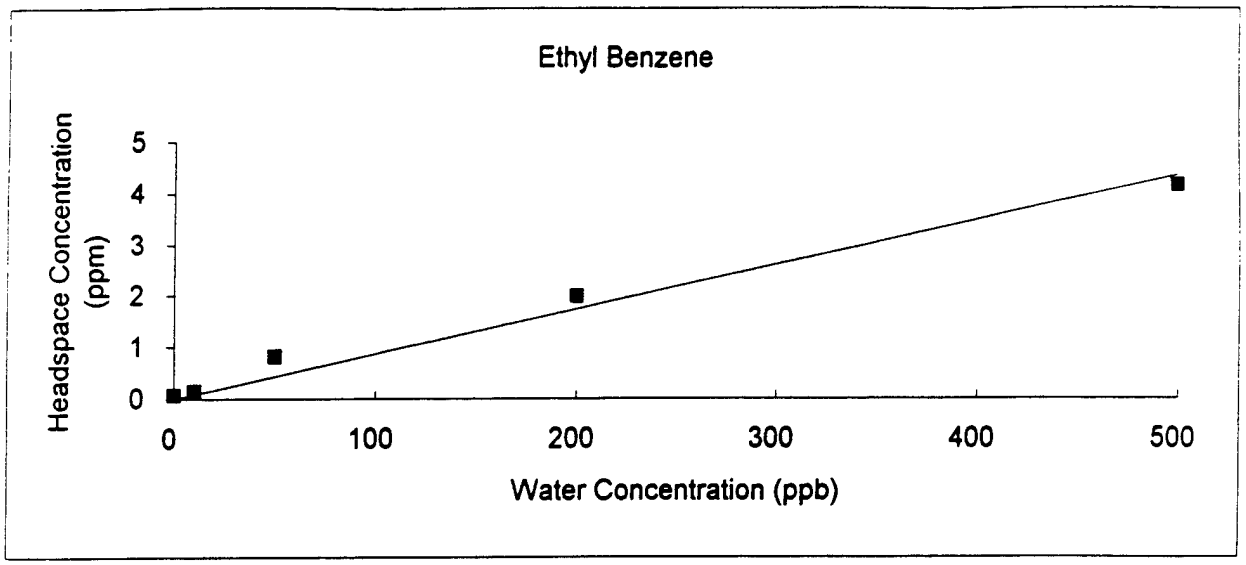


Figure 8. Concentration in Lab In A Bag Headspace Samples Versus Spiked Water Concentrations (Continued).



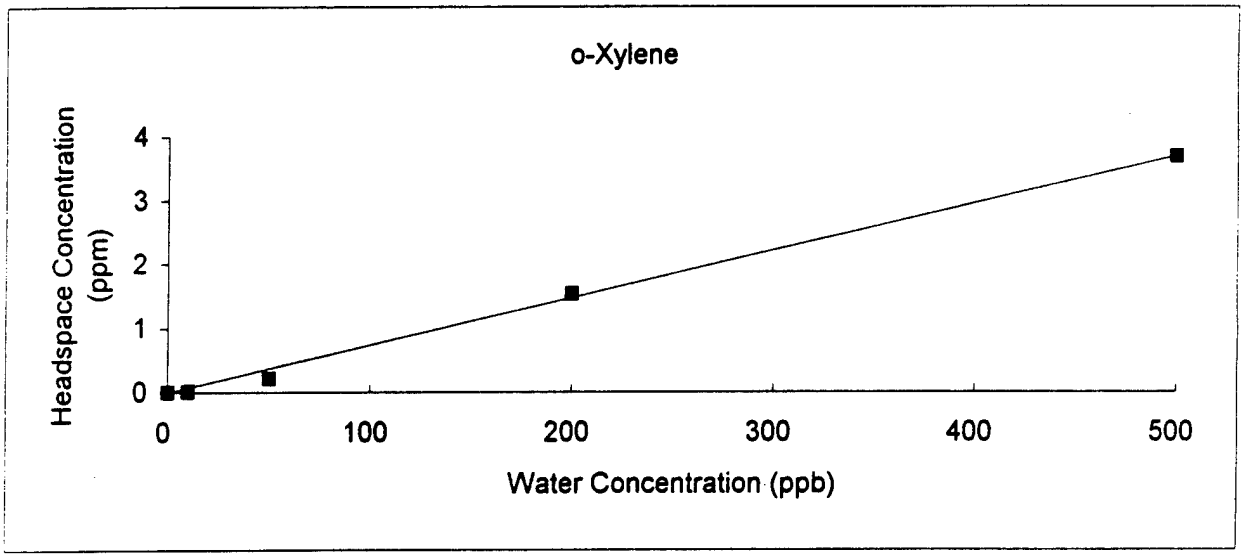
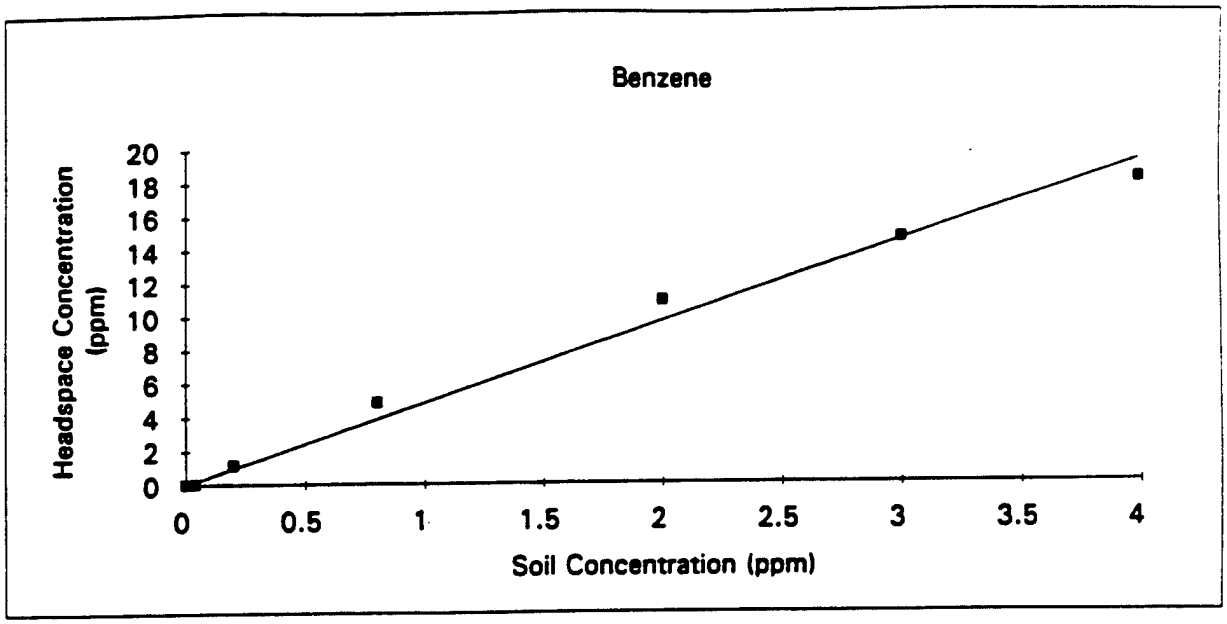


Figure 8. Concentration in Lab In A Bag Headspace Samples Versus Spiked Water Concentrations (Concluded).



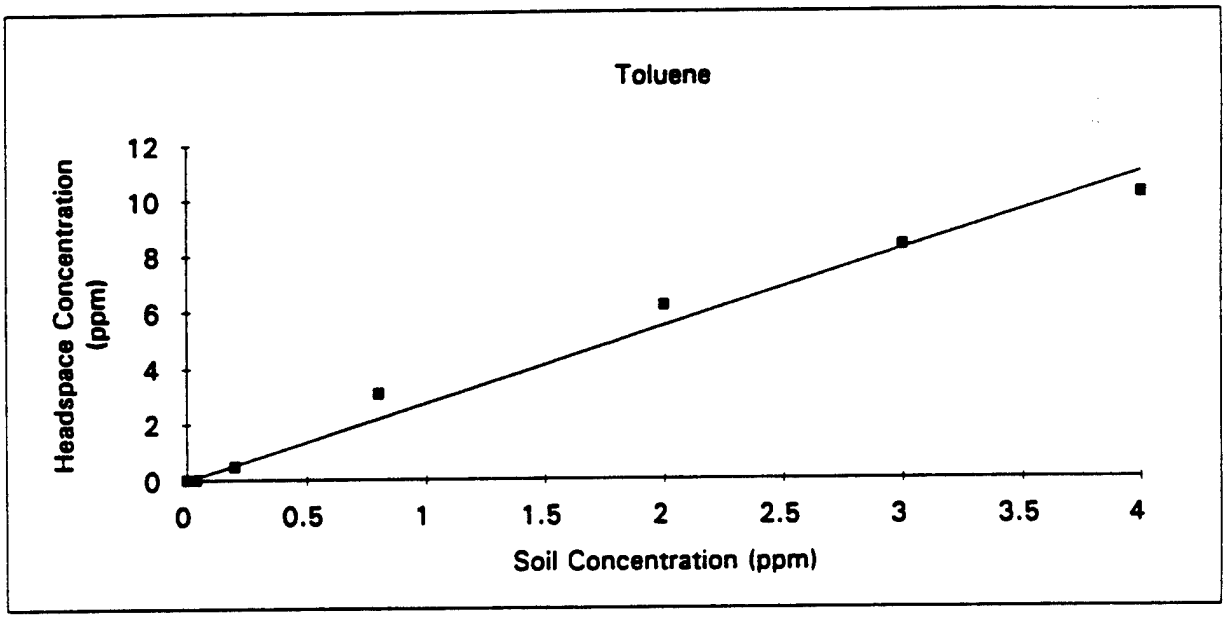
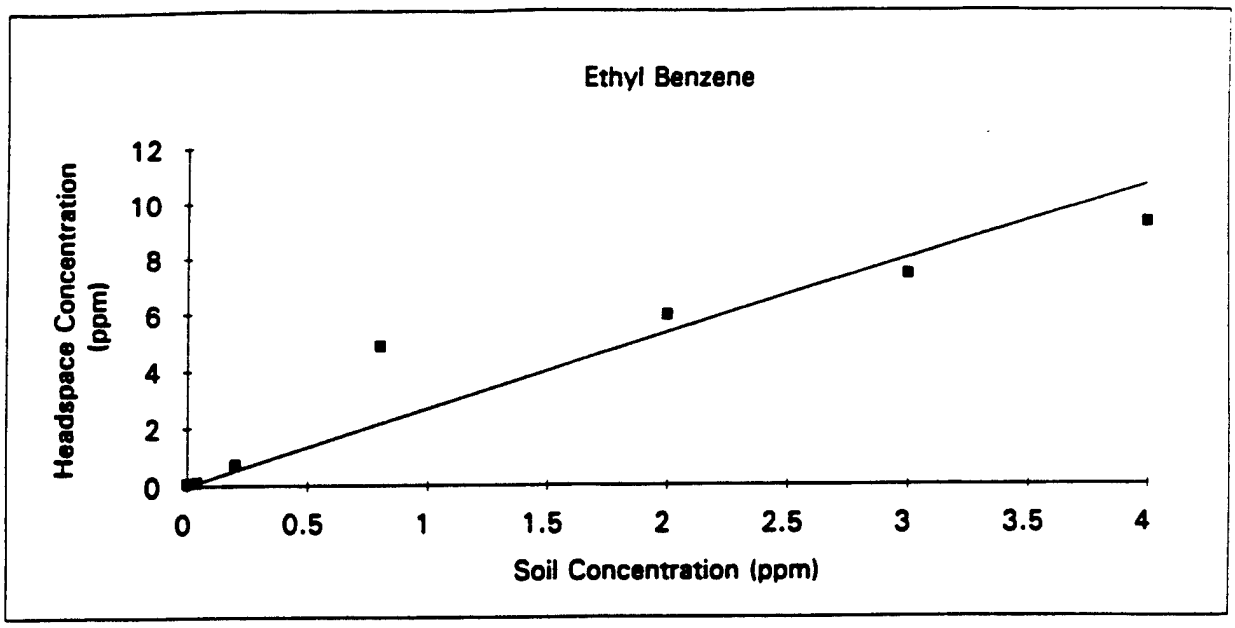


Figure 9. Concentration in Lab In A Bag Headspace Samples Versus Spiked Soil Concentrations (Continued).



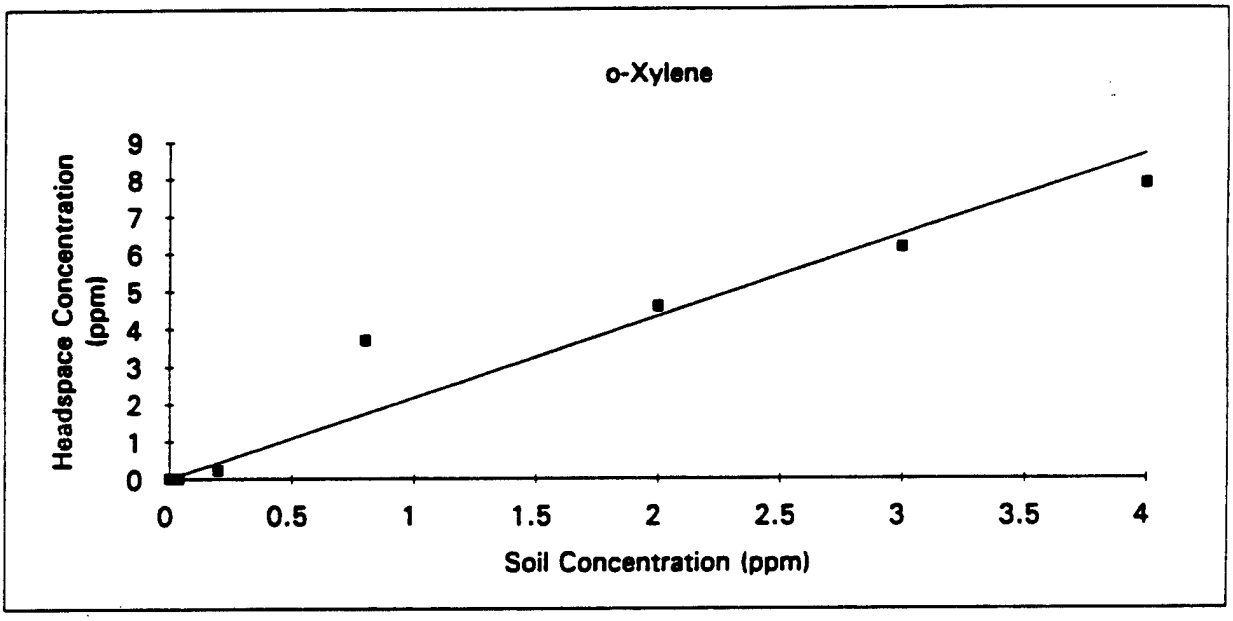


Figure 9. Concentration in Lab In A Bag Headspace Samples Versus Spiked Soil Concentrations (Concluded).

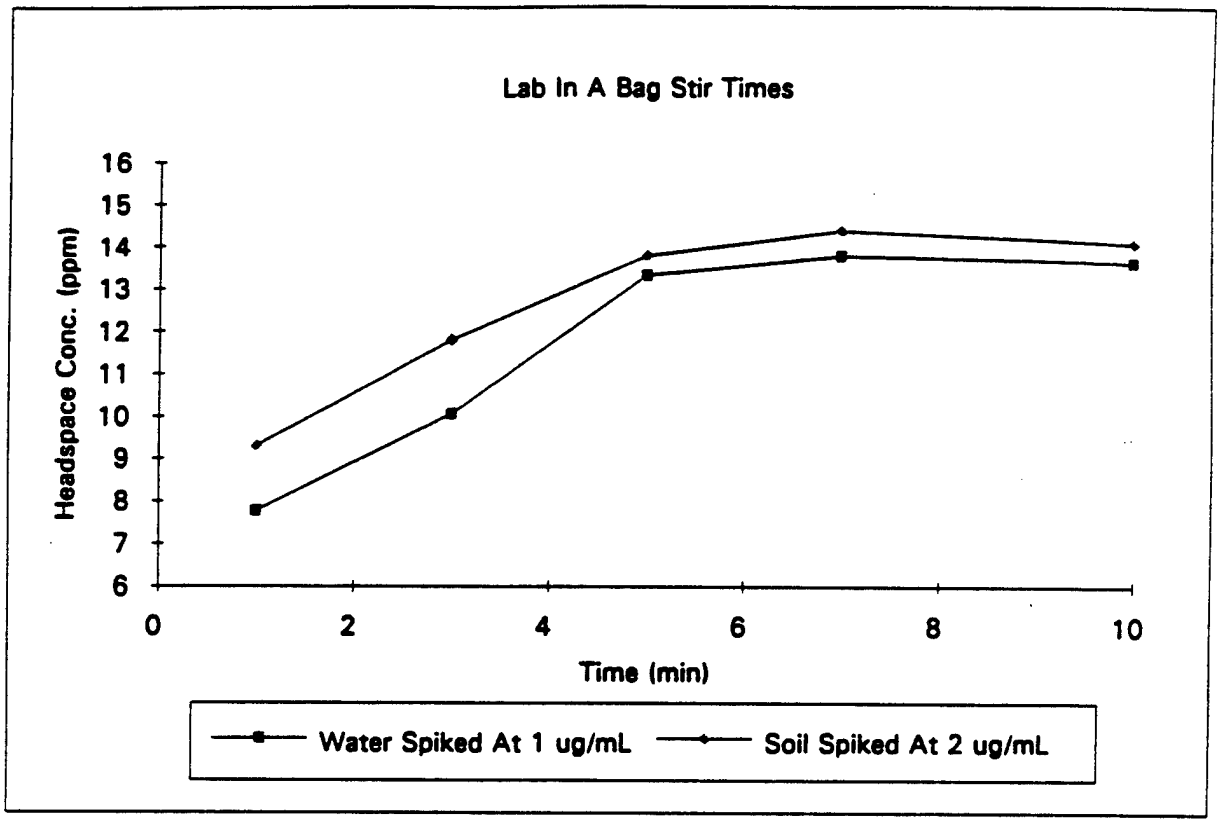


Figure 10. Effect of Stir Time on Water and Soil Samples Containing Benzene.

Sample carry-over can be a problem when analyzing low-level contaminated samples immediately after analyzing a high-level sample. A blank sample should be analyzed after a high-level sample to guarantee that the system is completely flushed of contaminant.

Temperature fluctuation can have an adverse affect on the analytical results. Warmer temperatures will cause more of the volatile compounds to move into the bag headspace than would be present at cooler temperatures. The instrument has no temperature control. For the most accurate results, calibration standards should be run at the same temperature as samples.

Sometimes the bags leak. If the bag does not appear to be fully inflated after the stir equilibrium time, the bag may have leaked, and the sample should be discarded. This problem may be avoided by checking for bag leakage prior to analysis by pressure testing the bag using the procedure presented in Section III C. 2. Soil samples can be more of a problem than water samples. Soil particles can prevent a tight seal of the bag. Sometimes it is difficult to start the stir bar spinning in the soil sample. If this happens, the bag must be manipulated, mixing the soil and water enough to allow for proper stirring. Manipulation of the bag may increase the chances of creating a leak in the bag.

5. Training Required

The operator's manual included with Lab In A Bag is very simple and easy to use. Each step of the procedure is accompanied by a photograph. In addition to the step-by-step procedures for analyzing soil and water, the manual includes a discussion of the theory of operation and helpful hints on sampling and calibration. Though the test is simple to perform, an operator should spend several days in a laboratory setting to become familiar and confident with the operation of the system. This will maximize the reliability of field results obtained.

6. Cost

The system costs approximately $2,000. Other costs associated with this technique include standards and analyst's time. Costs of standards are quite variable, depending on the analyte and the purity requested. In our experience, a headspace sample can be prepared in approximately 5 minutes. Depending on the experience of the laboratory analyst, 6-8 samples per hour can be prepared and analyzed. As with detector tubes, the cost per sample using Lab In A Bag and a suitable detector could be estimated by factoring the cost of an analyst into the number of samples that can be processed per day (48-64 samples per day).

D. MSI-301A ORGANIC VAPOR MONITOR

The MSI-301A Organic Vapor Monitor is a field-portable, commercially available GC designed for the analysis of specific VOCs. This model is set up for the detection of BTEX. The instrument provides controlled conditions for field analysis of soil-gas or water and soil headspace

without requiring large amounts of equipment or supplies. The unit can be operated on AC power or battery power using scrubbed ambient air as carrier gas.

1. Method Detection Limits

The instrument is quite sensitive to BTEX, with a lower detection limit of 1 ppb (by volume) in air. The accuracy of measurements in the low ppb range is hard to verify because accurate gas standards are difficult to prepare and verify in this range. However, the instrument reliably measures relative levels of BTEX in ambient air present in the low ppb range. The upper measurement limit of this device is about 100 ppm. Higher concentrations of BTEX could foul the detector and should not be introduced into the instrument, as recommended by the manufacturer.

2. Precision

Instrument precision was found to be quite good for analyses performed within the same day (see Tables 9 and 10). The results of replicate gas standard samples at 10 ppm analyzed with the MSI-301A displayed %RSD for all analytes of less than 5 percent, comparing quite favorably to laboratory-grade GC instrumentation (Table 11).

Day-to-day measurements had greater variability than within-day measurements. Benzene was quite reproducible from one day to the next; toluene and ethylbenzene showed up to 20 percent variation from one day to the next, and o-xylene showed up to 40 percent change within one day (Figure 11). The detector can change its sensitivity depending on how long the instrument is left on. This change of sensitivity is not a problem if the instrument is calibrated on at least a daily basis.

TABLE 11. REPLICATE MEASUREMENTS OF BTEX AT 10 ppm GAS USING THE MSI PORTABLE GC.

	MSI CONCENTRATION (ppm)			
	Benzene	Toluene	Ethylbenzene	o-Xylene
	11.29	10.04	10.31	10.31
	10.67	10.02	10.23	10.05
	10.13	9.60	9.73	9.51
	10.23	9.82	9.90	9.64
	10.24	10.27	10.57	10.45
	10.16	10.00	10.12	9.83
	10.15	10.38	10.78	11.01
	10.10	10.04	10.19	10.02
	10.13	9.91	10.04	9.82
	10.19	10.10	10.37	10.26
	10.20	10.14	10.35	10.24
	10.02	10.02	10.27	10.08
MEAN	10.29	10.03	10.24	10.10
%RSD	3.3	1.9	2.6	3.8

Abbreviations: ppm = parts per million; %RSD = percent relative standard deviation.

3. Accuracy

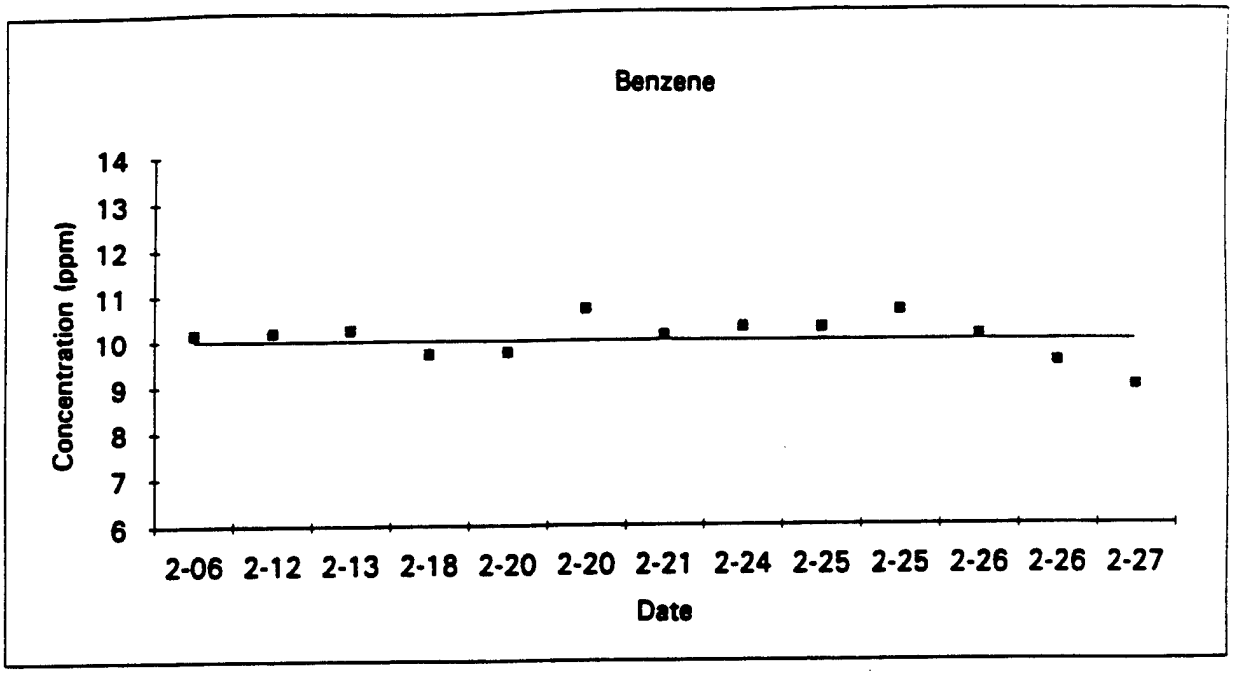
The accuracy of the MSI was determined by analyzing replicate samples of a gas standard (Table 11) and samples taken from a soil column and analyzed on both the MSI and a laboratory GC (Hewlett-Packard 5890). As seen in Table 11, the mean values for measurements of the gas standards on a single day were close to 10 ppm with little variance. This demonstrates that once calibrated to a known gas standard, the instrument reliably reproduced those measurements during a single days' operation.

A soil column was built for this project to provide a homogeneous soil matrix for analysis. The column was spiked by flowing a gas-phase standard containing 10 ppm BTEX through the column. While the column was being spiked with BTEX, the soil gas was monitored by the MSI and the HP5890 at the same time until the column had become saturated. The two GCs showed very good correlation (Figure 12). The diagonal line in Figure 12 represents an ideal correlation (i.e., the

results from the two instruments are exactly the same). Most of the points for benzene and toluene fall close to this line. The spread of difference between the two instruments was larger as the concentration approached 10 ppm. The cause of the differences cannot be determined from this experiment because the true value of gas concentration within the columns was not known. The ethylbenzene and o-xylene generally fall above the ideal line (i.e., the MSI reading was higher than the HP-5890). This may indicate a bias toward slightly higher readings for the MSI.

4. Ruggedness

The MSI-301A is designed for hands-on operation for personnel without extensive experience in analytical instrumentation; therefore, it is simpler to operate than most GCs. The instrument was transported by airplane for demonstration, and was moved and recalibrated numerous times. It appears to be quite well suited for field work. Most of the instrument conditions are pre-set for BTEX analysis, thus requiring ve little extra work on the field analyst's part. An extensive manual is included with the instrument, which can provide help in troubleshooting the instrument if a problem develops.



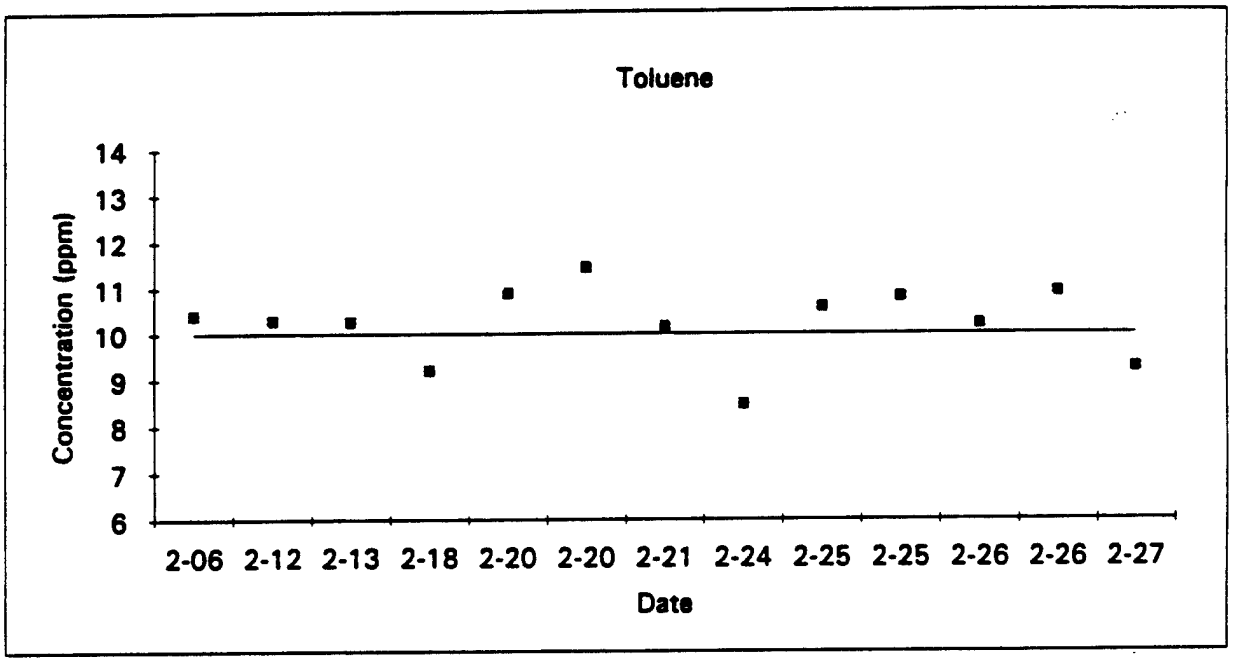
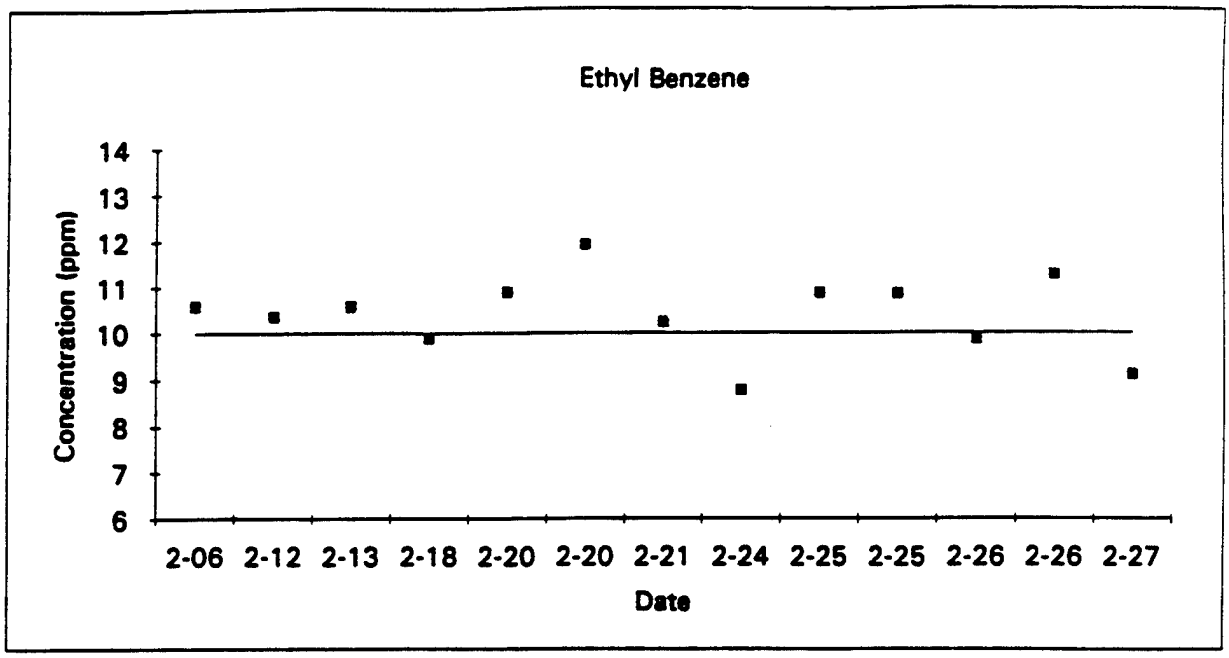


Figure 11. Daily Measurements of a 10 ppm Gas Standard Showing Instrument Drift and Recalibration Points (Continued).



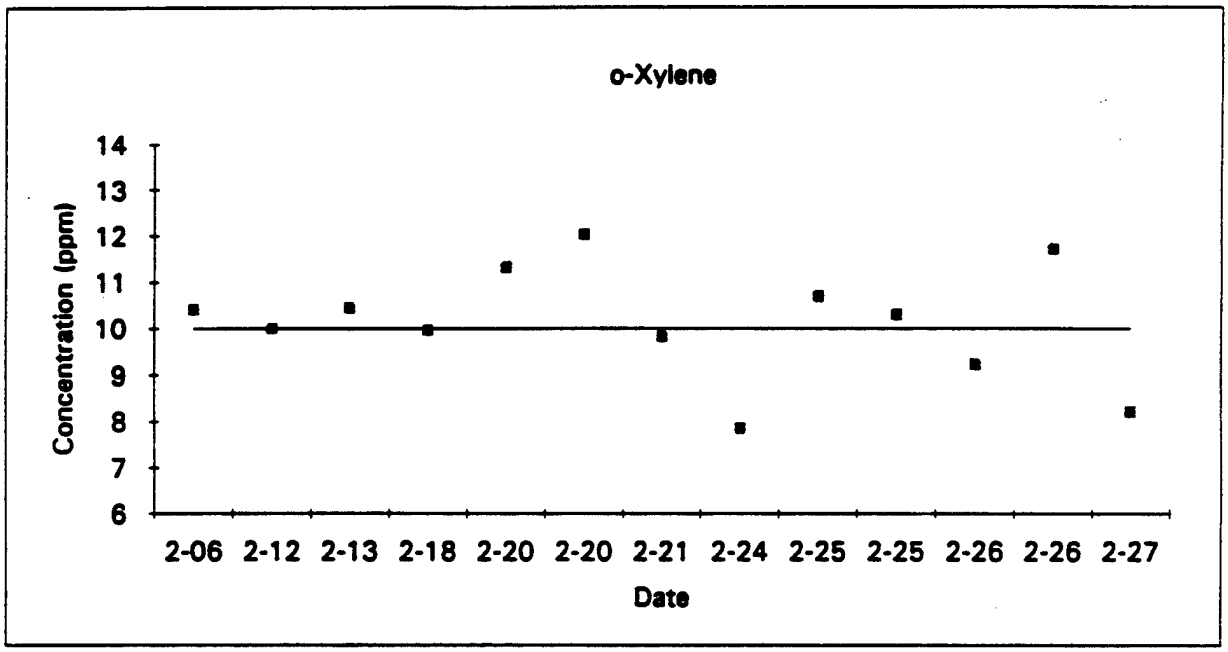
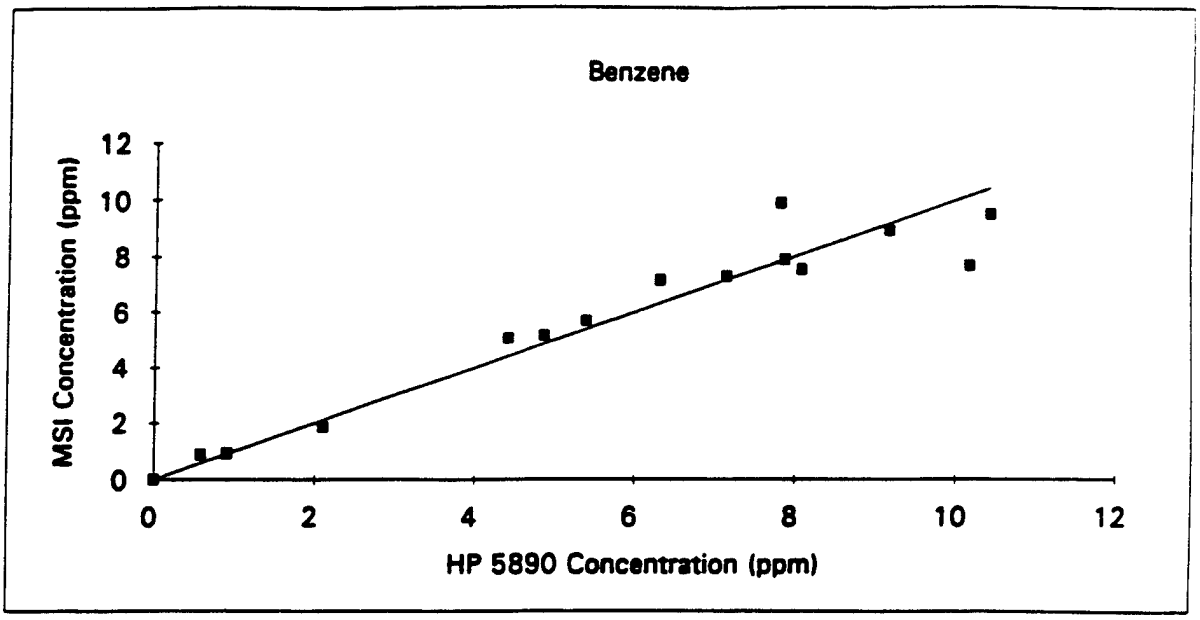


Figure 11. Daily Measurements of a 10 ppm Gas Standard Showing Instrument Drift and Recalibration Points (Concluded).



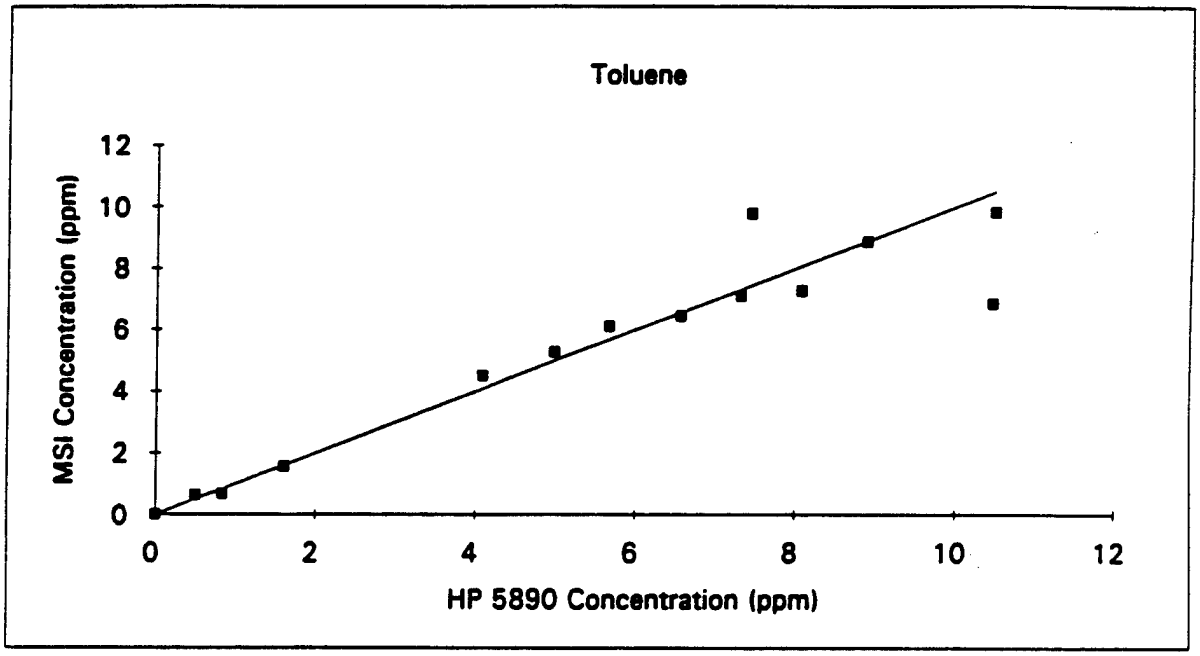
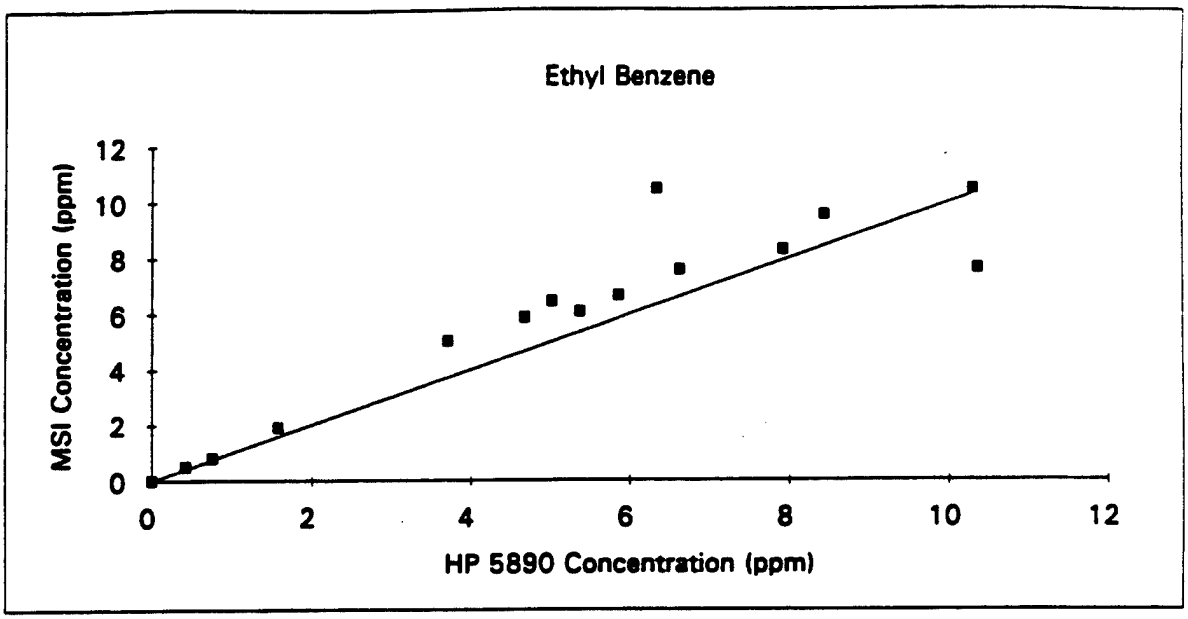


Figure 12. Comparison of MSI-301A Versus HP5890 for BTEX in Soil Gas. The Diagonal Line on the Plot Represents an Ideal Perfect Correlation Between the Two Instruments (Continued).



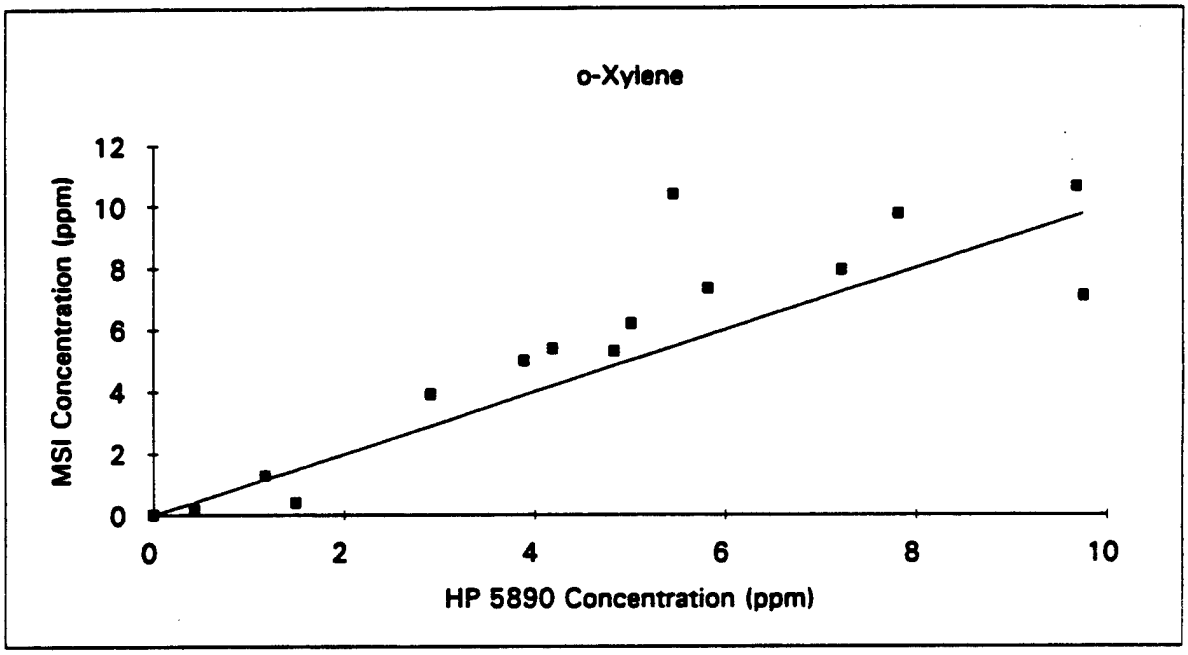


Figure 12. Comparison of MSI-301A Versus HP5890 for BTEX in Soil Gas. The Diagonal Line on the Plot Represents an Ideal Perfect Correlation Between the Two Instruments (Concluded).

5. Training Required

Some background in instrumentation would probably be helpful in learning to operate this instrument. However, the instrument is simple enough to operate that no extensive background in chromatography or instrumental analysis is required. Carefully studying the instruction manual, along with two or three days of hands-on experience, should be sufficient training for the operation of this instrument.

6. Cost

The instrument costs approximately $9,000. Other major costs and potential costs are the same as those described for Lab In A Bag, including standards and analysts time. Depending on the experience of the analyst, six to eight samples per hour can be prepared and analyzed. The cost per sample could be estimated by factoring the cost of an analyst into the number of samples that can be processed per day.

SECTION V

CONCLUSIONS

The Antox immunoassay test is simple to perform and can be used as a quick indicator of BTEX contamination in water. This test provides a reliable, qualitative indicator for BTEX at levels above 75 ppb. Although the manufacturer claims sensitivity to 25 ppb, 75 ppb appears to be a more practical method detection limit.

Detector tubes are simple to use and can provide a semi-quantitative determination for BTEX in water.

The Lab In A Bag sample extraction system provides a reliable means to prepare water and soil samples for volatile hydrocarbon analysis. Low detection limits (10 ppb) are achievable when used with a portable GC.

The MSI GC provides accurate and precise quantitation for BTEX. This instrument offers the advantage of chromatography, allowing quantitation for individual target analytes.

Table 12 provides a summary which compares the techniques evaluated in this study.

TABLE 12. COMPARISON OF FOUR FIELD METHODS.

Method	Method Detection Limit	Precision and Accuracy	Training Required	Equipment Cost	Cost of Supplies (per analysis)
Antox Immunoassay	75 ppb (water)	Qualitative	Several hours	$600.00	$9.00
Detector Tubes	500 ppb (water)	Semi-quantitative	Several hours	$890.00	$20.00
Lab in a Bag	10 ppb (water) 40 ppb (soil)	Quantitative	Several days	$2,000.00	$2.00
MSI	1 ppb (air)	Quantitative	Several days	$9,000.00	$2.00

SECTION VI

RECOMMENDATIONS

All the methods investigated can be used as presently available with few or no modifications; however, further investigations should be undertaken using these procedures under field conditions at actual contaminated sites. Most of the data generated during this study was produced under laboratory conditions. This data provides information as to the potential performance of the method, although performance may vary under field conditions. Until additional field performance data become available, the results of these field methods should be confirmed with the results of laboratory analysis of split samples.

Calibrations should be performed on-site, as site-specific variables, such as temperature, may alter a calibration curve from one site to another. Personnel performing the procedure should be well versed in the method techniques prior to arrival at a field site.

These methods are best suited for use as screening procedures at sites where BTEX is a known or likely contaminant. Chemicals similar to BTEX may interfere with specific quantification. It is recommended that samples taken from sites with unknown contaminants be initially characterized using more exhaustive analytical approaches, such as mass spectrometry. However, at sites with known BTEX contamination, these field screening techniques offer distinct advantages: the lower cost allows a larger number of samples to be analyzed than could be achieved using more refined laboratory methods, providing more thorough site characterization. Additionally, the quick turnaround time obtained through use of these methods allows priorities to be set at a site in a more expedient manner.

SECTION VII

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